An Introduction to Metallurgical Laboratory Techniques

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Preface

WHEN dealing with such a wide subject as metallurgical laboratory techniques in a book of this length, there is a need for selectivity in the choice of techniques to be described. Some of the techniques are really basic, but well proven, laying a foundation upon which the reader may create new and improved methods, relating to the practical aspects of laboratory work, by experimentation, practice and experience.

Doubtless, objections will be raised by some readers because they feel that important subject-matter has been omitted. However, since no two laboratories are run with exactly the same emphases, what is rightly considered as essential material for one may equally be regarded as superfluous by another. In a book of this size and scope it is impossible to cover more than a small section of the simpler laboratory techniques, and I can only plead that I have tried to make my selection as objectively as possible. For those who wish to delve into the many more complex and sophisticated techniques which are available today to the physical metallurgist, there is no lack of more erudite volumes on the market, some of which are listed in the Bibliography.

This book is intended primarily for students studying City and Guilds of London Institute courses, correspondence courses, and the Ordinary National Certificate in metallurgy. I hope also that technical and university students will find the contents of interest as an introduction to further study.

I wish to acknowledge the helpful discussion and continual advice given by Dr. A. A. Smith, Lecturer in Metallurgy at King's College, during the preparation of this book. My thanks are due also to the various suppliers of laboratory equipment for supplying suitable photographs for use as illustrations, and to Mr. M. Harrington for providing Fig. 5.3.

King's College, 1966

Chapter 1

Metallography

IN THE early 1860's H. C. Sorby adapted geological techniques
for the examination of metals under the microscope, and it can
perhaps be said that from his work has developed that branch
of Metallurgy known as Microscopical Metallography.

Microscopic examination of polished and etched specimens is
carried out to determine the size of the grains in the specimen,
and the phases present in polyphase alloys. It is also used to
detect deformation and possible impurities and flaws. To pro-
duce a specimen for microscopical examination it is necessary
to prepare one whose surface appears perfectly flat and scratch-
free when viewed through a microscope. To do this the surface
of the specimen must be ground flat and subsequently polished to
remove the scratches produced by grinding. There are two basic
methods for polishing specimens: one mechanical, in which the
metal is removed by abrasion, and the other electrochemical,
where the attack is by chemicals.

Preparation of Specimens

A suitable specimen for examination may have to be selected
from a ferrous material, such as pure iron, cast iron and steels,
or a non-ferrous material such as aluminium or copper; but
whether ferrous or non-ferrous, the same amount of care and
deliberation must be maintained throughout the whole process
of preparation. An ideal approximate size for a specimen should
be $\frac{3}{4}$–1 in. in diameter and $\frac{1}{2}$ in. in thickness. Specimens below
these proportions, such as thin metal section, become difficult
to grind and polish without rocking and should be mounted in a

simple jig of perspex or a similar substance and further reference will be made to this method later in this chapter. In some laboratories large quantities of regular-shaped specimens may be required. Should this be the case, then the use of a cut-off machine as shown in Fig. 1.1 is desirable. A machine with some form of stop which may be adjusted to the thickness of the specimen required is beneficial. From the stock material the number of specimens required, say, for the whole school year, the duration of a course or phase of research, may then be cut off. Some machines have a micro-adjuster which is fitted adjacent to the machine's vice. This is extremely useful for cutting thin sections of accurate thickness for later microscopical examination. Having selected a specimen of suitable size, one face of the specimen must be prepared for grinding. This may be carried out in the following manner. The face of the specimen which is to be subjected to examination is decided upon and the face or surface is then made as flat as is possible by machining, using a lathe, shaper, or milling machine, or by hand filing. Great care must be observed during the machining or hand filing of the surface to avoid the danger of overheating or distorting the structure of the specimen in any way. The work should be carried out slowly, and if necessary the specimen cooled with water or oil. Overheating or distortion caused by excessive working can in some instances radically alter the structure of the specimen, and although some degree of surface distortion is inevitable this **must** be kept to an absolute minimum. Excessive distortion will result in the destruction of the true structure when the specimen is polished and the whole preparation will be rendered useless. Inclusions present in steels are apt to be torn from their cavities by the action of coarse emery paper. Graphite in cast iron usually suffers in this manner.

Having given the specimen a reasonably flat surface by machining or hand filing, the edges must be chamfered or rounded off to guard against tearing the emery paper. If the edge is the area to be examined, then this edge must be protected in some way, such as clasping metal guards on either side, or by plating with chromium, copper or nickel.

Grinding the Specimen

There are two general methods used in laboratories for grind-
ing specimens, and the first is as follows. A piece of coarse
emery paper, about 280 grade or its equivalent grade, is placed
onto a piece of plate glass approximately ¼ in. in thickness and
measuring approximately 4 in. by 10 in. Grinding is commenced
by holding the specimen in the fingers and rubbing its surface
backwards and forwards on the emery paper. The plate glass
ensures that the emery paper remains flat, so allowing the surface
of the specimen to be ground flat. Great care must be taken to
ensure that the specimen is not rocked in any way during the
grinding operation. Rocking tends to produce a curved of
irregular surface which not only makes further grinding diffi-
cult, but renders the remainder of the preparation almost im-
possible, as illustrated in Fig. 1.2. To guard against a curved or
irregular surface, the specimen should be moved with firm,
positive, even, unhasty strokes. It should be remembered that in
all stages of grinding, speed is of no importance.
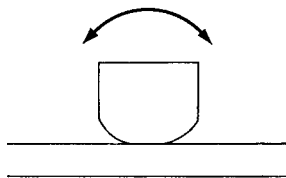
Fig. 1.2. Surface of a specimen due to rocking during the grinding
operation.

Having obtained a reasonably smooth surface by using the
coarse grade of emery paper, observation will show that the
action of grinding has produced scratches on the surface of the
specimen. These scratches should appear to be of an even,
parallel nature, in sharp contrast to the irregular scratches left
by the initial machining or hand filing. Before proceeding with
grinding using subsequent finer grades of emery paper, the

3

specimen, hands and the plate glass **must** be washed under running water and then dried, or wiped down thoroughly with clean cotton wool which should then be discarded. This is very important because should grinding be continued without washing the specimen, any particles of metal or grit left on the surface of the specimen from the previous papers will produce more and possibly deeper scratches. Having washed and dried the specimen, grinding may be continued on the subsequent grades of emery paper, the specimen being rotated through 90° so as to grind the surface across the scratches left by the previous paper. The grinding should be continued for a short time after all the original scratches have been obliterated, as there is every possibility that the previous scratches go deeper than was anticipated. Care should be taken to wash and dry or wipe the specimen, hands and the plate glass between grades of emery paper. The grinding operation may then proceed until the finest or precision paper has been used, at which stage the surface of the specimen will be smooth and free from excessive scratches.

The second method of grinding specimens generally used in laboratories by more experienced workers is to grind the specimen using an apparatus as shown in Fig. 1.3. This simple apparatus is manufactured commercially and consists essentially of a sloping surface to which is clamped bands of emery paper. The paper is supplied in handy rolls of various grades and the rolls are contained in a magazine inside the apparatus. As the emery paper becomes worn a fresh piece can be pulled into position and the worn emery paper discarded. The apparatus is so designed that water is allowed to flow continuously down the emery paper; this automatically washes away grit or particles of metal that would otherwise scratch the surface of the specimen. Specimens that have been heat treated should be kept cool during the grinding operation, and use of this type of apparatus ensures this. An efficient apparatus can be made easily and quite cheaply in the laboratory or workshop.

At this point it should be emphasized that there are no short cuts to the preparation of a first-class specimen. Conscientious grinding of the specimen will certainly be repaid when the time

4

comes to polish, etch and examine the specimen. It is quite use-less to leave noticeable scratches on the surface of a specimen and hope that these will disappear with polishing or etching. Al-though considerable time and energy may have been spent grinding, polishing and etching the surface of a specimen, the failure to observe simple precautions will result in complete obscurity when the specimen is placed on the microscope stage. This is due to the criss-cross lines which will show up under the microscope, signifying the presence of unremoved scratches. Should this be the case, it will be necessary to go back to one of the finer grades of emery paper, and start all over again. The essential points to be remembered during the grinding operation are to wash and dry the specimen frequently and before pro-ceeding to the next grade of emery paper, and to refrain from rocking or placing undue pressure on either side of the specimen. Finally, only moderate to light pressures must be employed, especially when the finer grades of emery paper are used, as at this stage the surface can so easily be rescratched.

Polishing the Specimen

Mechanical polishing

So far the operation has been one of grinding the specimen, and if this has been carried out correctly a specimen whose sur-face is covered with very fine scratches left by the final grade of emery paper will have been produced. The polishing operation differs from that of grinding because it removes the fine scratches by means of burnishing. After the specimen has been well rinsed under running water, it may be polished by means of a polishing wheel, which normally consists of a metal plate to which is affixed a polishing cloth. The polishing wheel is usually rotated by an electric motor, the speed of the wheel being varied by changing the belt drive to another pulley.

There are several commercial grades of cloth available for metal polishing, with varying degrees of nap depending on the

polishing medium used and the metal to be polished. Selection of cloth is best left to personal choice; practical experience will probably determine this. Various types of polishing compounds have been suggested in the past, alumina mixed to a paste with water being the most common. This material, however, tends to be rather messy, and usually involves frequent cleaning of the machine and surrounding area. A diamond abrasive compound has been found to be more satisfactory, and if used intelligently will last for a very reasonable time. Diamond abrasive compounds are manufactured and most of them are supplied in plastic syringes containing various grades and quantities. The grades selected should be suitable for the metal being polished and the degree of polishing required. However, as abrasive compounds are rather expensive, the impregnating of the polishing cloth is best left to a worker with the necessary experience so as to avoid where possible any unnecessary wastage.

Several liquids for lubricating the polishing cloth to ensure that the specimen does not drag are also manufactured commercially, but it has been found that paraffin is a good substitute.
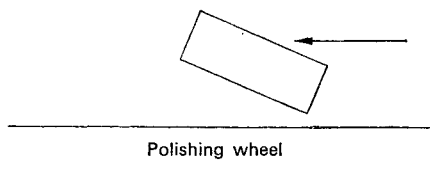

Polishing wheel

Fig. 1.4. Suggested method of applying a specimen to the polishing cloth to avoid an inadvertent "dig in" as well as excessive wear on the cloth.

A lubricant is essential, for should the polishing cloth be allowed to become dry, the pile may be rubbed from it and adhere to the surface of the specimen. This may possibly scratch the specimen too and cause overheating. For the same reasons light pressure should be used during the polishing operation. Moreover, the use of light pressure is less likely to cause the specimen to dig into the cloth and be thrown across the laboratory. Figure 1.4

illustrates a suggested method of applying the specimen to the polishing cloth.

The method used for polishing is to grip the specimen in a similar fashion as for grinding the specimen. Polishing should be carried out with a circular sweep of the hand **against** the rotation of the wheel, not the back and forth motion used in grinding. The specimen should never be allowed to stand still on the wheel, and if it is held at the outer edge of the wheel, polishing will be somewhat faster due to the peripheral speed of the wheel. It should be mentioned that there is a danger of "wobble" at the edge of the wheel, especially if the polishing machine happens to be old and worn, which may produce faults in the surface of the specimen. When starting to polish the specimen, it is better to commence the operation near the edge of the wheel and for the final polishing to slowly work towards the centre, where polishing will be somewhat slower. It is important to remember throughout the whole polishing operation that absolute cleanliness should be maintained if a scratch-free surface is to be obtained. Periodically throughout the operation the specimen should be checked to see how well polishing is progressing.

At this stage a glass dish containing a cleaning solution such as Teepol should be at hand. By placing the specimen in the solution the abrasive compound can be washed from the specimen and the fingers; the polished surface of the specimen may then be wiped gently with the finger, providing there is still some soap solution on the finger to ensure that the surface of the specimen is not scratched. The specimen may then be transferred to running water and the soap solution washed away. This operation should be carried out at regular intervals during polishing, and when finally the specimen has a scratch-free, mirror-like surface, the polishing operation should be completed as follows. The specimen is placed in the soap solution and all the abrasive compound thoroughly washed from its surface, taking **great** care **not** to touch the polished surface with the fingers. It is then rinsed under hot running water, and after removal the operator should gently blow onto the surface of the specimen to remove the surplus moisture. The specimen is quickly

7

transferred to a dish of acetone. The specimen is removed and, while it is still warm, is gently blown on to assist in drying off the surface. Care should be taken not to inadvertently spit at the same time, as obviously the specimen will again have to pass through the washing and drying sequence. It should be noted that if the specimen has been mounted in perspex, it should not be dipped into acetone, or the perspex will be dissolved and left as a film over the surface of the specimen when the solvent evaporates. In this case the surplus moisture can be removed from the surface of the specimen by the careful use of a piece of cotton wool. A gentle wiping of the surface is all that is required. Care should be exercised during the washing and drying operation not to leave a film of grease on the polished surface, or to stain the surface in any manner. Both are quite detrimental to the further operations. The surface of the specimen can be wiped with cotton wool soaked with benzene, if a persistent grease film remains on the surface. Speed of drying is **all important,** especially in steels, because moisture left on the polished surface will soon produce rust.

When polishing a large quantity of specimens, a polishing machine as shown in Fig. 1.5 will be found to be an advantage. While one specimen is being automatically polished another specimen may be either ground or etched. This technique enables the grinding and polishing to be carried out at a faster rate.

A word or two about polished surfaces. Most metals will shine from the action of polishing, but when polishing bearing metals, the surface of the specimen often does not shine with a mirror-like finish as do other metals. When this is the case, then common sense must prevail and the worker must learn to judge by inspection when a specimen is suitably polished.

To summarize, the important factors affecting a successful finish to a specimen are:

(a) to avoid overheating the specimen during the grinding and polishing operations;

(b) to ensure absolute cleanliness throughout the whole sequence of grinding and polishing;

(c) as with the grinding operation, **not** to attempt to rush the polishing. **All** the scratches must be removed from the surface of the specimen if later examination is to be successful;

(d) to dry thoroughly to avoid staining.

Technique for Soft Metals

When preparing specimens for examination it is important to remember that special care should be taken with soft metals. As examples of this the preparation of lead and dental alloy will be described, since the technique used for these alloys is general for most soft metals.

The preparation of lead is difficult owing to the softness of the metal. A specimen should be cut from the stock or sample material, using a very sharp hacksaw, and grinding should be effected with emery paper well lubricated with paraffin or oil. A very gentle pressure should be used during the grinding operation, otherwise the surface may be deeply scratched. It is advisable to finish the grinding using well-worn emery paper which is free from contamination by grit particles.

Polishing should be carried out using the polishing wheel at the slowest speed, and a liberal quantity of lubricant applied to the polishing cloth to ensure that the specimen does not drag. If the polishing wheel does not have provision for varying its speed, then polishing should be carried out near the centre of the wheel where, as mentioned earlier, the speed of polishing will be somewhat slower.

To prepare a specimen of dental alloy for examination great care must be taken. Grinding should be carried out using worn emery paper well lubricated with water. Frequent washing of the specimen is desirable and light pressures are essential when grinding.

Polishing of a dental alloy is carried out by way of a polish-etch attack, using the polishing wheel at a slow speed. The polishing cloth is impregnated with a fine diamond abrasive compound,

and the etching reagent is used to lubricate the cloth. This method enables the specimen to be polished and etched slowly, during which time it should be inspected at frequent intervals to avoid the risks of distortion or overetching. The etchant used in this case is a solution of concentrated ammonia and 20 per cent of 20 volume hydrogen peroxide. The main disadvantage when using this technique is that the etchant may cause the polishing cloth to disintegrate, and to minimize this danger the cloth should be washed after use.

Mounting the Specimen

Specimens to be mounted prior to grinding and polishing usually fall into the following categories:
  (a) thin pieces of metal, wire or rivets;
  (b) a sectioned specimen which requires examination of the edge;
  (c) specimens, other than (a), that are too small to handle comfortably when being ground and polished;
  (d) irregular shapes which are to be subjected to hardness tests.

HOT MOUNTING

Most specimens can be hot mounted unless the nature of the specimen is such that the microstructure is altered or damaged by the high temperature necessary for hot mounting. A good example of a specimen of this type is a dental amalgam. At the temperature of hot mounting, mercury separates from the alloy, thus completely breaking up the microstructure.

Hot mounting consists of surrounding the specimen with perspex powder in a mould of suitable dimensions. The powder is compressed in the mould and heat is applied to the outside of the mould. The perspex powder melts around the specimen, is allowed to cool, and after a predetermined time the now mounted specimen is ejected. A mounting press is usually used to

mount the specimens; a suitable press is one which will compress the perspex powder to approximately 4000 lb/in². Figure 1.6 shows a typical press used for this purpose. The press is basically a cylinder in the bottom of which is a piston, operated by means of hydraulic pressure. Surrounding the cylinder is a water-cooling jacket which in turn is surrounded by a heating element.

A specimen is placed into the cylinder, to rest on the piston with the surface to be examined face downwards. A generous amount of perspex powder is poured into the cylinder, to allow for the contraction of the powder under heat and pressure, and a contra piston is fitted and locked into the top of the cylinder. The press is pumped up by means of the lever until a pressure of approximately 2000 lb/in² is recorded on the pressure gauge. At this time the heating element is switched on, and as the perspex begins to melt the pressure in the press will fall. This displacement of perspex as it melts is rectified by pumping the press and maintaining a pressure of about 2000–3000 lb/in². The pressure in the press will continue to fall until the perspex has completely melted, whereupon the press may be pumped until a pressure of approximately 4000 lb/in² is recorded. The pressure is held at this point for a couple of minutes. The heating element is then switched off and the water-cooling turned on. As the mounting press cools down the indicated pressure drops until it again reaches approximately 2000 lb/in², when the hydraulic pressure may be released and the mounted specimen ejected from the press.

The mounted specimen removed from the press will now only need to have any sharp edges removed from the mount before grinding and polishing is commenced. This can be done by the use of a linisher, but care and frequent cooling must be observed during this operation as there is a danger of melting the perspex around the specimen. This will produce, as shown in Fig. 1.7, a mounted specimen with channels around it which retain dirt, grit and moisture. In extreme cases the specimen will fall out of the mount. When a linisher is used, it is advisable to have a beaker of water close by, to periodically cool the specimen and mount. When a mounted specimen is cooled by dipping it

11

into water, it must be thoroughly dried before it is stored away otherwise, as in the case of steel specimens, rust will develop on the surface of the specimen.
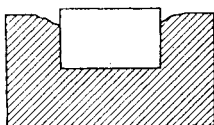

FIG. 1.7. Groove formed from excessive linishing of a mounted specimen.

At this stage it is well worth illustrating some methods of mounting in perspex specimens which have awkward areas to be examined, as in Fig. 1.8.

End of a small
diameter rod

Oblique edge

Specimen of
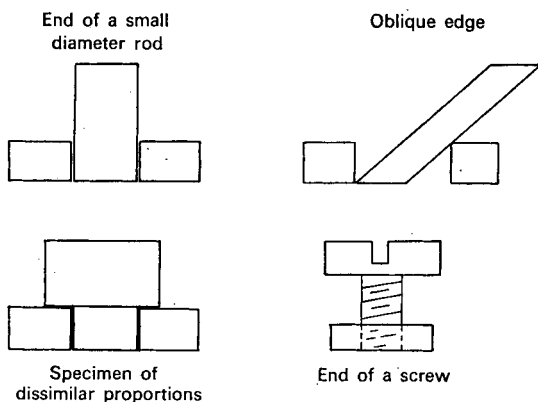dissimilar proportions

End of a screw

FIG. 1.8. Methods of mounting specimens which have awkward areas to be examined.

COLD MOUNTING

Cold mounting may be used for specimens whose microstructure would be affected by the application of heat. An example of a specimen requiring mounting by this method is a

12

dental alloy or dental amalgam. For this type of specimen a manufactured cold mounting material is desirable.

To cold mount a specimen a small piece of tube with the approximate dimensions $\frac{1}{2}$ in. deep and $\frac{3}{4}$ in. bore is used as the mould. These dimensions may be adjusted for larger specimens.
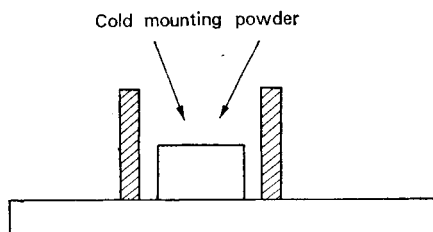
Cold mounting powder

FIG. 1.9. Method for cold mounting a specimen, showing a section of the mounting ring, into which the cold mounting powder is poured.

The specimen with the piece of tube over it is placed face downwards onto a piece of plate glass. The cold-mounting powder is poured into the tube. To this is added a hardener which reacts with the powder and the whole eventually solidifies (see Fig. 1.9). When this stage has been reached, the mould can be removed from the plate glass by means of a sliding action and the specimen is in turn removed from the mould. It should now have any sharp corners or edges removed, and it is then ready for grinding and polishing.

Specimens may also be mounted in plasticine **providing** the specimen has previously been ground, polished and etched. This is beneficial where specimens are in daily use and it is necessary to check the condition of the specimen. A simple device is available for pressing specimens into a piece of plasticine on a glass slide. The mounting device ensures that the specimen is mounted parallel to the microscope stage, for examination, and Fig. 1.10 illustrates this mounting device. When using this apparatus to mount a polished and etched specimen, a filter paper or similar material placed over the polished surface of the

13

specimen will ensure that it does not become scratched. Examination of the specimen at this stage will not show the crystal structure, but only such effects as cracks, inclusions, etc.
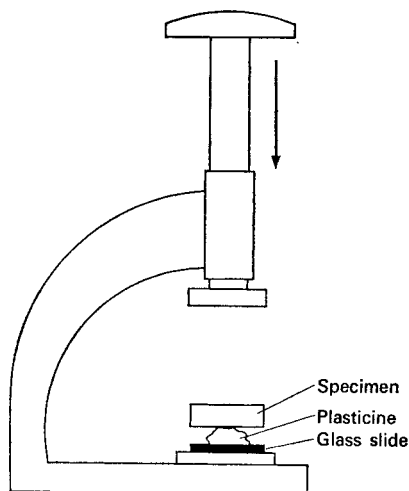

FIG. 1.10. Apparatus for mounting a specimen onto a glass slide.

Etching the Specimen

In metallography, etching is the process by which structural details are revealed by the preferential attack of reagents on a metal surface. An etching reagent will attack the surface of the specimen and remove the flowed layer produced by polishing. This layer is known as the Beilby Layer after Sir George Beilby, the first man to realize its presence.

The structures of a metal revealed by etching are known as the **Macrostructure** and the **Microstructure**. The macrostructure is that revealed through macroscopic examination, by means of the naked eye or by the low-power magnification of a magnifying glass. The microstructure of a metal is that as revealed by a microscope, by greater than $10 \times$ magnification.

14

CHEMICAL ETCHING

Before the specimen is etched it must be absolutely clean, otherwise there is a possibility that it will etch unevenly. The failure to etch correctly can usually be attributed to inadequate cleaning and the film of grease which remains on the polished surface. If this is the case, then the surface of the specimen should be wiped with cotton wool soaked in benzene. The specimen must be dry because the presence of moisture can radically affect the chemical behaviour of some etchants, particularly those in alcoholic solution such as nital and picral. For etching purposes the specimen should only be handled with a pair of crucible tongs, to guard against leaving smears from the fingers on its surface. For the sake of clarity, the technique to be described relates to the etching of a steel specimen.

Having ensured that the specimen is free from grease and moisture, some 2 or 4 per cent nital is poured into a porcelain crucible and some acetone into a glass dish. The specimen, held in a pair of tongs, is plunged into the etching reagent and gently agitated. A slight "bloom" effect on the surface of the specimen should be detected, indicating that the etchant has taken. The specimen must be quickly transferred to hot running water, to wash away the etching reagent, and then, while the specimen is still hot, is plunged into the acetone for a short time, removed and allowed to dry. The surface of the specimen should be slightly dull in appearance and should be inspected to determine the extent of etching. Initial inspection is usually carried out using the naked eye or by low-power magnification, and afterwards by use of a microscope. If the structure of the specimen cannot be seen clearly through the microscope, because of "fogginess" or "ghosting" of the structure, then the specimen has been underetched. In this case, the specimen should be immersed in the etchant for a longer time. If the microstructure is very dark in appearance, with possible staining by the etchant, then the specimen has been overetched. If this is the case, the only solution is to revert to the polishing wheel and to repolish the surface of the specimen, in order to remove the effect of the

etching reagent. The specimen is re-etched but for a shorter period of time. If staining is persistent, it is advisable to ascertain whether or not the acetone used for drying the specimen has absorbed too much water. If necessary, fresh acetone should be used.

The time required to etch a specimen varies with different metals. Some metals, such as the bronzes, can be etched in a few seconds while some stainless steels, being somewhat resistant to attack by most reagents, may take much longer. It is better to etch the specimen in short stages, inspecting it between stages until a satisfactory structure can be seen, rather than for a fixed time. This method should avoid the possibility of over-etching and the inconvenience of repolishing the specimen. In the case of the inexperienced worker, etching of specimens requires some degree of trial and error to obtain satisfactory results. With practice, however, the worker will soon acquire the ability to judge, with some degree of accuracy, when a specimen has been etched satisfactorily, and no attempt should be made to rush this operation. It should be noted that should persistent scratches remain on the surface of the specimen after prolonged
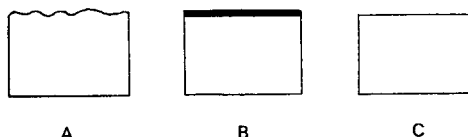
Fig. 1.11. Principles of etching: (A) specimen before polishing, (B) flowed layer produced by polishing, (C) flowed layer removed by etching reagent.

polishing, they can occasionally be removed by purposely over-etching the specimen and then repolishing. The etchant attacks the surface layer of the specimen and in so doing removes the scratches to some degree, which will probably be sufficient to enable the specimen to be examined.

The principles of etching are described in Fig. 1.11. A selection of the many reagents that are used in laboratories and some precautions that should be observed are given in Tables 1 and 2.

TABLE 1. STANDARD ETCHING REAGENTS

Metal	Reagent
Common steels Quenched steels Tempered steels	*Nital* Solution of nitric acid in alcohol, 2–4 per cent in strength.
High carbon steels	*Picral* Picric acid in alcohol, 5 per cent in strength.
Stainless steels	*Vilella etch* 3 parts concentrated hydrochloric acid, 1 part concentrated nitric acid, 6–8 parts glycerine. *Note.* First heat the specimen in hot water. The etchant must not be allowed to stand for long periods of time.
High alloy steels in general	Hydrochloric acid 5 ml Picric acid 1 g Alcohol 100 ml
Chromium steels and nickel chromium steels	Nitric acid 10 ml Hydrochloric acid 20 ml Glycerol 20–30 ml
High-speed steels and tungsten steels	Hydrochloric acid 10 ml Nitric acid 3 ml Methyl alcohol 100 ml

TABLE 2. STANDARD ETCHING REAGENTS

Metal	Reagent
Pure aluminium	(a) *Tuckers etch* 180 ml hydrochloric acid 60 ml nitric acid 60 ml hydrofluoric acid 100 ml water. *Note.* Reveals the macrostructure for macroscopic examination. (b) 2 per cent solution of hydrofluoric acid. *Note.* Reveals the microstructure for micro- scopic examination. *[continued over*

TABLE 2 *(continued)*

Metal	Reagent	
Brasses and copper alloys	2 per cent solution of ferric chloride	
Pure copper	880 Ammonia Hydrogen peroxide (a few drops) Ammonium persulphate *Note.* Make up fresh for each application.	
Aluminium and its alloys	(a) 10 per cent solution sodium hydroxide in water	
	(b) Sulphuric acid	20 ml
	Water	80 ml
	(c) Nitric acid	20 ml
	Water	80 ml
Aluminium bronze	(a) Nitric acid	20 ml
	Hydrofluoric acid	10 ml
	Water	150 ml
Pure lead	(a) Nitric acid	
	(b) Solution of perchloric acid in water	600 g/l
	(c) Acetic acid	30 ml
	Hydrogen peroxide	(30 vol) 10 ml
Tin	(a) 2 per cent nitric acid in alcohol	
	(b) Acetic acid	50 ml
	Water	50 ml
	Hydrogen peroxide (30 vol)	1 drop
	(c) Ammonium persulphate	5 ml
	Water	95 ml

ELECTROLYTIC POLISHING

Probably the greatest contribution to the success and development of electrolytic polishing has been made by Jacquet, by whom it was originally introduced.

The basic theory of electropolishing (as it is usually called) will be readily understood if the effect produced by the initial preparation of the specimen is considered. The surface is covered

with irregularities which resemble hills and valleys, and the irregularities must be removed to obtain a relatively smooth surface. If the specimen be made to form the anode of an electrolytic cell, then the anodic dissolution under specific conditions will result in the smoothing and brightening of the surface of the specimen. By placing the specimen in the electrolyte, its surface is subjected to attack by the electrolyte and the dissolved metal
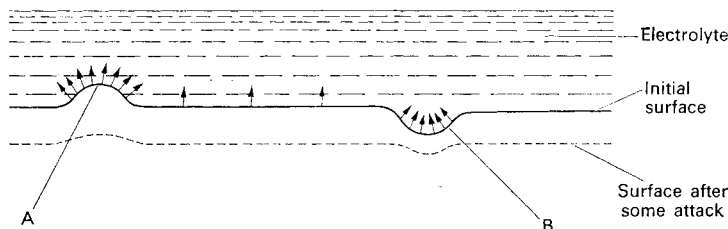

FIG. 1.12. The smoothing effect produced by chemical dissolution. At a protuberance (A) the surface area over which attack can occur is large compared with the volume of metal attacked beneath the surface; metal ions in solution can diffuse away rapidly into a large volume of electrolyte (shown by arrows). At a pit (B) the reverse is the case, and a "bottleneck" of concentration is built up in the electrolyte, slowing down the rate of attack. (Courtesy of Butterworth, *Chemical Engineering Practice*, Vol. 8. A. A. Smith.)

forms a layer on the surface of the specimen. In the valleys the layer is thick while on the hills it is thin. Consequently the diffusion of active ions from the electrolyte to the specimen, and the products or dissolved metal of this attack, away from the surface of the specimen is slower from the valleys than from the hills. As a result an overall smoothing of the surface of the specimen is obtained. Figure 1.12 will clarify this.

For routine work where satisfactory abrasive techniques are established, electropolishing has little to offer, unless it can perhaps be said that in the case of some metals such as chromium steels, electropolishing is somewhat quicker. The particular advantages of electropolishing are that an excellent scratch-free and undeformed surface can be obtained. For example, spe-

cimens containing deformation structures and specimens that have been subjected to microhardness comparison tests are unsuitable for abrasive polishing techniques. Electropolishing is also useful if only certain parts of a specimen are to be polished; the remainder of the surface may be protected by coating with a shellac or plastic paint which is suitably resistant to the electrolyte. The specimen may be pre-polished using one of the finer grades of emery paper, which reduces the overall polishing time.
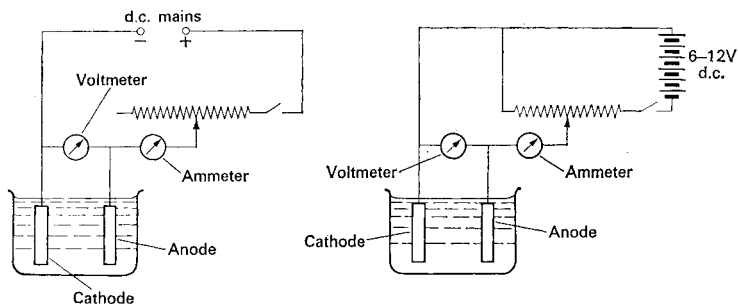

FIG. 1.14. Basic electropolishing circuits: (a) high current density (this bath will normally contain some cooling device, a thermometer, and a stirrer (omitted for clarity)); (b) low current density; this bath will normally contain a stirrer (omitted for clarity). (Courtesy of Butterworth, *Chemical Engineering Practice*, Vol. 8. A. A. Smith.)

There can be no hard-and-fast rules regarding electropolishing techniques since this is initially a matter of trial and error in making necessary adjustments to the electropolishing apparatus. Repeated attempts at polishing should be carried out, until a satisfactory polished surface has been obtained. The settings of the apparatus for each particular metal that is polished should be noted and the records will be found useful for future occasions. The relevant literature should always be consulted prior to polishing the specimen, to obtain information concerning recommended electrolytes and instructions for operating the apparatus. Figure 1.13 illustrates an apparatus for electropolishing and etching specimens under laboratory conditions, while Fig. 1.14 shows a typical electrolytic circuit.

ELECTROLYTIC ETCHING

Etching by means of electrolysis is normally carried out using the conditions for electropolishing, but with a lower current density. Once again, the finer adjustment of conditions for this technique is a matter of trial and error, and frequent microscopical examination to determine the extent of the etching is desirable. Of course, etching of an electropolished surface may

TABLE 3. STANDARD ELECTROLYTES

Metal	Electrolyte	
Iron, alloy steels, carbon steels	Perchloric acid (density 1·61)	18·5 ml
	Acetic anhydride	76·5 ml
	Distilled water	5·0 ml
	Note. The solution should stand for 24 hours before use.	
Austenitic steels	Perchloric acid (density 1·61)	10 ml
	Acetic anhydride	20 ml
	Note. The solution should stand for 24 hours before use.	
Steels and general reagent for many metals	(a) Perchloric acid (density 1·61)	20 ml
	Ethyl alcohol containing	
	3 per cent of ether	80 ml
	(b) Perchloric acid (density 1·61)	10 ml
	Glacial acetic acid	100 ml
	(c) Nitric acid	10 ml
	Methyl alcohol containing	
	5 per cent ether	90 ml
Stainless steels (18 per cent Cr, 8 per cent Ni)	(a) Orthophosphoric acid	37 ml
	Glycerol	56 ml
	Water	7 ml
	(b) Orthophosphoric acid	67 ml
	Sulphuric acid	20 ml
	Chromic acid	2 g
	Water	11 ml
	Note. This is a slow-acting reagent.	
	(c) Sulphuric acid	15 ml
	Acetic acid	55 g
	Water	30 ml

be done by the standard chemical etchants (see Table 1) whereas Table 3 gives a selection of the many electrolytes used for polishing and etching by electrolysis.

The appropriate literature should always be consulted regarding the use of the electrolyte. Many of the recommended reagents contain perchloric acid and acetic anhydride, and there is a definite risk of explosion unless the solution is kept cool. This applies both to mixing and to the use of the electrolytes. Similar risks occur with perchloric acid–ethyl alcohol mixtures and it cannot be too strongly emphasized that explosions have occurred, using perchloric acid mixtures, under **normal** operating conditions in which **every** precaution was **apparently** taken. Ethyl perchloric, for example, has been compared with nitroglycerine in its explosive properties. The preparation of such mixtures is best left to a worker who has had the necessary experience in dealing with these acids and is fully aware of their potential danger.

There are a number of commercially manufactured electrolytes available and are usually supplied ready for use. Once again the manufacturer's literature should be consulted before deciding which electrolyte suits the need of the worker.

Storage of Specimens

Prepared specimens in either an etched or unetched condition should always be stored in a storage cabinet or desiccator, to avoid the possibility of loss or damage to the specimen.

Metallurgical specimen storage cabinets normally use small containers of silica gel crystals to keep the atmosphere in the cabinet dry. These containers require drying out regularly, and are renovated by placing them in an oven for approximately 1 hour. To show the moisture content, the crystals used are self-indicating. They are pink in colour when saturated with moisture, and blue when completely dry. The atmosphere in a desiccator may be dried by using the usual solid desiccants.

Microscopy

The visual examination of polished surfaces of metals is one of the simplest ways of gaining information about metals and alloys. Metals are made up of interlocking masses of minute crystals or grains, the size and shape of which are largely responsible for the properties of the metal. Two castings may have identical chemical compositions, and yet one of them could be useless for the task for which it was designed because the size and shape of the grains of which it is composed are not correct. This method of examination will also usually reveal small cracks and pores in the specimen and the presence in steels of small quantities of sulphur or phosphorus, which form very brittle sulphides or phosphides between the grains and decisively lower its strength. Microscopic examination will also yield considerable knowledge regarding the history of the specimen, particularly in respect of heat treatment and mechanical working.

THE METALLURGICAL MICROSCOPE

The metallurgical microscope differs from the more common microscope in that it is specially designed for the examination of opaque specimens. Figure 1.15 illustrates a simplified microscope system.

A typical bench-type microscope consists of a base or stand to which is attached the microscope arm, the stage to support the specimen, usually adjustable to admit specimens of various shapes and sizes, and the tube. The tube carries a side arm containing the source of illumination; the objective lens is contained in the lower end of the tube, and the eyepiece at the upper end. The objective lens is the most important and expensive of the two lenses, since it has to resolve the fine details of the specimen being examined. The light source is carried through the arm, deflected onto the specimen, and reflected back along partially the same course through the objective and eyepiece. The width of the illuminating beam is controlled by the aper·
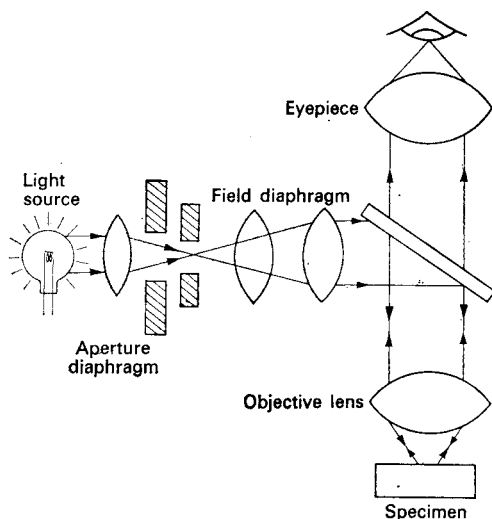
FIG. 1.15. The metallurgical microscope simplified.

ture diaphragm. Generally this should be closed down so that the beam of light is sufficient to cover the back of the objective lens. If the diaphragm is opened too widely, light will be reflected from the sides of the microscope tube, causing glare in the field of view.

MAGNIFICATION

The magnification given by the objective lens depends on its focal length. The shorter the focal length, the higher the magnification. The overall approximate magnification of the microscope can be found from the following: LM/F, where L = the tube length of the microscope measured from the back of the objective lens to the lower end of the eyepiece; M = the power of the eyepiece; F = the focal length in millimetres of the objective. From the formula it can be calculated that a microscope having a tube set at 20 cm (200 mm), and using a 4 mm focal length objective with a 6 × eyepiece, will have an overall magnification of $(200 \times 6)/4 = 300$. All objectives have

24

initial magnifying power, that is the objective is capable of magnifying the specimen a positive number of times without the use of the eyepiece. Usually the magnification or power of the objective is engraved on the side of the objective lens. Eye-pieces also have initial magnifying power, and the power of the eyepiece is also engraved on the lens mount. The purpose of the eyepiece is to enlarge the image as formed by the objective lens and so render it visible.

The magnification produced by the microscope is determined by the choice of a suitable eyepiece and objective lens. However, magnification is not the sole criterion; it is obviously useless to obtain a large image if the details are blurred and indistinct. Hence the resolving power of the objective lens must also be borne in mind, i.e. the smallest distance between lines which still appear separate in the field of view. The resolving power of the lens is related to another quantity, the Numerical Aperture, which is also usually engraved on the objective lens, by the formula $\delta = \lambda/2NA$, where δ = the resolving power in milli-metres; λ = the wavelength of light; NA = the numerical aperture. Thus for small values of δ we need light of short wave-length (hence the use of filters for high-magnification micro-scopy) and/or high numerical aperture for the objective. Now the numerical aperture is defined by the formula $NA = \mu \sin \alpha$, where μ is the refractive index of the medium between the specimen and the objective (usually air, for which $\mu = 1$), and α is the semi-angle of the maximum cone of light which can enter the lens. The value α is variable, becoming larger for high-power lenses, but of course never reaching 90°; the numerical aperture is also increased by increasing μ, which may be done by substi-tuting an oil, for which $\mu > 1$, instead of air between the specimen and the objective. Hence the highest-power lenses, for which a high degree of resolution is required, are used as oil immersion lenses. A thin film of immersion oil, or cedar wood oil, is applied to the objective lens and one or two drops to the surface of the specimen. The microscope stage is adjusted until the two films of oil meet; careful adjustment is then required to bring the specimen into focus.

25

CARE OF OPTICAL PARTS

Immersion oil should never be allowed to dry and harden on the objective lens or, for that matter, on the surface of the specimen. After use, the lens must be cleaned with cotton wool or lens tissue moistened with xylene. Solvents such as acetone and benzene must never be used, as these solvents will loosen the lens in its holder by dissolving the lens-fixing medium. To avoid accidentally scratching the fine finish of the lens care should always be taken to avoid touching the surface of the optical glass with the fingers. Any smears or marks may be removed from the lens with cotton wool moistened with xylene, and dust should be removed using a soft camel-hair brush. To guard against accidental damage, the lenses should always be replaced after use and stored in their appropriate boxes or containers, which are usually supplied with the microscope. The microscope should always be stored in a dust-free container when not in use or, failing this, completely covered with a cover made of plastic or similar material. It is of great importance to keep dust from the microscope and optical parts, for should dust particles penetrate to the inside of the lenses of the microscope, the affected instrument or lens may have to be returned to the manufacturers for cleaning. The microscope or lenses must never be tampered with. A microscope is a precision instrument and should be treated as such, and its accuracy will only be maintained if it is handled with care on every occasion. A typical bench-type microscope is shown in Fig. 1.16.

FIELD DIAPHRAGM

The object of the field diaphragm is to reduce the internal glare and light reflections within the microscope. Providing the microscope has been focused correctly, the edge of the diaphragm will be visible in the field of the image. There is a reduction in the size of the image, when the diaphragm is closed down, and an increase in contrast without any apparent effect on the resolution.

OBLIQUE ILLUMINATION

Oblique illumination is used if there is little contrast of the structure of the specimen, also when it is necessary to accentuate certain parts of the structure which have been cast into shadow by structure that is standing in relief. It should be remembered, however, that oblique illumination will also emphasize surface defects such as etch pits and scratches.

DARK FIELD ILLUMINATION

The more elaborate types of microscope usually make provision for this technique, which may be useful on occasion to highlight certain features of a microstructure. Under normal incident illumination, light is only reflected back up the objective lens from the level parts of the surface of the specimen; any light falling on irregularities or filtered parts of the surface of the specimen is scattered, and so these effects appear dark (e.g. grain boundaries). Dark field illumination gives the reverse effect. By using light which falls obliquely on the surface of the
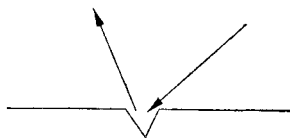
FIG. 1.17. Dark field illumination. Light strikes the grain boundaries and is reflected back through the objective lens. The grain boundary appears light, the remainder of the specimen appears dark.

specimen (at a much greater angle of obliquity than is the case for oblique illumination), only irregularities are able to reflect light up the objective lens (see Fig. 1.17). Hence, in general, grains appear dark and grain boundaries light.

PHASE CONTRAST AND POLARIZED LIGHT

Phase contrast and polarized light are used in metallography for specialized examination of metal structures. But since the subject is too vast to be dealt with in a book of this length, reference should be made to more appropriate literature.

EXAMINATION

Before examining a specimen it must be ensured that it has been prepared adequately and mounted by one of the methods described earlier. The specimen is placed on the microscope stage and brought into focus by using the coarse adjustment and then the fine adjustment for final focusing. By looking through the eyepiece the light will get brighter as the objective lens nears the focal point; adjustment is made, with care, until the surface of the specimen is in full focus. Since the objective lens has generally been designed to operate at a fixed tube length, under which conditions optimum results are obtained, the tube carrying the eyepiece should be drawn out to the position indicated on the tube. The aperture diaphragm in the illumination system should be closed down to a position where illumination begins to decrease. This is to limit glare due to reflection in the tube. Great care should be taken when focusing the microscope using a high-power objective lens. The position of the objective lens is usually very close to the specimen, and it is very easy to adjust the microscope past the focal point and so strike the specimen with the objective lens. This causes damage not only to the objective lens but also to the surface of the specimen. If necessary, further adjustment may be made to the light source until the desired effect has been obtained.

A low-power objective lens should always be used during the initial stage of examination, to give a general impression of the structure of the specimen. The low-power objective lens is then replaced with one of higher-power magnification for more detailed examination. Common sense must prevail when exam-

ining specimens; do not assume that the first view of the structure is typical of the whole specimen. Any change in grain size, distribution of phases in polyphase metals and impurity contents should be noted. These are frequently different at the edge, and the properties at the edge may be very important, e.g. in a specimen that has suffered a fatigue fracture. Only after careful inspection under low-power magnification should the specimen be examined under high-power magnification, selecting a typical field and noting, by way of sketches if necessary, any details of the structure, shape of the grains and any preferential orientations. It is important to realize what the various blemishes which may occur in even the best-regulated laboratories look like. Any specimen which has not been examined immediately after polishing may exhibit rust marks, moisture stains, dust and even finger-prints, which under the microscope can give a very misleading appearance to the structure. It is probably true to say that a large part of metallography is the art of knowing what to ignore; the author has more than once had experience of students carefully sketching their eyelashes into the microstructure of steels, due to a reflection effect from the upper surface of the eyepiece! Some examples of the more frequently met imperfections are given in Fig. 1.18.

Photomicrography

After examination, it may be desirable to photograph the structures of the specimen. This is a useful way of recording microstructures, and in some instances may be essential. For example, it may be required to see how the microstructure varies with increasing depth below the surface, or how it varies before and after heat treatment or mechanical working. To obtain this information it would be necessary to regrind and polish the original surface. Photomicrographs are always more accurate than sketches of microstructures for inclusion in laboratory reports, and to that end every care should be taken to ensure that the microstructure is accurately recorded.

The microstructure may be photographed using a standard bench microscope; for this purpose it is essential to use a reflex camera, so that the required field of view may be accurately centered and focused prior to photographing it. Alternatively, adaptors are made for bench microscopes using photographic plates rather than a camera; these have the advantage of a field of view at the exact magnification required. They are not, however, as suitable as a camera if it is desired to take a series of pictures in fairly rapid succession. Ideal conditions and careful handling of the microscope are essential with this method, and even though suitable photomicrographs can be obtained, better results may be obtained by the use of a high-precision microscope specially designed for both visual and permanent recording of metallographic structures. Figure 1.19 shows an instrument of this type which has been designed with more elaborate and refined adjustments so as to produce an image of the highest possible quality.

It is highly convenient to have a really bright source of light for photomicrography, preferably a mercury vapour or carbon arc lamp. This gives a much brighter image on the focusing screen, making for ease of identification of structures and focusing. It also cuts down the inordinately long exposures which are necessary with the ordinary microscope bulb, and which are one of the major disadvantages of using an adapted bench microscope. This is especially true if there is any heavy machinery in the vicinity!

METHODS OF DETERMINING
CORRECT PLATE EXPOSURE

An important factor which gives rise to successful photomicrographs is the length of time that the photographic plate or film is exposed to the subject. The exposure time varies with the brightness of the subject, as seen on the focusing screen, and with the speed of the photographic emulsion on the plate or film. Unless the worker has had considerable experience in

dealing with exposure times, it is advisable to run off a test plate before attempting to take the final picture. A test plate is simply a photographic plate or film which has been exposed a portion at a time, whilst the exposure time for each portion of the plate or film has been varied.

For the first portion the slide cover is withdrawn, leaving a portion of the plate or film for exposure. As an example, this portion may be exposed for 8 seconds. The slide cover is then withdrawn further and the second portion of the plate or film is exposed for 4 seconds. The remaining portions may be exposed for 2 seconds each. In this particular example, the four portions of the plate or film have now been exposed for 16 seconds, 8 seconds, 4 seconds and 2 seconds. Although the initial exposure time may be varied at will, once the photographic plate or film has been processed, the worker will be in a position to decide which exposure is the most suitable for the production of a first-class photomicrograph. After a little experience it is usually possible to dispense with test strips for most standard metals and magnifications by making notes of past satisfactory exposures and the conditions under which they were obtained.

PRODUCING PHOTOMICROGRAPHS

One of the most important factors effecting a successful photomicrograph is the final condition of the specimen. By the very nature of the photographic process, it is advisable to ensure that the prepared specimen is of the highest quality, since otherwise it will be impossible to obtain a good photomicrograph.

The source of illumination should be carefully adjusted and the correct filter, if required, should be in position. Metallographic plates are usually corrected for use with certain light filters; data are given on the plate packaging. The objective lens and eyepiece most likely to give optimum results are selected, and the camera bellows are set at a distance which produces the desired total magnification. A guide to magnification can usually be obtained from the manufacturers' literature supplied with

the microscope and camera. Alternatively, the microscope may be calibrated for magnification by using a graticule.

When the specimen has been properly positioned, the image should be projected onto the focusing screen and refocused when making the final selection of microstructure to be photographed. Critical focusing is essential. If necessary, a focusing magnifier must be used to ensure this. The microscope must be free from vibrations, since they are obviously detrimental when producing a photomicrograph. If the correct exposure time has been established, a photographic reproduction of the specimen microstructure may be obtained. The microscope adjustments should never be tampered with once the test plate has been produced; to do so would make the predetermined exposure time invalid.

The focusing screen is replaced by the plate or film carrier and the microstructure of the specimen is finally photographed. This should be done carefully to avoid disturbing the focus of the microscope. Once the plate carrier has been secured in position, all that remains is to withdraw the slide cover from the plate or film carrier and operate the camera shutter. Having exposed the plate or film for the predetermined length of time, the slide cover is replaced and the carrier removed from the camera. The photographic plate or film is then processed.

The photographic plate must be loaded into a carrier in the darkroom. To ensure that the plate is inserted correctly, the side of the plate which (in the glow of the safelight) appears to be light in colour and of a glossy texture (the emulsion side) should be uppermost in the carrier.

To load sheet film into a carrier, the emulsion side of the film can be detected by running one's finger along the edges of the film. The film will be the correct way up in the carrier when a serrated portion is felt at the top right-hand side.

PROCESSING

The purpose of film and plate development is to reveal the image recorded on the film or photographic plate. The choice

of developing solutions and fixers depends upon the type of plate or film used, and reference to Table 4 will indicate which developers and fixers may be used.

TABLE 4. CHEMICAL DEVELOPERS AND FIXERS

Developer	Notes
D 61 A	Film and plates as used in microscopy. Use a solution of 1 part developer to 1 part water, and develop for 4 minutes in a dish.
D163	Used for all general papers and document papers. Use a solution of 1 part developer to 3 parts water for general papers, and 1 part developer to 7 parts water for document papers. The developing time is 2–3 minutes.
Velox	Used only for contact paper. Use a solution of 1 part developer to 1 part water. Develop in a tank or dish for 2–3 minutes. May be used to develop 35 mm, 127 and 120 roll film.

A special acting developer may be used in a tank to give a fine grain on a small negative. Examples of these developers are as follows:

Atomal	Manufactured by Agfa.
Microdol	Manufactured by Kodak.
Promicrol	Manufactured by May and Baker.

The manufacturer's instructions for these developers should be closely followed.

Fixers	Notes
Acid hypo fixer	Sodium thiosulphate is used and fixing time is 10 minutes.
Rapid fixer	A special formula, the fixing time of which is 1–3 minutes.
Quick-finish fixer	This is used in an undiluted state and the fixing time is 3 minutes.

Metallurgical Laboratory Techniques

Plates	Notes
0·250	These plates are specially made for microscopy. They are sensitive to a green light, therefore a filter should be used. The exposure time is approximately 20 seconds, and they must be used in conjunction with a No. 2 (red) safelight.

Sheet film	Notes
Commercial Ortho	This type of sheet film is not specially designed for microscopy, but it can be used for most general purpose work. It may be developed in D 61 A, and must be used in conjunction with a No. 2 (red) safelight.

PHOTOGRAPHIC PAPERS

Range	Notes
Velox	This type may be used for producing prints directly from a plate or negative.
Bromide	This type may be used for producing prints by way of an enlarger.

SAFELIGHTS

Type	Notes
00. Yellow	Used only for contact papers.
0B. Lime yellow	Used for bromide paper.
2. Dark red	Used when handling plates which are ortho-chromatic, and are not sensitive to a red light.
3. Green	Used for panchromatic films.

All fixers are used to preserve the impression on the prints or negatives. However, those given above are referred to by their trade name, and it is advisable to consult the manufacturer's literature when choosing a particular fixer.

CHEMICAL STOP BATH

A chemical stop bath may be used to treat the print when transferring it from the developer to the fixer. It is used to avoid contaminating the fixer with developer. The simplest formula for a stop bath is a solution of 2–5% acetic acid and water.

It is important to remember that the laboratory darkroom must be completely free from light intrusion. All chemical developers and fixing solutions should be stored in clearly labelled bottles and frequently checked for quality. Cleanliness is essential in the darkroom, and processing tanks and dishes should be regularly washed and kept free from dust and foreign bodies.

Chapter 2

High Temperatures

THE attainment, measurement and control of high temperatures is a prime need in any metallurgical laboratory. With the exception of zinc, lead and tin and their alloys, all the commonly used metals and alloys melt above 600 °C, and many common heat treatments, particularly those for steels, may involve temperatures up to 1000 °C. For the melting and heat treatment of the more refractory metals such as chromium, cobalt, nickel and molybdenum, even higher temperatures are necessary, and in addition there is a need for accurate control and measurement.

The two main sources of heating power available in the metallurgical laboratory are electricity and gas. Broadly speaking, the latter is only used for small-scale operations at temperatures up to about 750 °C; larger gas-fired installations are perfectly satisfactory, but are used mainly in industry. Electricity, with its absence of fumes, and in particular its easier control and greater heating power, is really much more suitable for the relatively small-scale operations met within the laboratory.

The three methods of electrical heating are known as resistance heating, induction heating and arc-heating. Resistance heating is the most commonly employed for furnaces, which are simple to construct and operate and are capable of reaching 1400 °C or even higher. This temperature is adequate for the majority of operations. It is particularly suitable where very accurate control of temperature is necessary, and the various devices used for controlling temperature will be discussed later in this chapter.

Induction heating and arc-heating have considerable uses,

36

which are not perhaps fully taken advantage of today, but are principally used in the attainment of extreme temperatures, and in cases where rapid heating is desirable.

Furnace Construction

The more usual types of furnace used in the laboratory are the tube, muffle or box furnace (as illustrated in Figs. 2.1 and 2.2) and the pot or crucible furnace. These furnaces are available commercially but with the exception of furnaces of very large proportions they may be made quite easily and cheaply in the laboratory or workshop. It is important to design a furnace with a sufficient power input to raise the temperature of the furnace above the maximum operating temperature. For example, a furnace operating at 1000 °C should be designed to operate adequately at 1100 °C. This increases the working life of the furnace and assists in reducing the excessive time necessary to heat the furnace from cold to the operating temperature. Also it will be possible to maintain control of the temperature more accurately.

COMPOSITION OF HEATING WIRE

For furnace elements operating up to 1100 °C nickel–chromium alloys may be used, while platinum or molybdenum wire may be employed for heating to a temperature of 2000 °C. The commonest medium-duty alloys are the nichromes, a group of alloys based on the composition 80 per cent nickel/20 per cent chromium. These alloys must not be used where there is any risk of contamination by sulphur compounds, which can cause drastic corrosion of the nickel. The use of molybdenum wire, too, in furnaces requires very careful sheathing or some other protection from the atmosphere, since molybdenum oxide is volatile at high temperatures.

CALCULATION OF WIRE SIZE AND LENGTH

The length of wire required to wind a furnace depends in the first instance on the rating of the furnace and on the heat distribution required along the tube. For the normal horizontal tube, a spacing of roughly 10 tpi (turns per inch) will provide a reasonably uniform temperature distribution, and if the turns are spaced more closely at the ends of the tube, heat losses in these areas are minimized. Given this spacing as a rough guide, and assuming that the voltage and the maximum current likely to be taken are known, the exact choice of wire gauge and the approximate length may be obtained from tables of wire resistance issued by manufacturers. For a typical small tube furnace of 1 in. diameter and 18 in. in length, a maximum of 5 amperes is ample to give temperatures up to 1000 °C, assuming there is a reasonable degree of insulation. Knowing the current and the voltage, the total resistance of the winding may be simply calculated from *Ohm's Law*, which states $V = RI$, where V is the voltage, R is the resistance (in ohms) and I is the current (in amperes). Thus for a maximum current of 5 amperes at 240 volts, the necessary resistance is 48 ohms. The required resistance per foot may then be found by dividing the length of the winding by the total resistance. Using this figure, a wire of the correct gauge with the correct resistance per foot length may be ascertained from the manufacturer's resistance tables. As the resistance of the wire varies with temperature it is important to check that the wire has correct current-carrying capacity. Resistance increases as the temperature rises and decreases as the temperature falls. Should the gauge of wire selected be too small, this may cause rapid oxidation and failure of wire through overheating. Hence, furnaces which use a wire to the maximum rating possible may have to be heated slowly on a low current during the initial stages of warming up. Figures giving the variation of current capacity with temperature are normally included in wire manufacturers' data books. Terminal leads are best wound double or even treble to lower the resistance

and keep the temperature lower; this prevents any damage to the terminals.

The length of wire required, allowing extra for the winding anchor leads, may be calculated from the formula $\pi \times D \times Ti \times L$, where D = the outside diameter of the tube, Ti = the turns per inch and L = the length of the tube to be wound. For the example given earlier, the approximate length of wire required would be $\pi \times 1$ in. $\times 10$ in. $\times 18$ in. $= 47$ ft.

THE TUBE FURNACE

The tube furnace is the simplest type of furnace to construct in the laboratory, and the heating element may be wound in the laboratory or workshop. Commercial replacement windings may also be used, but although muffle furnaces may be constructed quickly using replacement windings, certain disadvantages are involved when they are used for the construction of tube furnaces. The disadvantages include limitations as to the size of the tube, e.g. diameter of the bore and outside diameter, and length and this affects the wattage or heating capacity. Also a variable winding may be necessary for certain applications and in certain circumstances, e.g. if the furnace is to be used in a vertical position.

To construct a tube furnace, the refractory tube which carries the winding may be purchased with either a smooth surface or with a spiral indentation moulded into its outer surface The spiral moulding assists in obtaining a regular spacing of the wire, but with this type of tube it is impossible to use other than a uniform winding along the tube length, whereas it is frequently desirable to space the turns of the winding more closely at the ends of the tube. This compensates for loss of heat over these areas, and ensures a more uniform temperature over the length of the tube.

Winding the tube

The technique for winding the tube is as follows. Two retort stands and a length of round steel bar, of a length sufficient to extend beyond the tube, are arranged on a bench. The retort stands are used to support the steel bar in a horizontal position, at a height comfortable to the worker. Suitable weights are stood on the base of the retort stands to ensure that they do not overbalance under the weight of the tube. The furnace tube is then placed onto the horizontal bar, thus enabling the worker to rotate the furnace tube around the bar with one hand while manipulating the coil of wire with the other.

The first step prior to winding the tube is to make a firm anchorage at either end of the tube to which the winding can be attached. Some furnace tubes have small holes drilled at either end for this purpose, but it is inadvisable to use them to fasten the winding to the tube, since they have a tendency to break away, allowing the winding to unravel itself. A length of the winding wire may be used as the anchor lead, and this is done by winding the length of wire twice around the end of the tube and twisting the two ends of the wire together to form a lead. This operation should also be carried out at the other end of the tube. The wire for the winding should be fastened tightly to the anchor lead at one end, then by carefully rotating the tube, the winding is applied. Sufficient tension, to ensure that the turns of wire do not overlap or touch each other, must be maintained upon the coil of wire during the winding operation. Once the tube has been completely wound, the winding can be fastened to the second anchor lead, making sure that the twisted leads at either end of the tube are long enough to reach their termination points. Because these leads have been twisted together, they will provide a lead with only one-half of the resistance of the single wire, and this will assist in keeping the leads cool when electrical power is supplied to the winding.

Once the tube has been wound, it should be coated with a refractory cement to hold the windings in position, which otherwise tend to work loose due to repeated thermal cycling during
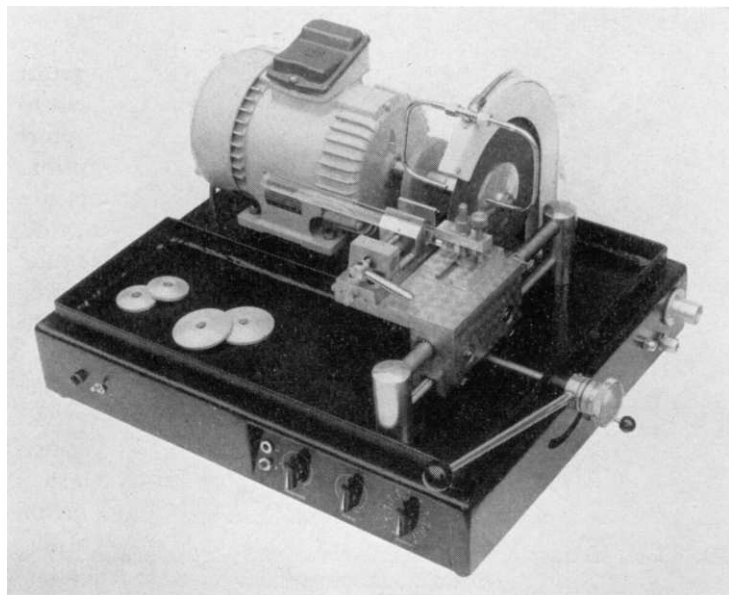
FIG. 1.1. Metallurgical cut-off machine. (Courtesy of Thorn Electronics Ltd.)
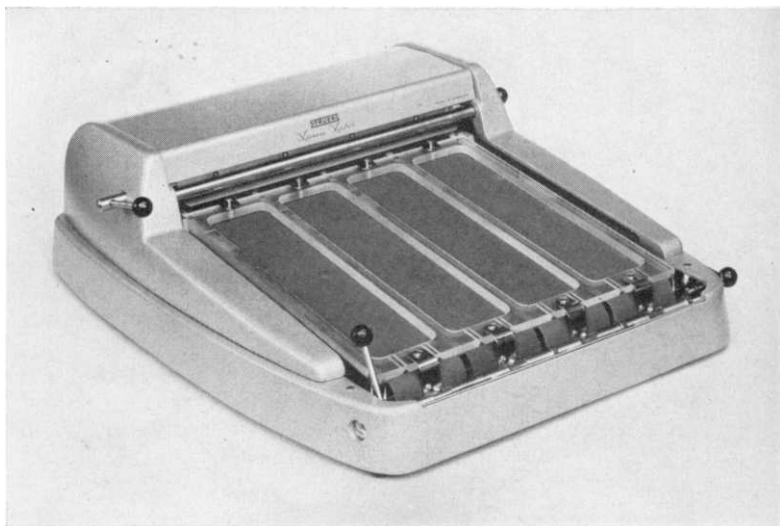
FIG. 1.3. Apparatus for wet grinding specimens. (Courtesy of H. Struers Chemiske Laboratorium.)
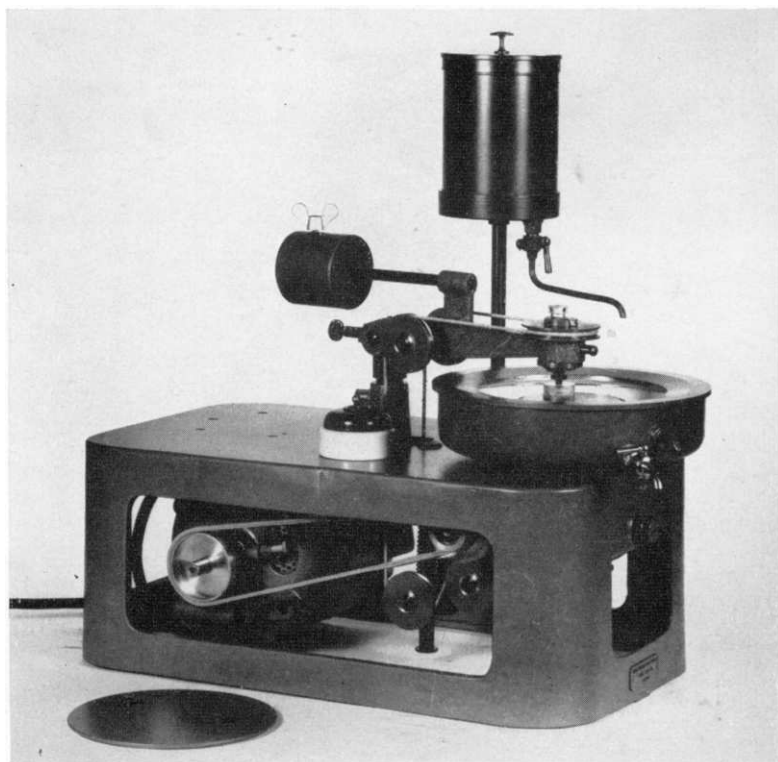
FIG. 1.5. Metallurgical polishing machine. Polishing may be carried out automatically by using the specimen retaining arm. (Courtesy of Vickers Instruments Ltd.)
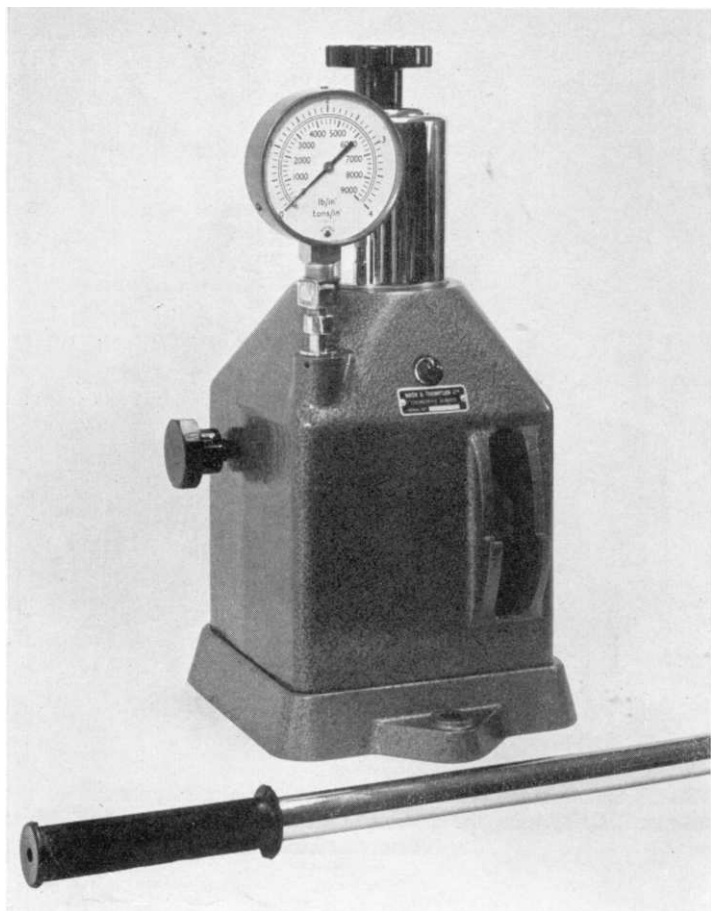
FIG. 1.6. Metallurgical mounting press. (Courtesy of Thorn Electronics Ltd.)

Fig. 1.13. Apparatus for electropolishing specimens. (Courtesy of H. Struers Chemiske Laboratorium.)
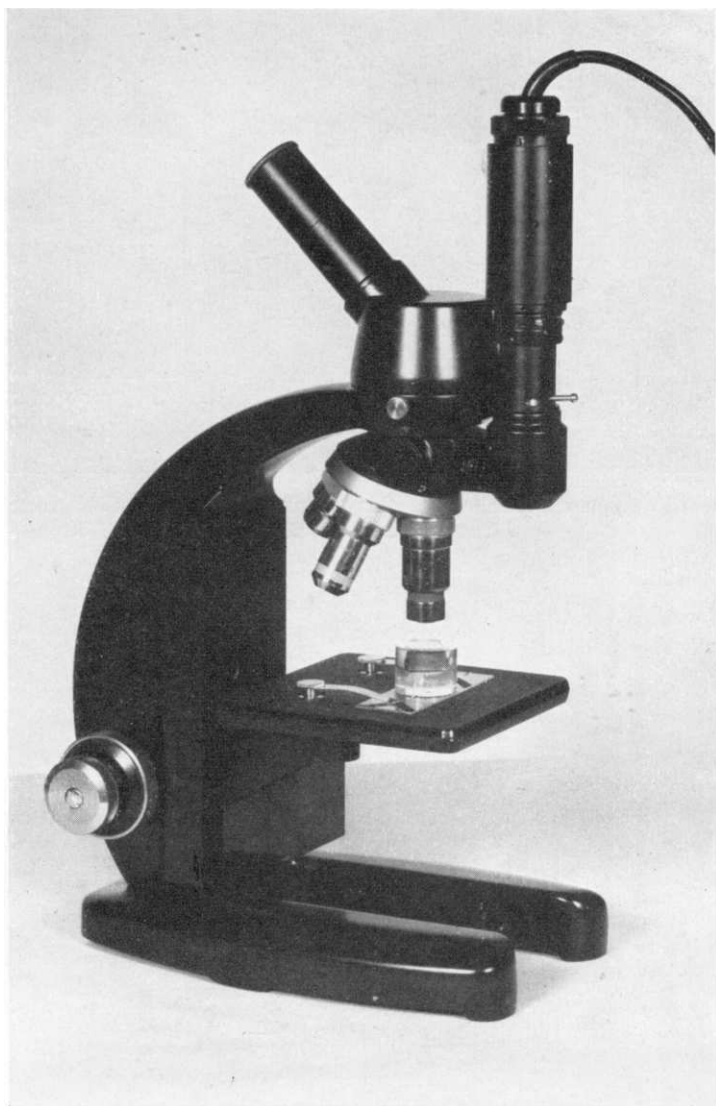
FIG. 1.16. Standard bench type microscope. (Courtesy of Vickers Instruments Ltd.)
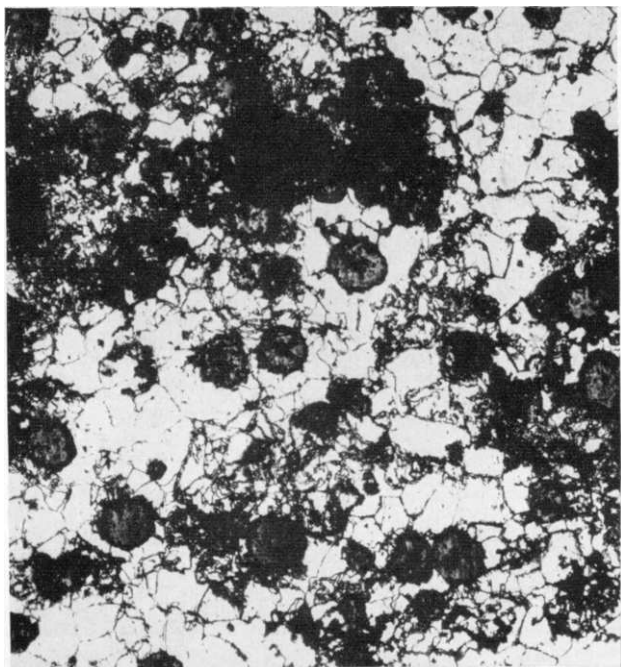
FIG. 1.18. Examples of imperfections on specimens which have not been correctly prepared. These imperfections also appear on specimens in daily use, and the laboratory worker must be able to recognize them. (a) Rust marks on the surface of a ferritic cast iron specimen.
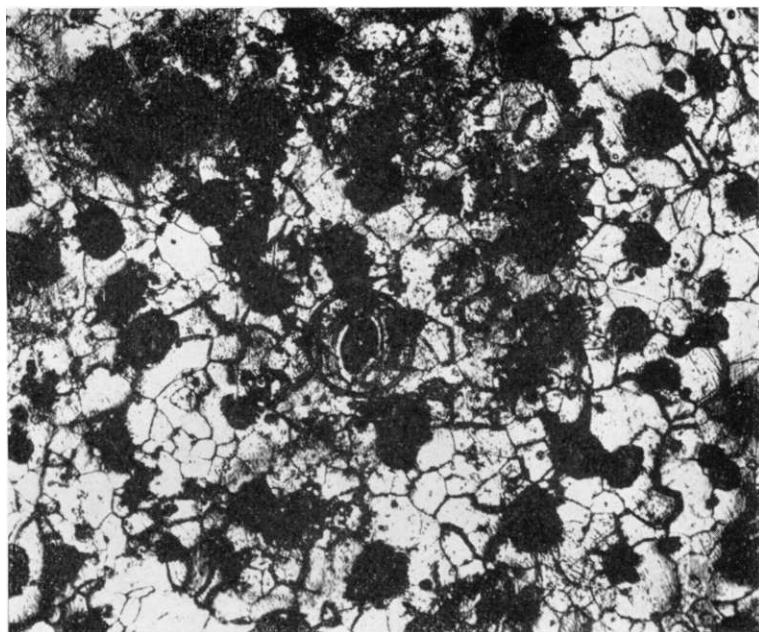
FIG. 1.18. (b) Moisture stain on the surface of a specimen.

FIG.1.18. (c) Dust adhering to the surface of a specimen.
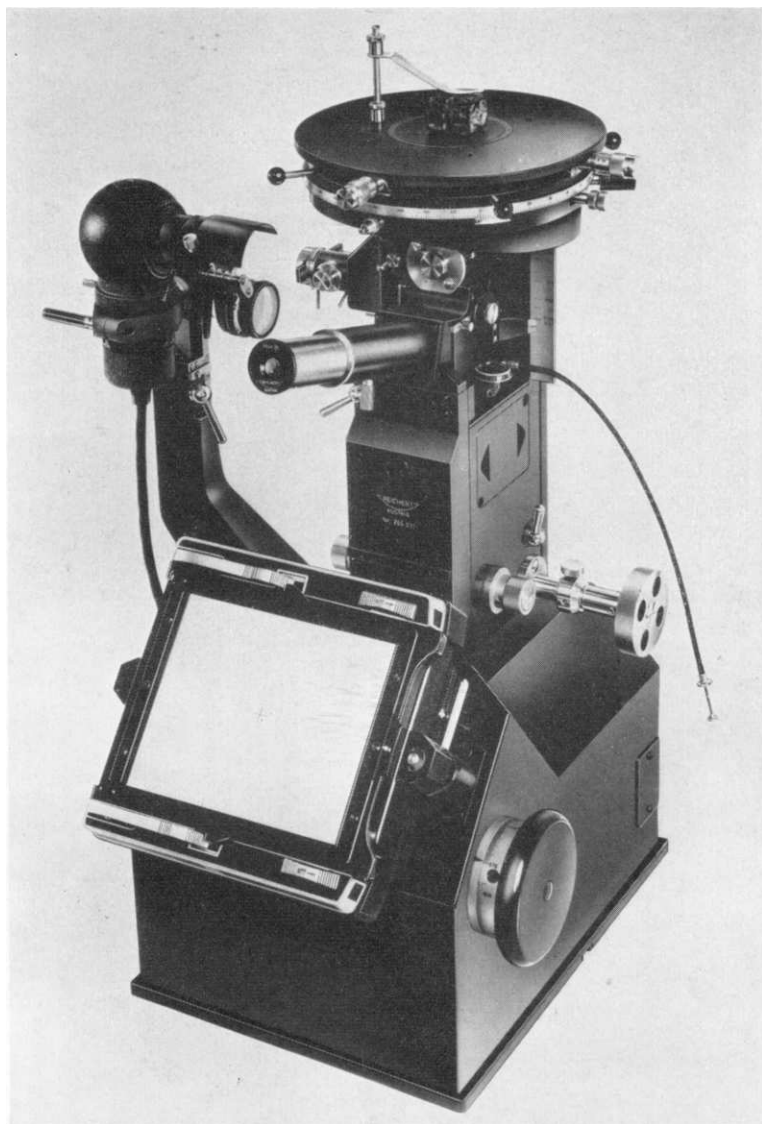
FIG. 1.19. The Reichert MeF universal microscope. (Courtesy of Reichert Ltd.)
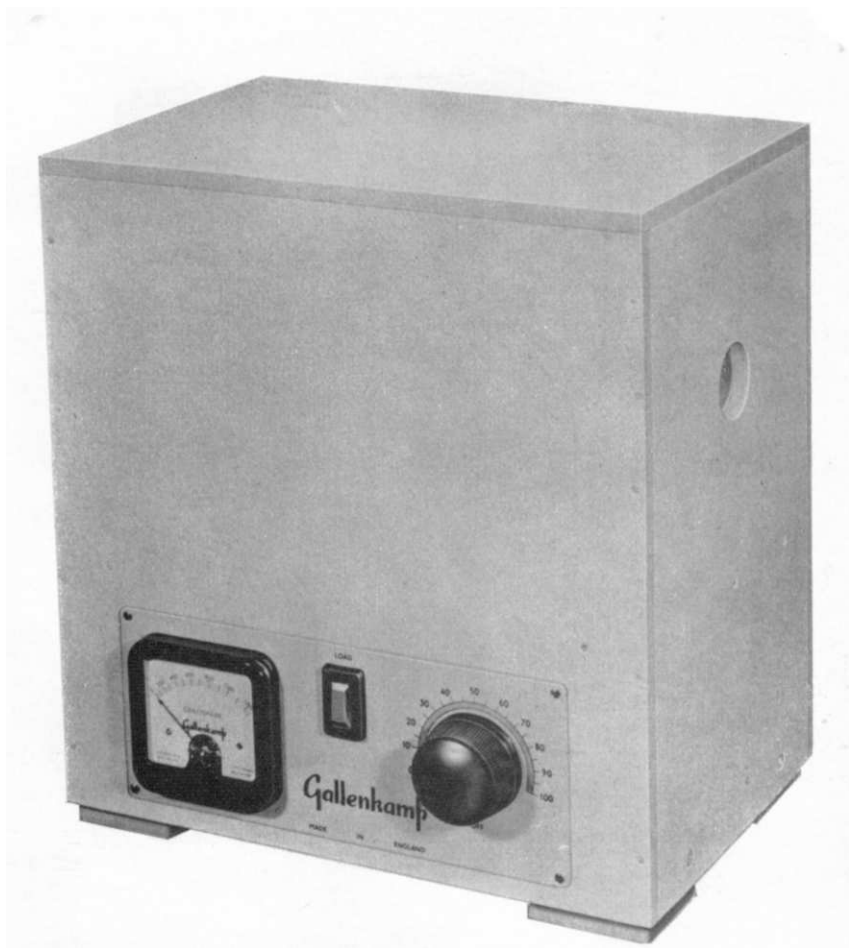
Fig. 2.1. Tube furnace. This furnace is capable of operating in the temperature range 0–1200°C. (Courtesy of Gallenkamp Ltd.)
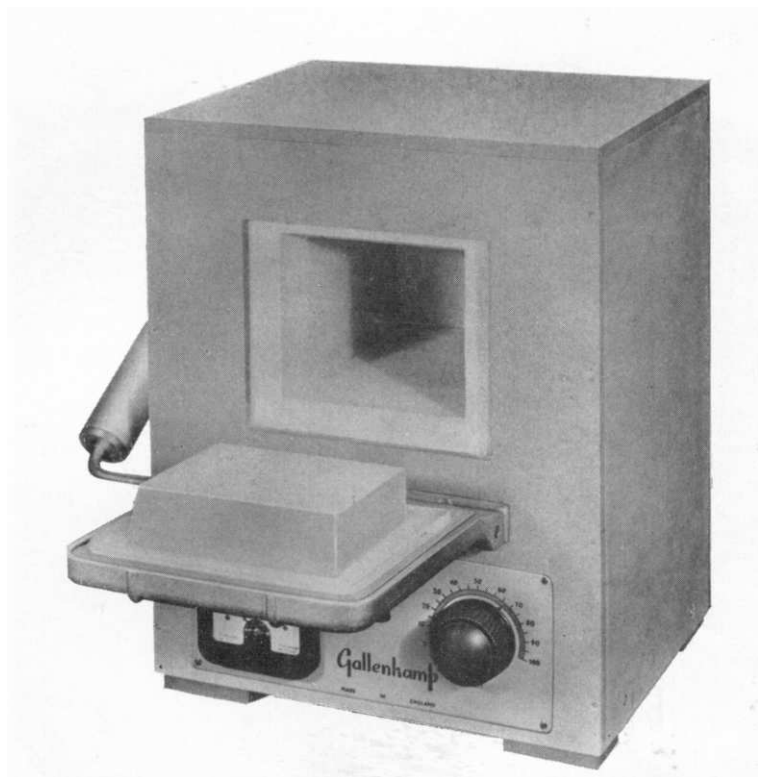
FIG. 2.2. Box or muffle furnace. This type of furnace is capable of being heated to 1200°C. (Courtesy of Gallenkamp Ltd.)
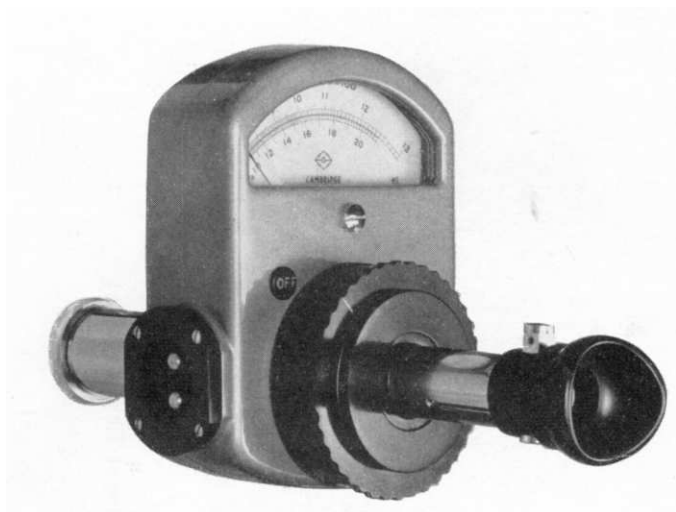
FIG. 2.6. Optical disappearing filament pyrometer.
(Courtesy of Cambridge Instrument Co. Ltd.)
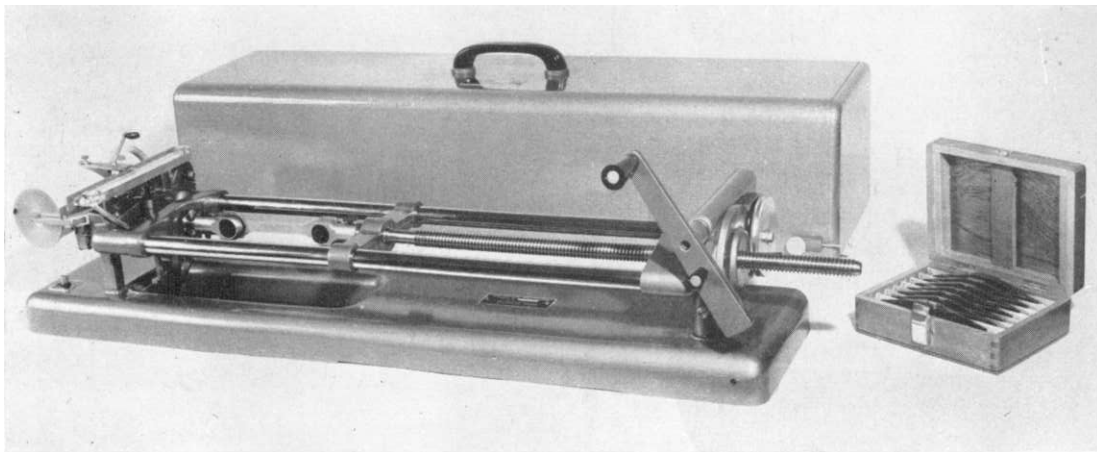
FIG. 4.7. Hounsfield Tensometer. (Courtesy of Tensometer Ltd.)

FIG. 4.16. Hounsfield balanced impact machine. (Courtesy of Tensometer Ltd.)

FIG. 4.24. Vickers pyramid hardness-testing machine. (Courtesy of Vickers-Armstrongs Ltd.)

the life of the furnace. The cement also ensures that the winding cannot react with the insulating material. Several grades of refractory cement are available, and the choice depends upon the temperature at which the furnace will be required to operate. The cement, which may be mixed with water to form a thick paste, is applied to the tube whilst it is held between the retort stands. The cemente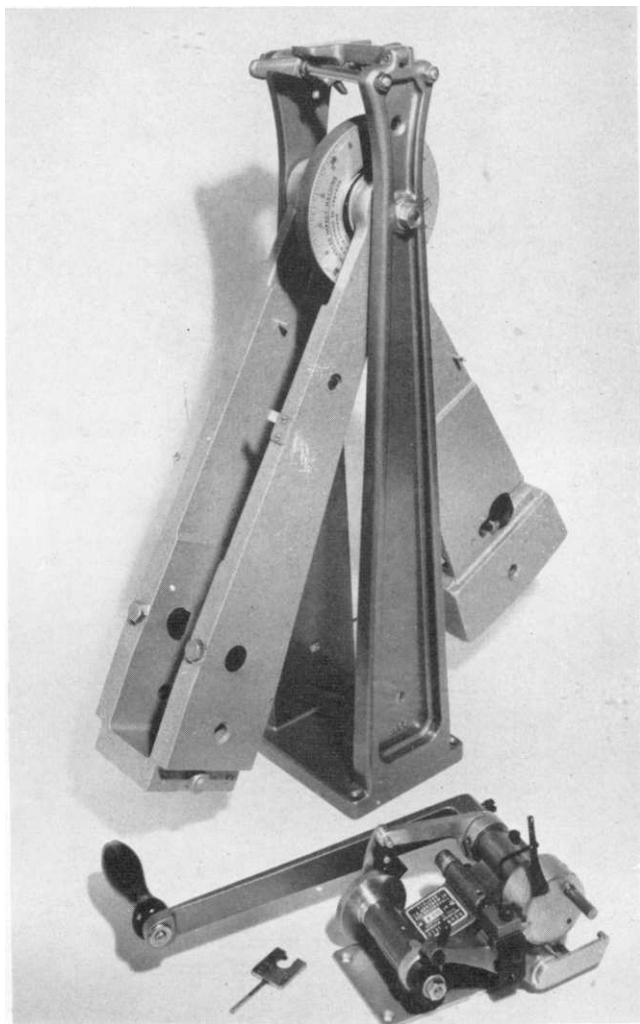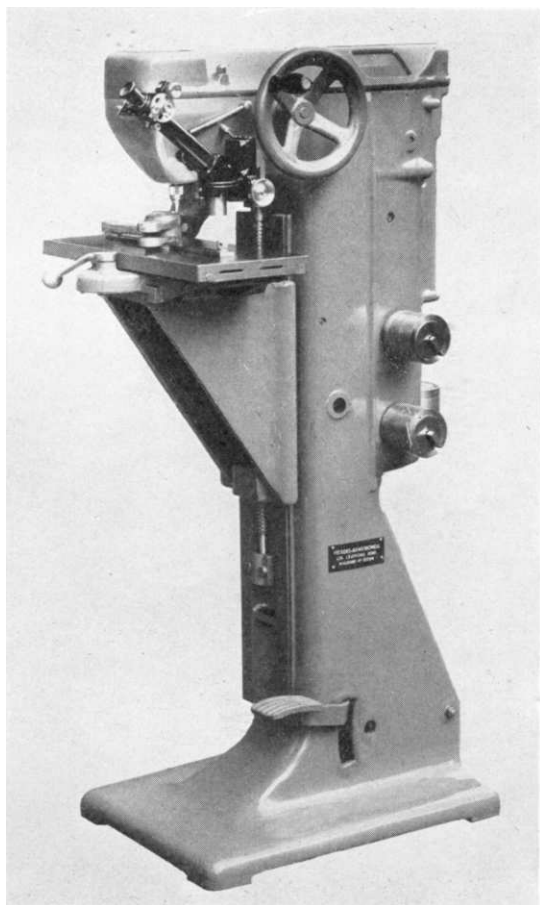d tube is then left to dry out at room temperature, final drying and curing being carried out by heating the tube **slowly** to its operating temperature.

<p style="text-align:center">SUPPORTING THE TUBE</p>

The next step in the construction of the furnace is to manufacture the supports for the tube. These may be made from cement–asbestos board, of which there are various grades and types. One type, "Sindanyo", has been found to be softer and not quite so susceptible to cracking when subjected to prolonged high temperatures. It may be sawn, filed or machined quite easily without adverse tool wear. The simplest shape for the end supports is circular. The circular pieces of board should be of a suitable diameter to allow room for an adequate amount of insulating material when the outer cover of the furnace is fitted.

Four pieces of board are required to form the end supports, and two of them should have a hole bored centrally, in keeping with the dimension of the outside diameter of the tube. Due allowance must also be made for a slight expansion of the tube. The remaining two pieces should be bored centrally to the dimension of the bore of the tube. Four equally spaced holes, $\frac{1}{2}$ in. in diameter, should be drilled in each piece of board to accommodate the threaded tie rods which are necessary to support the end pieces. Provision should also be made in one pair of end supports for fitting the furnace lead terminals.

EXTERIOR FINISH

To complete the construction of the furnace, an outer cover must be provided; thin duralumin sheet is a suitable material. Very thin sheet approximately 0·015 in. thick may be wrapped around the furnace by hand, while thicker material is best shaped by rolling it in a rolling machine to the desired dimen-
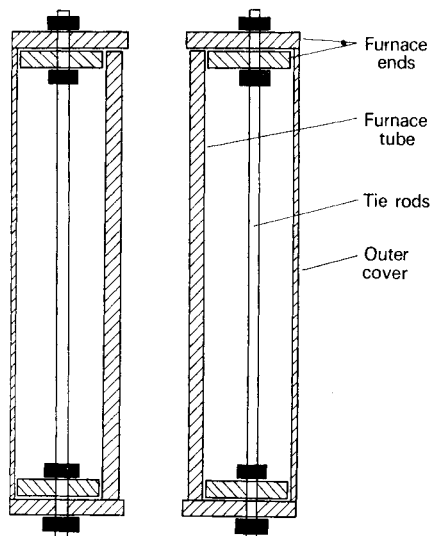
FIG. 2.3. Assembling the furnace. The lower furnace end should be placed in position. The tube and outer cover may also be positioned and the insulation poured until level with the top furnace end. Care must be taken to keep the tube upright.

sion. Having formed the sheet material into a cylinder, the furnace is then assembled. One pair of end supports with the tie rods locked into position are stood vertically on the bench. The wound and cemented tube should be carefully placed in position, ensuring that it engages correctly into the hole bored in the end support. The cylinder of duralumin is then placed in

42

position, and a length of string or wire fastened around the top and bottom to retain the shape of the cylinder. Where the two edges of the cylinder overlap, a series of equally spaced holes are drilled down its length, to accommodate rivets or screws and nuts. These replace the string, and at this stage the cylinder is filled with insulating material, ensuring that the tube is held perfectly vertical. The insulating material should be packed to the top of the cylinder, and the leads from the winding connected to their respective terminals. The remaining end supports are then placed in position and the tie rods tightened. Figure 2.3 illustrates the method of assembling the furnace.

It should be emphasized that there must be a slight movement of the tube to allow for expansion, and adjustment of the end supports on the tie rods will ensure this. The furnace should be tested to check the resistance of the winding, and for possible errors in construction. Water-cooling may be provided, especially at the exit ends of gas train furnaces and furnaces used in a vertical position, to help keep these areas cool. Whether or not the furnace has been designed to operate in a horizontal or vertical position, a stand should be provided to carry it, unless of course the furnace is to be built into existing apparatus. A simple stand can be provided by using one of the varieties of aluminium or steel slotted angle that are available commercially. Alternatively, the furnace end supports may be suitably shaped to retain the furnace in position on the bench.

MUFFLE FURNACES

Muffle furnaces are useful in the laboratory for heat-treating specimens in small quantities. A furnace may be more easily constructed if a replacement winding is used, but the winding should be carefully selected to obtain the correct temperature. Construction of the furnace may be carried out as follows. Using cement–asbestos board, a box of suitable size to accommodate the muffle and sufficient insulation is constructed. The front panel of the box should be fitted with a "cut-out", cut to the

inside dimension of the muffle. The bottom of the box is lined with lightweight refractory bricks and the muffle positioned on them. The surrounding area is then filled with a suitable insulating material. Provision should be made in the back of the box for terminals, to which the muffle winding is connected. A piece of cement–asbestos board is screwed to a suitably shaped refractory brick to provide a furnace door, the piece of board facilitating easy insertion and withdrawal.

LEAD AND SALT BATHS

Lead and salt baths may be constructed easily in the laboratory or workshop, and are useful for heat-treating steel specimens. The bath may be made from stainless-steel tube, with a stainless-steel plate welded to the top and bottom. The top plate should be bored to the inside diameter of the tube. Resistance wire insulated with insulating beads may be used for the heating element, but the bath tends to operate more reliably if wound with a "Pyrobar" heating element. These elements are obtainable commercially, but the manufacturer's literature should be consulted to ensure that the element obtained is of a sufficient rating to raise the bath to the desired temperature. For lead baths the "Pyrobar" should be surrounded with a sheet of stainless steel or nickel, to safeguard the element from pieces of lead oxide which may find their way down the side of the bath and burn out the heating element by slagging away its protective sheath. The complete bath assembly may be placed in a cement–asbestos box, on a floor of refractory bricks, and surrounded with a suitable insulating material. Terminals should be provided in the back of the box, to which the heating element is connected, and a suitable lid should be fitted to safeguard the laboratory personnel from excessive oxide fumes. Lead baths are usually operated with a covering layer of granular charcoal in order to minimize oxidation.

The temperature of the lead bath may be measured by immersing a thermocouple, enclosed in a suitable protective sheath,

into the molten lead. When the surface layer of lead oxide is disturbed, e.g. by placing specimens into the bath, the thermo-couple sheath may tend to float. This may be avoided by using a length of welding wire or similar material to anchor the sheath to the cement–asbestos box containing the bath.

GAS FURNACES

Gas furnaces are used in most laboratories, and are useful for melting metals on a small scale. With the ordinary air/gas mixture a range of temperatures up to 900 °C may be obtained, and by enriching the air with oxygen temperatures up to 1500 °C are possible. For melting small quantities at low tem-peratures, a simple muffle furnace over a bunsen burner is ade-quate.

CONSTRUCTION

A gas crucible furnace is easily constructed in the laboratory. Any cylindrical object of suitable material may be used for the furnace–for example, a metal container of a size suitable to
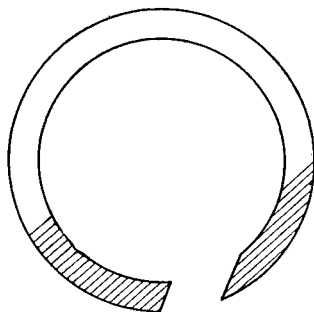

FIG. 2.4. Shows how a hole is provided tangentially in the wall of the furnace for a gas burner. The hole may be made by inserting a plug of cardboard into the wall of the furnace before the refractory material has dried.

accommodate the crucible. The bottom of the container may be lined with a layer of broken pieces of refractory brick covered with sillimanite cement. The sides of the furnace may be built up by ramming sillimanite cement around a wood, metal or strong cardboard plug of suitable size to form the combustion chamber. The plug may be later removed when the cement has been applied to the top of the container. While the cement is moist and workable a hole should be made through the wall of the furnace tangentially and as near to the bottom as possible for the gas burner. This will assist in the elimination of cold spots or areas around the gas burner, as illustrated in Fig. 2.4. A space must be left around the crucible to allow the gas flame to circulate when the furnace is in use. A suitable lid should be provided to retain the heat inside the furnace, but at the same time the lid should either be drilled or slightly raised to allow the burnt gases to escape, and to allow the introduction of a thermocouple. After initial drying of the cement at room temperature, drying may be somewhat hastened by firing the furnace at a low temperature.

Temperature Control

In the field of engineering science, temperature control has in recent years become more advanced than ever before and more especially in large industrial concerns. The purpose of a controller is to control and maintain the temperature of an electric furnace to within very fine limits. It cannot be too strongly emphasized that the accuracy to which temperature is controlled is of the utmost importance, especially during heat-treatment operations. Although the theory of temperature control will not be discussed here, information on this subject is readily obtainable and adequately dealt with in works of reference. However, a few methods for controlling temperatures which are readily available to laboratory workers are now described.

RHEOSTATS AND VARIABLE TRANSFORMERS

The simplest method of controlling temperature is by means of a rheostat, which is a cheap and easily obtainable instrument capable of controlling the power input to the furnace windings. By observing the temperature obtained in the furnace for a series of rheostat settings, a calibration curve for any particular furnace and rheostat combination may be quickly obtained. This method has the disadvantage that it is vulnerable to fluctuations in the mains voltage, but even so, it is possible to maintain reasonably accurate temperatures. Similarly a variable transformer may be used to control temperature, fluctuations being somewhat reduced with this method. Calibration of the transformer must be carried out in the same way as for a rheostat. There is a danger of "overshooting" the desired value because the control is based upon manual settings and adjustment to the controller. Trial and error is time consuming and may cause erratic control, especially should the furnace be in use continually over a long period of time.

THERMOSTATIC REGULATORS

Thermostatic regulators may be used to control temperature, the control being actuated by a simple circuit breaker. Since this operates from the actual furnace temperature, it is independent of mains voltage fluctuations. The simplest method employs a bi-metallic strip, which opens when the preset or desired temperature is reached and closes when it falls below this value. The percentage drop in temperature before the circuit breaker closes is usually determined by the manufacturers of the controller and in some instances the drop in temperature may be quite excessive before this happens. This type of control usually provides a straightforward on/off control, and because of large fluctuations of temperature involved, the controller requires frequent adjustment and is more suited for operations where precise control is of secondary importance, e.g. melting alloys for casting on a laboratory scale. This may be improved by wiring

47

up the controller in such a way that instead of providing on/off control it switches from "on" to a lower current. One of the disadvantages of a straight on/off control is that unless the furnace winding is capable of taking the full input current, it will burn out. Should a furnace winding be of a light gauge wire, then a suitable transformer must be incorporated into the circuit.

BI-METALLIC REGULATORS

Another device is a regulator consisting of a bi-metallic strip surrounded by a small heater winding. The winding is connected in parallel with the furnace winding, and is powered by the same input voltage. The bi-metallic strip may be adjusted to open and close over a range of temperatures by means of a control knob, which actuates a cam. When the power to the furnace is turned on, the furnace winding and the winding around the bi-metal strip are heated until the strip reaches a temperature sufficient to open the contact and the power to both windings is turned off. Since the winding around the bi-metal strip has a smaller heating capacity, it will heat to temperature, and cool, at a faster rate than the furnace winding. Hence the power is turned on and off more frequently. The higher the setting of the control knob, the greater the length of time the furnace is on. If through fluctuations the mains voltage is low, the smaller winding around the bi-metal strip will remain "on" a greater length of time to keep the furnace at the correct temperature.

AUTOMATIC CONTROL

Automatic controllers are available commercially which allow the desired temperature to be preset by the laboratory worker. The controller switches the current off or to a lower value when the temperature of the furnace reaches the preset value, and on to a higher value when the temperature drops, the operation being governed by the cycle of the controller.

This method of automatic control is becoming more widely used in laboratories. The controller usually consists of a galvano-meter used as the measuring instrument (as is the case with most automatic controllers), and a pointer to indicate the temperature. The pointer operates a simple photo-electric system to control the input voltage. The temperature is anticipated by a resistance/condenser circuit incorporated into the control mechanism which is brought into operation immediately the indicating pointer enters a proportioning zone. When the desired temperature has been reached, the control circuit will automatically adjust the ratio of power "on" to power "off" over a given time cycle to maintain the desired temperature. Hence "overshoot" and "undershoot" experience with normal on/off controllers is almost entirely eliminated. Temperatures may be controlled to within very fine limits, the correct temperature being indicated at all times. The intervals at which a controller checks the temperature are important. Thus if the intervals are smaller, better control will be maintained, because there will be less temperature fluctuation. The controller is normally fitted with a device to eliminate the danger of burning out the furnace windings, should the thermocouple fail during usage.

Temperature Measurement

The measurement of high temperature is an essential part of metallurgy as a science, since it is frequently concerned with melting alloys and with crystallographic changes in metals which occur well above temperatures normally encountered in most other subjects. Accuracy of control is dependent on accurate measurement. For example, the temperature at which steels are cast may be above 1400 °C, and in some instances above 1600 °C, copper at approximately 1100 °C, brasses at about 1000 °C, and aluminium alloys at about 600–700 °C. The ordinary thermo-meter is useless for temperatures of this order of magnitude, and

two alternative methods are used. These are (a) direct measurement of the temperature by means of a *thermocouple*, or resistance thermometer, and (b) the measurement of the radiation from the body whose temperature is to be measured, using a *pyrometer*. The second method is chiefly used in measuring the temperature in large commercial furnaces – for example, blast furnaces in steel works. Thermocouples are widely used in the laboratory, since they are easy to handle and may be read quickly.

THEORY OF THERMOCOUPLES

The principle of thermocouples was discovered in 1823 by Seebeck, and is quite simple. If two wires of dissimilar metals are welded or soldered together at both ends, and if a temperature difference is established and maintained between the two junctions, then a current will flow in the circuit proportional to the temperature difference and varying somewhat depending on the two metals employed. An electric current cannot be maintained in a circuit of a single homogeneous metal, however varying in section, by the action of heat alone at any point in the circuit. The thermoelectric effect is due to unequal flow of electrons in each metal and thus causing a current to flow. In a homogeneous metal the heating effects will be equal and opposite on both sides of the point heated, thus cancelling each other out.

THE LAW OF INTERMEDIATE METALS

The algebraic sum of the thermoelectric effects in a circuit composed of any number of dissimilar metals is zero if the temperature is constant throughout the circuit. This law has a corollary which states that if we have three thermocouples composed of metals A and B, A and C, and B and C, respectively, and if the temperature of their hot junctions is T_1 and the temperature of their cold junctions T_2, then $E_{ab} = E_{ac} + E_{bc}$, where $E =$ the e.m.f. produced in any of the thermocouples. From this

relationship, the e.m.f. of any pair of metals can be found over a range of temperatures if the e.m.f.s are known against one metal taken as a standard, and a series of curves may be obtained. In practice, the metal platinum is usually taken as the standard. The sign convention for the e.m.f. is that if the current in a thermocouple flows from metal A to metal B at the cold junction, then A is said to be positive with respect to B.

For most purposes, it is desirable to have a high rate of charge of e.m.f. with respect to temperature, and therefore thermocouples are usually made up of one metal which is positive with respect to platinum, and one metal which is negative. Obviously, however, the choice of metals will also depend to some extent on the range of temperatures which is to be covered, and the resistance of the metals to the conditions prevailing, e.g. the temperature, furnace atmosphere etc. under which the measurements will be made.

THE LAW OF INTERMEDIATE TEMPERATURES

This is the second important law concerning thermocouples, and states that the thermal e.m.f. developed by any thermocouple at temperatures T_1 and T_3 is equal to the algebraic sum of the e.m.f.s produced by the thermocouples at temperatures T_2 and T_3. Concisely, $E(T_3/T_1) = E(T_2/T_1) + E(T_3/T_2)$ where the first suffix is the temperature of the hot junction and the second suffix is the temperature of the cold junction.

For high temperatures (up to 1500°C) and for corrosive conditions, a thermocouple made up of platinum and a 10 per cent rhodium–90 per cent platinum alloy is used. This thermocouple was introduced by Le Chatelier in 1888, and has since remained the standard high-temperature version. It gives good results, but the e.m.f. developed is relatively low, and the components are expensive. Therefore, for general use, under not too arduous conditions and up to temperatures of about 1000°C, base metal thermocouples are used. The commonest thermocouple is the chromel–alumel, which is reasonably corrosion

resistant over this range of temperature. Chromel is an alloy of 80 per cent nickel and 20 per cent copper, whilst alumel is 97 per cent nickel with the balance made up of silicon, aluminium and manganese. Chromel–alumel alloys originated during a search for a metal to replace the iron element in the iron–nickel thermocouple. The alloy chromel was so successful that a substitute for the nickel was found in the alumel alloy. The presence of small quantities of silicon and manganese would appear to be essential, for it was found that although the pure nickel–aluminium alloy stood up to high temperatures, it became brittle with use at lower temperatures. Other base thermocouples frequently used are copper–constantan (55 per cent copper, 45 per cent nickel) and iron–constantan. The practical disadvantage of this thermocouple is that the iron is liable to rust in humid atmosphere. It should be remembered that the e.m.f. of base thermocouples decreases with prolonged exposures to very high temperatures, so frequent recalibration may be necessary.

CONSTRUCTION OF THERMOCOUPLES

Thermocouples may be constructed easily in the laboratory and are usually made to take advantage of the intermediate metal law. The thermocouple wire can be purchased, coiled on a spool, in a wide range of wire sizes. Thermocouples made from base or noble metals may be welded with the flame from an oxygen-acetylene gas torch to form the hot junction. The wire should be twisted together for a few turns to give mechanical strength and the ends fused. Light gauge wire may be joined by spot welding the ends. A stock supply of thermocouples may be produced by these methods, this being especially useful in teaching laboratories, where practical demonstrations using high-temperature equipment and techniques are involved. Should a thermocouple fail to function during the demonstration, it can quickly be replaced.

CALIBRATION OF THERMOCOUPLES

Although the wire supplied commercially usually agrees with the manufacturers' tables of e.m.f.s versus temperature, and may be used without further calibration, thermocouples in regular use should be periodically recalibrated. The simplest method is
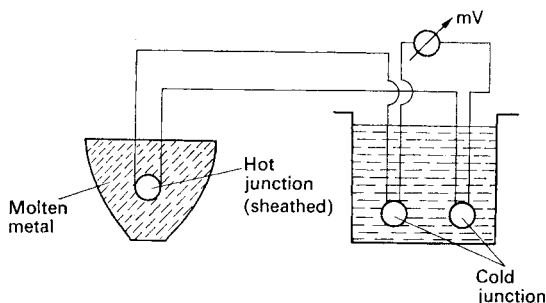
FIG. 2.5. Simple thermocouple circuit. This circuit may be used to calibrate thermocouples; the crucible may be heated over a bunsen burner.

to check the doubtful thermocouple with a carefully calibrated standard thermocouple, and compare the e.m.f.s over a suitable range of temperatures.

An alternative method is to make up the thermocouple as the circuit illustrated in Fig. 2.5, using copper wire to connect the cold junction to a measuring instrument, in this case a milli-voltmeter. The thermocouple may be calibrated by determining the e.m.f.s of the following standards. Boiling point of pure water (100 °C), melting point of tin (232 °C), melting point of lead (327 °C) and the melting point of zinc (419 °C). The metal may be melted in a small crucible over a bunsen burner, the surface of the metal being covered with a layer of charcoal to prevent oxidation. The thermocouple should be placed in a sheath and inserted into the metal, so that the hot junction is well below the surface level. As the metal cools, readings of time are taken at short intervals as the millivoltmeter reading drops. When the

53

metal is solidifying, the millivoltmeter pointer will remain stationary. This reading will correspond with the freezing temperature of the metal, at a known cold junction temperature. Of course, the thermocouple calibrated by this method can only be used to measure temperatures from 100°C to 419°C. A graph may be plotted from the readings of e.m.f. versus temperature to form a calibration curve for the thermocouple. Points plotted on the graph do not lie in a straight line, but as a very slight curve. Table 5 gives the standard temperature list of temperatures most often used, since pure metals always melt at constant temperatures.

TABLE 5. STANDARD TEMPERATURES

Freezing point	°C
Pure mercury	– 38·9
Tin	231·8
Lead	327·3
Zinc	419·4
Antimony	630·5
Silver	960·5
Copper	1083·0

COMPENSATING LEADS

Compensating leads are wires which, although of a different composition than the thermocouple wire, have the same thermo-electric constants. They are used when the temperature indicator or controller is situated at some distance from the furnace, when it would be too expensive to use the thermocouple wire over long distances. Compensating leads are unable to withstand high temperatures, hence any temperature involved near these leads must not be allowed to exceed 100°C. Above this temperature there is a possibility of damaging the leads, and the e.m.f. versus temperature relationship will not match that of the thermocouple.

COLD JUNCTION CORRECTION

When measuring temperature, the possibility of variation in the cold junction temperature must be taken into account, and whenever it varies from 0 °C compensation must be made. If the e.m.f. versus temperature curve is linear over a temperature range, as is the case with base metal thermocouples, the temperature at the cold junction should be added to the apparent temperature. When the temperature is not linear, as with noble metal thermocouples, the e.m.f. at the cold junction and **not** the temperature should be added. The e.m.f. readings in millivolts must be converted to degrees of temperature using suitable conversion tables. It should be noted that a millivoltmeter may be calibrated directly to give a temperature reading scale in degrees of temperature, and in this case is referred to as a temperature indicator.

Should the thermocouple wires be connected directly to the terminals of a temperature indicator or millivoltmeter, then these terminals are the cold junction of the thermocouple and corrections as indicated above should be made for accurate work. Where the cold junction is not an integral part of the measuring instrument, the cold junction may be placed into a flask containing pieces of broken ice, to give a reasonably constant temperature of 0 °C. Alternatively the cold junction of the thermocouple may be placed into boiling water, the wires being contained in a test tube or similar container. This method may be subjected to small variations due to barometric pressure. In both instances the temperature of the cold junction may be measured by an accurate mercury-in-glass thermometer. If very accurate temperature measurement is of no importance, the cold junction may be determined at room temperature. Temperature indicators or measuring instruments are usually fitted with an automatic cold-junction corrector. This avoids making repeated adjustments to the indicator to compensate for variations in room temperature. The mechanism for automatic cold-junction correction is fitted inside the indicator and usually consists of a bi-

55

metallic strip which moves the pointer indicating temperature at the hot junction sufficiently to compensate for variations in room temperature. When using indicators with automatic cold-junction correction, great care must be taken to ensure that the thermocouple is the correct type for the cold-junction compensation provided. Some types of instruments not equipped with automatic compensation have a scale with an .adjustable zero. If this type of instrument is employed, the pointer normally set at zero may be set to read room temperature on open circuit, and the addition of the temperature at the cold junction will automatically follow. Observation for any variation in the room temperature should be made, and further adjustment to the indicator carried out.

The usage of thermocouples may be summarized as follows. In the laboratory thermocouples are often used bare. Under these conditions they respond quickly and provide precise measurement of temperature. However, should the thermocouple be used under conditions that may cause it to deteriorate, e.g. permanently in use in a furnace or, as mentioned earlier, immersed continuously in lead baths, it should be protected by inserting it into a closed-end protective tube made from alundum, stainless steel, or high chromium–nickel alloy. Other refractories or metals that are capable of withstanding the furnace conditions are quite satisfactory for thermocouple protection, and when they are used in gas-fired furnaces, they should be installed directly in the path of the flame. Thermocouples should always be installed into electrical resistance furnaces so that they will measure the average temperature. Errors in temperature measurement can usually be attributed to one of the following:

(a) Short circuit of the thermocouple wires.
(b) A broken hot-junction connection.
(c) A loose cold-junction connection.
(d) Corroded thermocouple elements.
(e) Incorrect immersion into the heated medium.
(f) Cold-junction temperature not compensated for.
(g) Parasitic e.m.f.s or stray currents.

Table 6 lists the more common thermocouples used in laboratories.

TABLE 6. COMMON THERMOCOUPLE MATERIALS

Material	Maximum temperature (°C)
Platinum	1500
Platinum–13 per cent rhodium	1500
Platinum–10 per cent rhodium	1500
Chromel–alumel	1100–1300
Iron–constantan	950
Copper–constantan	400

TEMPERATURE-INDICATING CRAYONS

Crayons that are sensitive to temperatures may be used to determine whether a heated surface is above or below a particular temperature. The crayons are available as a series, suitably marked with their melting points, usually ranging from approximately 50 °C to 850 °C. During usage the heated surface is simply stroked with each of a particular range of crayons to leave a chalk-like mark. The temperature is indicated by the crayon mark between the one which remains solid and the one that has turned into a liquid smear.

PYROMETERS

Quite often it is necessary to measure temperatures under conditions beyond the range of the ordinary thermocouple. If this is the case, or if it is not possible to place a thermocouple in contact with the object whose temperature is to be measured (as might be the case in an induction furnace), a pyrometer may be used. The pyrometer does not come into direct contact with the hot body, but measures (a) the total radiation or (b) the intensity of light of a given wavelength emitted by the hot body.

The first of these involves the use of a total radiation pyrometer, its use being secondary compared to the more common optical disappearing filament pyrometer illustrated in Fig. 2.6. This type of pyrometer may be regarded as a simple form of photometer in which the intensity of light of a given wavelength emitted by the hot body is compared with that given by a standard hot body within the same narrow waveband. Within the instrument, an M-shaped filament of a specially constructed lamp is placed at the focal point of an objective, which in conjunction with an eye-piece forms an ordinary telescope for superimposing on the lamp filament an image of the source of heat. A red glass fitted into the eyepiece usually passes approximately monochromatic light of a wavelength of 0.65μ.

OPERATION

To take a reading of the hot body, the current through the lamp is varied by means of a rheostat until the tip of the fila-ment is of the same brightness as the hot body being examined. Thus it becomes invisible against it; in other words, the filament disappears. The temperature of the hot body is read directly on an indicator which is calibrated in degrees centigrade. The rheo-stat should be turned slowly from the "off" position, in a clock-wise direction. The pointer indicating temperature will first deflect to the left of the scale, but as the temperature of the lamp filament increases it will return to the zero position, and then proceed up the scale. It should be noted that the lamp in the pyrometer is battery operated. (The author has knowledge of an optical pyrometer of this type being plugged directly into the mains supply, with disastrous results.) For this purpose a 2·5 volt rechargeable battery is supplied by the manufacturers. It should be noted that the battery contains an electrolyte which is alkaline, and **not** acid, and should be replaced approximately every 2 years. During the course of usage, any loss of electrolyte, due to evaporation, should be made good with distilled water.

The pyrometer is calibrated for black body conditions. If the

temperature of a furnace is taken by observations through a small sight hole in the furnace door, then these readings can be relied upon to be of a high degree of accuracy, because the interior of the furnace closely approaches black body conditions. Pyrometers are used for measuring temperatures between 700°C and 3500°C, usually on a single scale or a range of 700–1600°C and a double range, e.g. 700–1300°C and 1000–1800°C upwards. If a double range instrument is used, the lever that selects the range, situated at the back of the instrument, must be set to its appropriate position.

EMISSIVITY CORRECTION

Radiation from all materials, at the same temperature, is not the same. There is, however, a maximum amount of radiation from a surface at a given temperature. Such a surface may be referred to as a "full radiator". As mentioned earlier, the disappearing filament pyrometer measures the "brightness" of a temperature, and is calibrated in terms of black body temperatures. For many applications of temperature measurement, the hot body to be measured will not be a "full radiator". Hence the reading on the pyrometer scale, when the filament is matched, will not be the true temperature of the hot body.

Emissivity is the term used to define the amount of radiation from a surface other than a "full radiator". This may be defined

TABLE 7. EMISSIVITY FOR SOME TYPICAL MATERIALS ($\lambda = 0.65\ \mu$)

Material	Emissivity $\varepsilon\ \lambda\ E$
Copper (unoxidized)	0·02
Copper (oxidized)	0·70
Carbon	0·90
Iron (unoxidized)	0·37
Iron (oxidized)	0·85
Cast iron	0·95
Steel (liquid depending on the composition)	0·5

as the ratio of radiation emitted at a given temperature to that emitted by a "full radiator" at the same temperature. It is not possible to give the emissivity values for all the various materials, since much will depend upon the individual working conditions, but Table 7 may serve as a guide in estimating the true temperature of the hot body.

CARE AND MAINTENANCE

To maintain the accuracy of the pyrometer some simple servicing is necessary, the chief danger being the fine dust so often met under conditions of use in the laboratory. The presence of a film of dust on the lenses, or on the cover glass inside the objective tube, can impair the efficiency of the instrument, causing errors in the readings of the true temperature. Similarly finger marks or dirt on the glass envelope of the pyrometer lamp will also cause errors. The lenses and all the glass surfaces should therefore be carefully wiped with a lens brush or tissue, but the lamp unit should never be removed, unless dirt is actually visible through the eyepiece. Should there be any dirt on the track of the rheostat, the needle, indicating the hot body temperature, will "kick" when the rheostat is turned. When cleaning a rheostat, it should first be ensured that the battery has been disconnected. Old and dirty grease is then removed from the wire tracks and the moulding, by washing them in trichlorethylene, carbon tetrachloride or any other suitable solvent, and after washing a thin smear of mineral oil should be placed on the wire tracks. Should the needle stick at any point during operation, the pyrometer should be returned to the manufacturers for attention. On no account should the movement be tampered with, since its removal is a delicate operation requiring special skills, and failure to observe this factor will undoubtedly alter the calibration of the pyrometer. The possible cause of a sticking needle is that the pivots in the movement may have been damaged, or dirt may have entered the movement.

Induction Heating

This method of heating is used more rarely in the laboratory, principally since it involves the need for expensive and elaborate equipment. A second drawback is that it is much less susceptible to fine control than resistance heating. On the other hand, extreme temperatures may be attained very rapidly. Also since there are no electrical connections and no actual contact between the charge to be heated and the heating coil, it can be useful as a source of heat in vacuum chambers, or in places where the windings of a resistance furnace would be impracticable. Since control is difficult, it is of more value in melting operations than in heat treatments (especially is this so in industrial concerns), which lie within the range of resistance heating anyway.

Current is induced in the charge by a high-frequency alternating current in a surrounding coil; this can be a source of electrical problems. Once the current is induced in the charge, the mechanism of heating is exactly the same as in the resistance furnace. That is to say, the heating power is given by RI^2, where I is the induced current and R is the electrical resistance of the charge. However, calculation of the resistance of the charge is complicated by the fact that the induced current only flows in the surface of the charge, the depth of this current-carrying skin being dependent on the resistivity of the charge and the frequency of the current. It should be noted that the **size** of the charge plays an important part in determining both the rate of melting and the maximum possible temperature attainable in the furnace.

It has been found that the most favourable heating conditions for a cylindrically shaped furnace are obtained when $d \simeq 4D$, where $d =$ the diameter of the charge, and D is the depth of the current-carrying skin of the charge. The optimum induced current, which may not always be obtainable, is given by

$$\frac{I^2}{I_0^2} = \frac{16n^2h^2}{5}$$

where $I =$ the induced current, $I_0 =$ the current in the induction

61

coil, n = the number of turns of the coil, h = the height of the charge cylinder. Should $d = 2D$, the value of I^2/I_0^2 is less $(2n^2h^2)$, and for values of d less than $2D$, the power is insufficient to reach high temperatures. Under these conditions higher frequencies must be used.

A wide variety of techniques is possible for induction heating, and basically these consist of an induction coil, a container for the charge, and some form of insulation or shield between the coil and the charge. Multi-turn coils having approximately five to thirty turns, and in some cases even more, are suitable for most laboratory operations. The coil is usually made from copper tube, approximately $\frac{1}{4}$ in. in diameter, to allow water to circulate around the coil for cooling purposes (the coil itself is not a direct source of heat, but suffers intense radiant heat from the furnace charge). With some types of large high-frequency generating coils, having large inductance, more turns are required. Hence it is sometimes necessary to flatten the copper tubing so that a sufficient number of turns may be obtained into the desired height of the cylinder. The coil may be mounted on a cement–asbestos board. It is important to avoid the use of conducting material as far as possible in the support structures of the furnace, since they may also become heated by induced currents when the furnace is in operation.

Usually high frequencies are better for heating small quantities of metal with very high electrical conductivity. When lower frequencies are used, any difficulties in obtaining sufficient heat to melt the charge may be overcome by using conducting radiation shields to retain heat. The shields are used in place of other forms of insulation. For melting purposes graphite crucibles may be used, because for most sizes of charge, graphite has a specific resistance, this being more favourable for induction heating than the metal being melted. Insulation is absolutely necessary to prevent excessive heat losses, as it is in other methods of heating. Carbon black is suitable, as it will not decompose or become electrically conducting at the high temperatures usually involved.

Electric Arc-heating

Refractory metals may be successfully melted by arc-heating. Melting is usually carried out by striking an arc between a water-cooled tungsten electrode and the charge to be melted. A water-cooled copper crucible is used to contain the charge, and when the arc is struck, the charge forms its own crucible by the action of the cold copper crucible retaining a thin layer of metal, frozen to the sides and bottom. The arc is struck above the charge, and only the top layer melts; the remainder in contact with the cold copper remains solid. To completely melt the charge, it is usually inverted and the bottom is melted in a second operation. By adding more to the charge during the operation of the arc, and melting in layers, a large ingot may be obtained. The melting operation is usually carried out in a chamber in an inert atmosphere, e.g. argon, applying a direct current of several hundred amperes for a small furnace, to produce approximately 1 lb. of molten metal. The charge is usually the positive electrode, being hotter than the tungsten electrode. Actual casting by this method is virtually impossible because of poor control over homogeneity when producing alloys. This is because the melting of the charge, being carried out a little at a time, segregates the alloying elements in the ingot being produced.

Refractories

Refractories are non-metallic materials which will withstand very high temperatures without deforming, melting or suffering a change in composition. For relatively low temperatures and small-scale usage, Pyrex glass is quite satisfactory in many instances, but it softens at about 500 °C, and is not normally available in such useful dimensions as the commoner refractories.

The most important aspects of a refractory are that it should be able to provide thermal insulation, support metallic windings for electrical resistance furnaces and be able to contain solid or liquid

metals without entering into any chemical reaction with them. The final choice of a suitable refractory is usually governed by the following properties: melting point, thermal conductivity, mechanical strength and electrical conductivity at high temperatures, thermal coefficient of expansion, thermal shock resistance, resistance to spalling, and chemical inertness to liquid metals and the surroundings in which the refractory is used.

COMMON REFRACTORIES

Alumina

Alumina is the most widely used refractory, being available commercially in the form of crucibles, tubes, rods and muffles. It is available in a high state of purity, and has a high melting point, good resistance to thermal shock and low electrical conductivity at high temperatures. It is the last of these properties which makes it an excellent material for the construction of furnace tubes and muffles upon which a metal winding may be wound.

Clay–graphite

Clay–graphite is a material used for crucibles when melting non-ferrous metals. It is available as crucibles in a wide range of shapes and sizes and, as the name implies, is a combination of graphite bonded by clay. Together they couple the high thermal conductivity and resistance to oxidation of pure graphite with the toughness, resistance to thermal shock and strength of clay. This material has largely replaced the once common fire-clay crucibles which were used when melting non-ferrous metals.

Magnesia

Magnesia, because of its high melting point, chemical stability and cheapness, is one of the most useful refractories, even though it is usually unavailable in the form of high-purity crucibles. Hence crucibles obtained commercially usually contain large quan-

tities of fire-clay, but because of their apparent resistance to slagging from ferrous oxide, they are useful for melting iron or steel. It has been found that these crucibles are susceptible to cracking because of the strains involved in the rapid heating and cooling rates of induction heating, and that these cracks usually fail to cause serious harm if the crucible is surrounded and retained in position by a layer of tightly packed refractory powder. This presumably seals the cracks and prevents leakage.

Silicon carbide

Silicon carbide is mainly used for muffles for electric and gas furnaces. Because of its semi-metallic nature it has a much higher thermal conductivity than most refractories, and is used as an economic material for heating rods in high temperature furnaces up to 1400°C. Its only drawback in this instance is a slow ageing at high temperatures which results in an increased resistance which effectively limits the useful life of heating elements. Silicon carbide products are usually glazed to prevent oxidation during use, and the degree of glazing varies, so care must be taken to ensure that the correct grade is selected for use.

Zirconia

Zirconia should probably be used more often as a refractory material. It has a very high melting point coupled with a low coefficient of thermal expansion, which makes it very resistant to thermal shocks. This is particularly beneficial for high-frequency melting where rapid heating and cooling rates are prevalent.

Mullite

Mullite is available commercially as a refractory brick, and also in tube form, being ideally suited for general furnace construction, e.g. in lining or building furnace walls. It possesses good strength near its melting point, but because of its low

thermal conductivity it should be used with a secondary insulation. Mullite bricks may be divided into two classes: a heavier brick, more suited to industrial purposes, and a lightweight brick. The second of these is extremely useful in the laboratory, mainly because of the ease with which it may be cut and shaped using conventional hand tools. For furnaces designed to operate at temperatures well above 1600 °C, the lightweight brick may be faced with alumina. Because refractory bricks are manufactured to have a variety of limiting useful temperatures, care should be taken to select a brick that will withstand the desired working conditions.

REFRACTORY CEMENTS

Refractory cements may be obtained as a dry powder which should be mixed with water, to a consistency of thick paste, before application. Because of the many types of cement available, the manufacturer's literature should always be consulted to ensure that a suitable cement is obtained.

ALUMINA CEMENT

Alumina cement may be used to hold resistance windings in place on alumina tubes, this being more suitable than magnesia cement. Care should be taken to dry the cement out **slowly** and thoroughly before raising the winding to its working temperature. Magnesia cement can be used to line clay–graphite crucibles to prevent carbon contamination when ferrous metals are melted in them.

SILICON CARBIDES

Silicon carbide cements are useful for repairing silicon carbide muffles, while sillimanite cement can be used to line furnaces,

e.g. a liner for a gas furnace. Sillimanite converts to mullite when fired, and can be used quite satisfactorily up to 1500 °C while possessing excellent resistance to thermal shock.

INSULATION MATERIAL

Insulation is used to reduce heat transmission, this being effected by providing a layer of material of low heat conductivity between the internal hot part of the furnace and the outer case, so preventing the escape of heat. The temperature for which the insulating material is to be used is of great importance, as these substances owe their properties to a structure consisting of minute pores which are filled with air. Should the insulating material become fused through excessive heat into a solid block, then its thermal conductivity increases. Hence more heat than is desirable will be transmitted to the outer case of the furnace. Because of this, suitable literature should be consulted to determine that the correct insulating material is used and excessive heat losses are avoided. Refractory bricks, broken pieces of brick or loose powder may be used, in conjunction with standard insulating materials, as an effective insulation. Diatomaceous earth is another insulating material, and may be obtained commercially as a coarse lightweight powder. There are various grades, depending upon the temperature to which it is intended to be subjected. Diatomaceous earth may tend to react with alumina furnace tubes, but it may be used to insulate nichrome-wound furnace tubes. Because it is inclined to hold moisture and because of its close cell structure, it is difficult to degas should it be used inside a vacuum chamber. For temperatures up to 1100 °C, exfoliated vermiculite is a light, clean and easily handled insulating material.

The list of suitable laboratory refractory materials is quite extensive; a selection of the more commonly available materials is given in Table 8 (see page 68), together with their salientpro-perties.

67

Metallurgical Laboratory Techniques

TABLE 8. COMMON REFRACTORY MATERIALS

Refractory	Melting point (°C)	Maximum operating temperature	Thermal shock resistance	Uses
Alumina Al_2O_3	2020	1900	Good	Laboratory crucibles, tubes and furnace linings
Magnesia MgO	2800	2400	Fair	Furnace linings, used in contact with electrical elements, crucibles for melting ferrous metals
Zirconia ZrO_2	2700	2500	Sensitive to rapid temp. changes	Crucibles for melting pure chromium
Zircon ZrO_2–SiO_2	2650	1750	Quite good	Crucibles, used for melting in induction furnaces
Mullite $3Al_2O_3$–$2SiO_2$	1800	1700	Fair	Furnace tubes
Sillimanite Al_2O_3–SiO_2	1800	1700	Good	Furnace linings, crucibles
Silicon carbide SiC	2250	1650	Good	Muffles for electric and gas furnaces, heating elements
Carbon graphite C	3250	—	Excellent	Electrodes, furnace construction material, crucibles
Pyrex glass	550	400	Good	Low temperature melting
Soda glass	480	300	Very poor	Low temperature melting

Manufacture of Crucibles

The most important factor governing the selection of suitable crucibles is the degree of inertness of the crucible to the metal to be melted. No crucible is completely inert, but it is important to ensure that no change is involved in the properties of the metal from crucible contamination. Most low-melting-point metals such as tin, zinc and lead may be melted in practically any refractory without adverse results, but with higher-melting-point metals (1000 °C and above), consideration must be given to possible reactions between the crucible and the metal to be melted. Any crucible containing carbon should never be used to melt metals which form carbides, e.g. iron or chromium.

Although the more common types of crucible used are usually obtained commercially, they may be made in the laboratory using graphite. This is especially useful should a special-shaped crucible be required. Graphite may be obtained as round rod, in various types and diameters. It is a very useful material mainly because of its chemical inertness (with the exception of oxygen at high temperatures), high melting point, electrical and thermal conductivity, and reasonable mechanical strength which remains unaffected by high temperatures. It is important to have a material available in the laboratory from which a tube or crucible may be machined in a lathe, to the exact size required and in a short period of time. Graphite withstands the rigours of machining extremely well.

Most workers usually avoid working with graphite, because of the dirt and dust involved during machining. The dust tends to find its way into the working parts of a lathe, causing the slides to jam. Excessive wear to the moving parts is unavoidable. The penetration of graphite dust into electric motors should be avoided, otherwise serious damage may result. When machining a crucible or tube from a piece of graphite, it is advisable to have a small portable vacuum cleaner positioned adjacent to the lathe tool post. This will remove the bulk of the dust, and frequent cleaning of the lathe during the machining operation should reduce this hazard considerably. To manufacture a cru-

cible, a piece of graphite may be shaped externally (to conform to the more common crucible shape) by using a form tool as shown in Fig. 2.7. By carefully reversing the graphite in the lathe chuck, the graphite may be bored using a conventional lathe
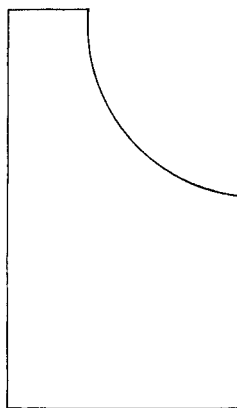

FIG. 2.7. Form tool used for the external shaping of graphite crucibles. The tool may be made from mild steel.

tool. Alternatively, a form tool may be used after rough machining the inside of the crucible to produce a wall of uniform thickness.

A wide range of crucibles of varying dimensions may be quickly manufactured by this method, and the graphite dust may be kept in a suitable container for future applications in the laboratory. One example of its usage is to mix the dust with paraffin for use as a lubricant in steel moulds used for casting alloys.

Chapter 3

Heat Treatment

THE heat treatment of metals necessitates a wide variety of techniques, many of which are outside the scope of small laboratories. However, the more important of these processes can be carried out adequately using small-scale apparatus, and therefore merit discussion.

The term "heat treatment" simply means the use of heat to modify some property (usually hardness or strength) of metals; the heat may be employed directly, as in **annealing** in which the heat itself brings about the desired change, or indirectly, as in **quenching** where it is the **change** in temperature which is the operative factor. It is also important to remember that some heat treatments are applicable only to certain groups of alloys, whilst others are of fairly general significance. Thus **normalizing** can only be applied to steels, whilst annealing is of universal application. In all cases, however, the temperature involved in the heat treatment varies from alloy to alloy; some data for various heat treatments of the commoner metals and alloys are given in Table 9 but for other alloys the necessary data must be consulted in other reference tables (see Bibliography).

The Effect of Heat on Metals

It is not the purpose of this book to provide a background of physical metallurgy, but if the various types of heat treatment for metals are to be intelligible, then some elementary discussion of the mechanisms of these treatments and the resultant changes is essential. Metals are crystalline solids; that is to say, any piece

TABLE 9. APPROXIMATE RECRYSTALLIZATION TEMPERATURES

Metal	Temperature (°C)
Iron	450
Nickel	600
Gold	200
Silver	200
Copper	200
Aluminium	150
Magnesium	150
Tungsten	1200
Molybdenum	890
Zinc	Room temperature
Lead	Below room temperature
Tin	Below room temperature

TABLE 10. ANNEALING TEMPERATURES FOR THE MORE COMMON METALS

Metal	Temperature (°C)
Mild steel	550–650
Nickel	700–900
Copper	350–650
Brass	450–700
Aluminium and aluminium alloys	350–425

of metal is made up of a large number of minute crystals, usually referred to as **grains**. Within each grain the atoms of the metal are arranged in an orderly geometric pattern, or **lattice**. The strength and elastic properties of any metal or alloy depend partly on the bond strength between individual atoms, partly on the crystal pattern of the atoms within the grains, and partly on the size and shape of the grains themselves which make up the metal as a whole. Heat treatment cannot alter bond strengths permanently, except indirectly, but it serves as a method of controlling the grain size of metals, and, on occasion, of modifying the crystal pattern within the grains. It is therefore a useful

and important method of controlling the mechanical properties of metals.

The action of heat on metals (apart from purely chemical effects such as oxidation) is to increase the thermal energies of the atoms in the grains, with the result that they become more mobile and less tightly bonded to each other. One result of this is the softening of metals at high temperatures, i.e. the atoms can slip over one another more easily (see Chapter 4); the ultimate stage of softening is reached when the increase in thermal energy of the atoms from the heating is sufficient to overcome the bond strength of the crystals, at which temperature the metal melts.

Most metals retain the same crystalline structure throughout the entire temperature range in which they are solid; however, a few change their structure at certain temperatures below their melting point. The only important common instance of this effect (known as **allotropy**) is shown by the metal iron. Iron exists in a form known as **ferrite** (α-iron) at room temperature, but transform to **austenite** (γ-iron) at 910 °C, and transforms again to δ-iron (which has the same crystal structure as ferrite) at 1390 °C before melting at 1534 °C. Although iron is the only common metal to exhibit allotropy (tin also exhibits allotropy, but the effect only occurs readily well below 0 °C, and is unlikely to be of importance in the laboratory), most metals change their crystal structure if they are alloyed with a sufficient amount of a second metal; the result of this is that many common alloys consist of mixtures of grains of two different crystal patterns known as **phases**. The fineness of the mixture of these phases, and to some extent their relative properties, can also be controlled by heat treatment. In order to determine which particular phase of an alloy is stable at any given composition and temperature, a plot of their two variables is made which is known as a **phase or equilibrium diagram**. Probably the most important phase diagram is the iron–carbon diagram, which is shown in Fig. 3.1. Although it looks extremely complex, in fact only the part within the dotted circle is of real importance in heat treating. A second version of the diagram showing the various areas of heat treatments is given in Fig. 3.2.
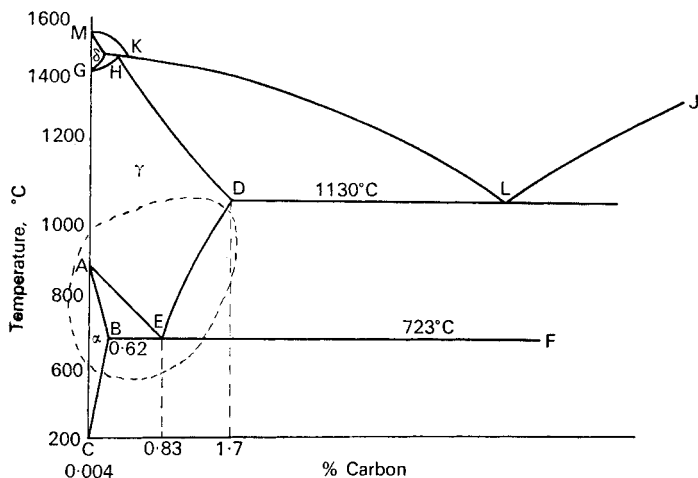
73

FIG. 3.1. The iron–carbon diagram.

Let us now examine the mechanism of one of the most important heat treatments, that known as **age-hardening**. It was first employed in light alloys (duralumin is the classic example) but it can be applied to many diverse alloy systems, and is now used extensively in the treatment of high-strength creep-resistant alloys. Consider the phase diagram shown in Fig. 3.3. The region to the left of the line *MNP* is that where metal B is completely soluble in metal A, giving (let us say) an α solid solution. To the right of *NP*, the content of B in the alloy is such that it cannot all go into solid solution, and the excess forms a second (β) phase. Thus in this area the alloy consists of a mixture of the α and β phases, the proportion of β phase increasing as the composition of the alloy moves to the right in the diagram.

Now take an alloy of composition X. If the alloy is heated up to a temperature T_1, it lies in the α region, and therefore the structure consists entirely of α grains (Fig. 3.4(A)). On allowing the alloy to cool in air to room temperature, the normal equilibrium structure of X will result. As the temperature drops below T_q, crystals of β phase separate out in the grain boundaries of the
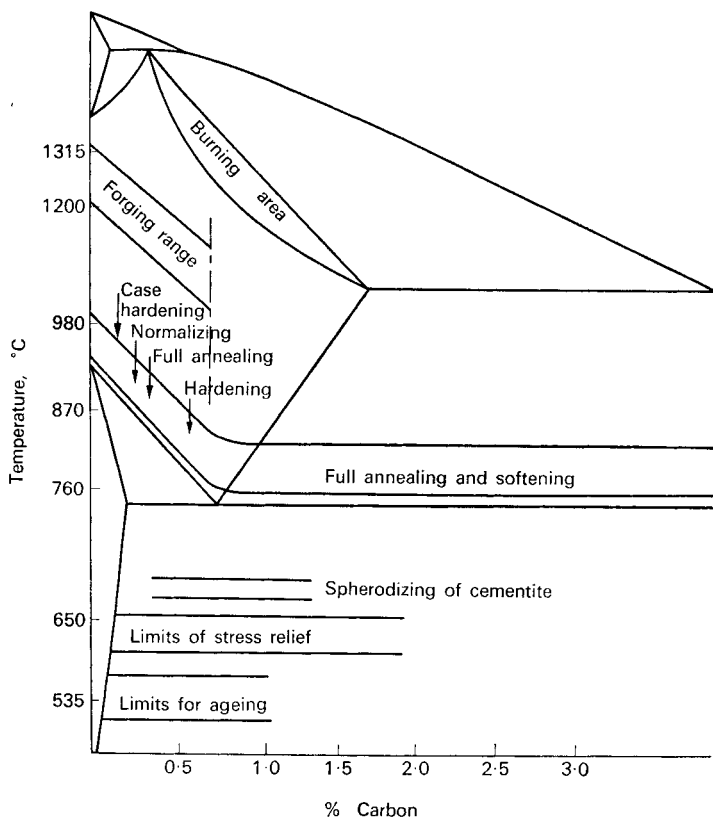
FIG. 3.2. Second version of iron–carbon diagram.

α phase and grow until they reach equilibrium size at room temperature. If, however, the alloy is cooled rapidly (quenched) to room temperature from T_1, then insufficient time is allowed for the metals' atoms to rearrange themselves and precipitate the requisite amount of β phase. As a result, the precipitation is suppressed, and we are left with a supersaturated α solid solution, since X per cent B remains in solution whereas the maximum equilibrium solubility at room temperature is only P per cent.

75

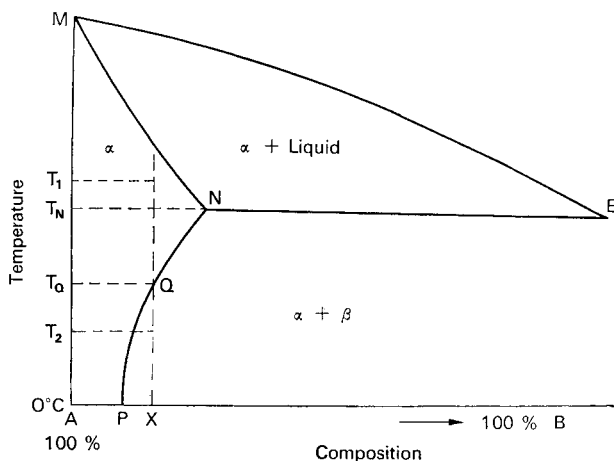FIG. 3.3. Phase diagram of alloy system made up of metals A and B, showing region in which B is soluble in A.
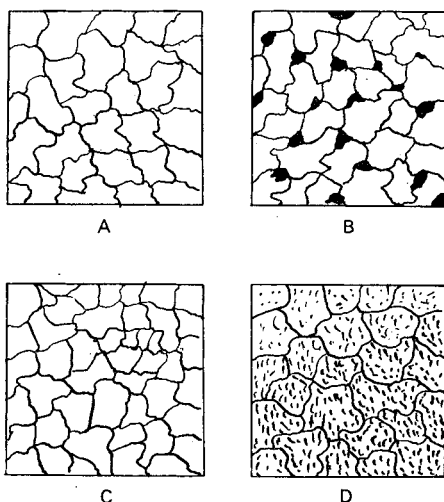
FIG. 3.4. (A) Structure at T_1. (Entire α.) (B) Structure after slow cooling to room temperature. (Mixture of α and β grains shaded dark.) (C) Structure after quenching to room temperature. (Supersaturated α.) (D) Structure after ageing at temperature T_2.

This means that the alloy is in a metastable state with a considerable degree of internal strain, although nothing of this is usually visible under the microscope (Fig. 3.4(c)). If the temperature is now raised to some intermediate value, say T_2, then the additional thermal energy at this temperature plus the effect of the internal stress is sufficient to cause precipitation of the β phase and thus allow the structure to relax. This stage is known as **ageing**.

However, under these conditions precipitation does not occur in a few relatively large areas, as illustrated in Fig. 3.4(B), but as a very fine, roughly uniform dispersion of particles of β phase. This stage of the process may take a considerable time. As the particles are precipitated, the metal becomes harder and the particles slowly increase in size. The presence of the precipitate in a finely divided form has a remarkable effect on the strength of the alloy. However, if the particles grow beyond a certain size the hardening and strengthening effect begins to diminish. There is thus an optimum particles size and therefore an optimum ageing time; this occurs when the particles are still in the early stage of growth and are sub-microscopical in size. If the particles grow sufficiently to be visible under the microscope, as illustrated in Fig. 3.4(D), then the alloy will have passed the stage of maximum strength, and is said to be **over-aged**. The strengthening effect of the precipitate is due to the fact that individual particles have in the early stages of precipitation a high stress field around them, which serves to anchor dislocations and thus prevent slip on any slip-planes in the vicinity.

This example of heat treatment illustrates a very typical feature of most heat treatments. The changes are brought about by diffusion of atoms within the alloy (which can only occur by raising the temperature of the alloy), but the temperature at which the diffusion occurs can profoundly affect the microstructure and properties of the alloy (compare Figs. 3.4(B) and 3.4(D)). Bearing this in mind, let us now consider the commoner heat treatments met within the laboratory.

Annealing

In an earlier chapter in this book we mentioned the ability of a metal to harden by deformation processes. Normally this is beneficial to the mechanical properties of the metal, but if carried too far, it may lead to embrittlement by excessive straining of the internal lattice. Annealing affords the simple means whereby the internal conditions of the metal is restored to its original state, without altering the actual physical degree of deformation. Thus annealing is extremely valuable if we desire to roll a piece of metal down to very thin strip in, say, fifty passes in a rolling mill. The metal might tend to crack from excessive work-hardening after ten passes; but if, after eight passes we anneal it, then a further eight passes may be carried out without any fear of failure, to be followed by another anneal, a further eight passes, and so on down to the requisite final deformation. Alternatively, annealing is frequently used to remove harmful residual stresses in metals which might otherwise render them unreliable in service.

Basically annealing consists of heating a metal above a certain temperature, known as the recrystallization temperature. It is allowed to "soak" at this temperature for a predetermined time in order to allow the atoms to regain their equilibrium positions in the lattice, and then cooled at a predetermined rate to room temperature. Recrystallization temperatures of some common metals and alloys are given in Table 9. The process of annealing ensures adequate grain growth, especially in metals that have been deformed plastically. Also grain growth causes a softening of the metal, thus allowing the metal to be worked further. Metals that have been cold-worked should be annealed to remove harmful stresses, but it is important to remember that working promotes grain growth, which may be beneficial. In this instance, a compromise must be made between the beneficial effects of work hardening and annealing. Mild steel is annealed at a temperature of 550–650°C for several hours depending on the size of the component. This form of annealing is called "close" or process

annealing, and is usually employed on sheet material. The process employed to remove stresses and strains from castings and forged components is called "full annealing".

The method of applying the heat is important also, because it affects the rate at which the temperature of the metal increases, and may contribute in preventing chemical changes in the metal, e.g. oxidation. It should be borne in mind, however, that the rate of heating is not usually constant, and often decreases as the metal undergoing treatment nears the desired temperature. At this temperature it is "held". Whilst dealing with the rate of heating, it should be remembered that the high rates of heating, or non-uniform heating, may be harmful, especially to components of complex shape. In both cases non-uniform expansion of the metal will take place, and may lead to distortion, particularly since metals deform readily above their recrystallization temperature. In brittle materials, the amount of stress caused by distortion may cause the casting or component to fracture.

Furnaces

Heat-treating furnaces are usually designed and equipped to enable the temperature in the furnace to be accurately maintained over the whole area of the heating chamber. Electricity and gas are the two main sources of heat, although for some applications lead and salt baths may be used. Electrical resistance furnaces, namely muffle and pot furnaces (see Chapter 2), are more commonly used in the laboratory. The atmosphere within the furnace is important, to avoid the formation of scale on the component being heat treated. In some instances the component may be protected by passing nitrogen or other protective atmosphere through the heating chamber. Furnaces usually have provision for this. (See section dealing with controlled atmospheres.)

Vacuum furnace

In Chapter 6 mention is made of the use of vacuum furnaces for sintering components made from metal powders. This type of furnace may also be used to anneal specimens, and in this instance the danger of surface contamination from the furnace atmosphere is avoided.
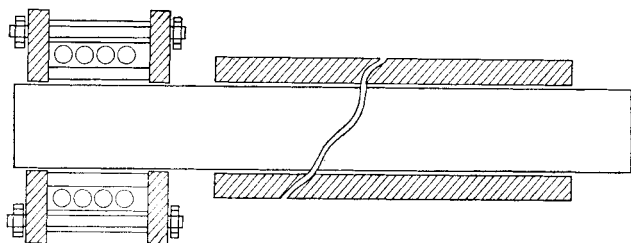

Fig. 3.5. Water-cooled specimen chamber fitted to the end of a furnace tube.

A furnace may be constructed in the laboratory, as discussed in Chapter 2. The tube furnace is quite suitable for annealing under vacuum. During the course of construction it will be found to be advantageous to water-cool one end of the furnace, normally the opposite end to the pumping system, to provide a cooling chamber for the specimens. This may be carried out by fitting a water-cooling jacket to the end of the furnace tube, as illustrated in Fig. 3.5. If a vacuum valve of sufficiently high conductance is positioned between the furnace and the pumping system, the furnace may be opened whilst it is still at a high temperature, and the specimens that have been at a high temperature may be drawn into the cold area, removed and further specimens introduced into the furnace. Once the furnace has been sealed and pumped down to the required pressure of vacuum, the specimens may be pushed into the hot area by way of a rod passed through a Wilson seal (see Chapter 6). However, if there is no provision for a cold chamber in the furnace, then the furnace must be allowed to cool to room temperature each time the specimens are removed or replaced.

80

When annealing is carried out under vacuum the specimens may be placed directly on the floor of the furnace tube. However, if a muffle furnace is used, the specimens should be placed on a stainless-steel tray before being inserted into the furnace. This will facilitate easy insertion and withdrawal of the specimens, and assist in keeping them free from contamination. Annealing is carried out for a predetermined length of time, after which the specimens are allowed to cool very slowly, usually in the furnace, to room temperature. In the case of a normalizing heat treatment, the specimens are withdrawn from the furnace and cooled in still air.
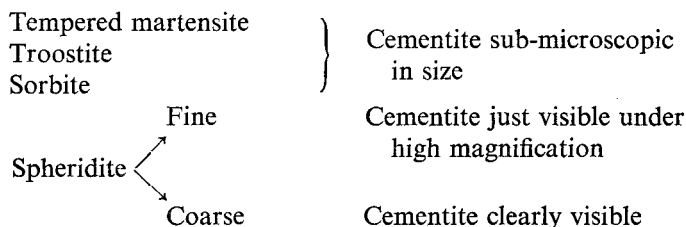
Hardening of Steels

Plain carbon steels in the normalized condition give a range of medium-strength alloys varying from the soft mild steels to the hard carbon silver steels. Fortunately, however, the versatility of steels is enormously enhanced by the process known as quenching and tempering, which extends their properties to a much higher range of strength and hardness values. Steel at room temperature contains virtually all its carbon as **cementite** or iron carbide, Fe_3C in the form of pearlite (see iron–carbon diagram, p. 74). If, however, the steel is raised into the austenite region, the cementite decomposes and the carbon goes into true solution in the austenite. Austenite is the important starting point for the hardening of steel, since the properties of the steel depend on the rate of cooling from the austenite region.

If we take the normalizing procedure first, the change austenite $\longrightarrow$ ferrite and pearlite takes place by a diffusion of carbon atoms within the austenite, and as such requires a finite time for its completion. It is possible to cool the austenite so fast that the carbon has no time to diffuse into the correct pattern for pearlite formation. The carbon is then trapped in the austenite lattice when the latter decomposes, with the result that the ferrite formed by the decomposition is violently distorted—so distorted

81

in fact that it has completely different physical properties and is known as **martensite**. Martensite is extremely hard and brittle (unlike ferrite), and there is an expansion of the crystal lattice and its formation. All these features of its formation are unfortunate in that, together, they greatly increase the vulnerability to cracking or distortion of the component.

Martensite is of very little use *per se* in plain carbon steels, except where extreme wear resistance is the only physical requirement of the steel, but it can be **tempered**. This is a further treatment carried out in the range 300–650 °C which allows the hard, highly stressed martensite lattice to gradually relax, accompanied by the precipitation of small spherical particles of cementite. The hardness and toughness of the final product is governed by the degree to which the martensite has relaxed and the cementite has precipitated. Although the transformation martensite $\longrightarrow$ ferrite + cementite (spheroidal) is a gradual and continuous one, several distinct stages are commonly referred to for convenience:

Tempered martensite	
Troostite	Cementite sub-microscopic
Sorbite	in size
Fine	Cementite just visible under
Spheridite	high magnification
Coarse	Cementite clearly visible

Generally sorbite marks the most useful stage of tempering, combining toughness with an adequate lack of brittleness, but troostite and fine spherodite are sometimes employed, depending on whether wear resistance or resilience is the most important quality of the component.

The whole process can then be seen broadly to resemble the age-hardening procedure mentioned in the theoretical introduction to this chapter.

Quenching Specimens

As mentioned earlier, quenching involves rapidly cooling the specimen to room temperature. This is carried out either to retain a desired high-temperature structure, or to create an entirely new structure as is the case when a metal is hardened. The structure developed or retained by the quench depends on the cooling rate of the specimen, which is in turn governed by the quenching medium used.

The most common metals that are heat treated in this manner are the steels, and the relevant techniques have been developed accordingly. Therefore, for the purpose of this chapter we will deal with the quenching of steels.

QUENCHING MEDIA

Liquids or gases are generally used for quenching specimens, liquids normally being preferred for specimens of the size usually encountered in the general laboratory. Liquids give the most rapid quenching rates, the commonly used liquids being water and oil. Water has more quenching power than, say, oil (by quenching power, we mean the ability to extract heat away from the specimen) and a rough list of quenching media in order of decreasing severity would be: water spray, brine, water, oil, hot oil, air blast. For quenching under vacuum, mercury is sometimes used. For most laboratory work, water is usually the first choice for rapidly quenching specimens. Choice of quenching medium is normally governed by the need to cool at the **minimum** rate necessary to give an adequate quench. This reduces the risk of distortion or cracking of the component. Suitable quench rates depend on the composition of the steel and (since the minimum satisfactory quench rate refers to the rate at the **centre** of the specimen) the size of the specimen. The effect of composition is complex and outside the scope of this book (see, for example, Brick and Phillips, *Structure and Properties of Alloys*, 2nd edition, chapters 10–12; McGraw-Hill, 1949) but very generally

it may be said that increasing alloying elements increases the hardenability (i.e. ease of quenching) of the steel.

It is important to remember that the oil used for quenching must have a sufficiently high flash point to avoid the danger of it catching fire when the hot specimen is immersed. Air is usually reserved for cooling specimens very slowly to room temperature, as in annealing. Gases are also used where a slow rate of cooling is required. However, very rapid cooling may be obtained if the specimen is very small in cross-section.

Actual quenching may be divided into three definite stages. Stage one commences when the specimen loses heat by conductance and radiation through an envelope of gas which initially surrounds the specimen. In some instances this stage would signify very slow cooling. Stage two is the fast stage, and is reached when the specimen becomes wet by placing it into the liquid quenching medium, which in turn vaporizes. The heat is rapidly lost by the conversion of the liquid to gas (steam when water is used as the quenching medium). The third and final stage is when the specimen or hot body is sufficiently cool, so that there is no vaporization. Hence the liquid conducts the heat away. To obtain the maximum cooling or quenching rates the quenching bath should be agitated, or a spray of quenching medium directed against the specimen, or the specimen may be agitated in the bath. This will prevent excessive development of vapour around the specimen, and generally assists in transmitting the heat away from the specimen. In the case of very small specimens, agitation may not be necessary because, owing to their size, these specimens are usually quenched to room temperature within a few seconds.

It is important to remember that the quenching bath must be sufficiently large in relation to the size and quantity of the specimens. Otherwise there is every possibility that the temperature of the liquid in the bath would be drastically raised during the quenching operation. Especially is this so if there is a large quantity of specimens to be quenched. In the case of an oil bath, the rise in temperature may be sufficient to cause the oil to catch fire. Also of importance is the method by which the specimen

or component is placed into the bath. Symmetrical-shaped specimens are usually quenched "end on" to avoid the possibility of cracking from the sudden shock of immersion (rapid reduction in temperature).

It is important to give some consideration to the design of the specimens to be quenched. Correct quenching may be impaired if the specimen contains varying thicknesses in its cross-section, or sharp corners. This is because when the specimen is immersed in the quenching medium, the larger or thicker areas will cool more slowly, and cracking may occur in the thinner sections due to uneven cooling. In the case of a casting, the difference in cooling rates may lead to distortion. Also, bearing in mind what has been discussed earlier in this chapter regarding the various structures produced by quenching and cooling rates, uneven cooling will produce different structures within the specimen. In the case of a component for use in service, any variation in the structure may lead to failure.

On an industrial scale, heat treatment and its salient processes are normally arranged as one continuous operation. Thus, components are heat treated, removed from the furnace, often on a conveyor system, and quenched by powerful jets of water or oil. In the laboratory, however, specimens are usually removed from the furnace using a pair of tongs, and immersion into the quenching medium is carried out as quickly as possible.

It is important to remember that the specimen, when removed from the furnace, is hot and gloves should be worn at all times. This may seem quite obvious, but on occasion a specimen has fallen to the floor of the laboratory, only to be picked up in the unprotected fingers of the laboratory worker. Also it is advisable to refrain from looking directly into the interior of the furnace, otherwise damage to the eyes may result. If, as mentioned earlier, the specimens are placed on a tray within the furnace, their removal should present no difficulties.

QUENCHING UNDER VACUUM

Specimens that are heat treated under vacuum may be readily quenched without disturbing the environment in the vacuum furnace. Figure 3.6 illustrates a simple apparatus for quenching a specimen in oil whilst still under vacuum. If a more rapid quench is desired than is possible with oil, mercury may be used as a quenching medium, provided that there is no risk of alloying with the hot specimen.
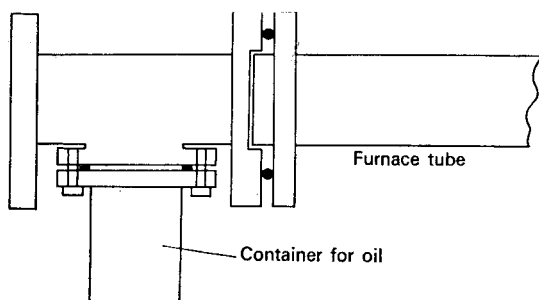

FIG. 3.6. Simple apparatus for quenching specimens in oil whilst under vacuum. Specimen is pushed along the tube into the container of oil. A valve may be situated between the tube and container, thus keeping the vacuum system under vacuum whilst removing the specimen.

The specimen may be suspended in a vertical furnace by a piece of wire which may be melted at the appropriate time by passing an electric current through it. This will allow the specimen to fall down the furnace tube into a container of diffusion pump oil. This type of oil will not have the desired quenching effect as would the oil generally used for quenching. However, it should be quite suitable for quick laboratory experimental purposes. Another method which may be employed is to use the tube furnace in a horizontal position. The container of oil is placed in a suitably designed chamber. By passing a rod through a Wilson seal the specimen is pushed along the furnace tube and into the container of oil.

Tempering

Tempering may be carried out in the laboratory, using the type of furnace described in this chapter and in Chapter 2. Small "one off" items may be tempered using a blowtorch, but the temperature control in this method is obviously rough and ready. As the steel is heated, a film of oxide forms on the surface of the component. Initially the film becomes pale yellow in colour, but as the temperature is raised, it gradually becomes darker. This change in colour continues as the temperature is raised still further, until it becomes dark blue in colour. This colour/temperature relationship is a useful guide for tempering specimens or small tools under laboratory conditions. It should be

TABLE 11. TEMPERING COLOURS

Approximate temperature (°C)	Colour	Item
220	Pale yellow	Turning tools, scrapers, counterbores
230	Straw	Hammer faces, planing tools, counterbores
240	Dark straw	Milling cutters, drills, reamers
250	Light brown	Taps, dies, punches
260	Purple/brown	Punches, reamers
270	Purple	Surgical tools, press tools
280	Deep purple	Cold chisels as used for steel
290	Blue	Cold chisels for use on wrought iron, screwdrivers
300	Dark blue	Wood saws, springs

remembered, however, that this method of matching colour with temperature applies only to plain carbon steels. Table 11 lists a selection of typical items and their colour/temperature combination.

Tempering baths

Tempering baths are often employed in this phase of heat treatment. Liquids will transfer heat more uniformly, and they have a higher heat capacity. Oil is often used for low-temperature baths, and salts for high temperatures. The salts may be mixtures of sodium nitrate and potassium nitrate, and are suitable for temperatures up to 500 °C.

An important item of furnace and bath heat treatment is temperature control. Usually the temperature is controlled by pyrometric devices, but temperature crayons have been used successfully when tempering is carried out in the laboratory. Especially is this so when a blowtorch is used as the source of heating (see Chapter 2).

Temper brittleness

When a steel has been hardened it is tempered, as discussed above, and its mechanical properties undergo a gradual alteration. Specimens that have been tempered in the temperature range 300–500 °C and allowed to cool **slowly** in air often give lower Izod impact values (loss of toughness) than the values obtained from specimens which have been hardened but are in an untempered condition. This phenomenon is known as **temper brittleness**. It is interesting to observe that the condition of this phase of heat treatment which produces this phenomenon has no effect on any other mechanical properties of the specimen. As already mentioned, temper brittleness occurs in specimens or components that are slowly cooled through a critical temperature range, e.g. 650 °C down to 300 °C. It has been found, however, that temper brittleness can be avoided by cooling the specimen or component quickly, from the tempering temperature, by quenching in water. Even though the exact cause of temper brittleness has not yet been clearly established, it is thought to occur through some form of precipitation at grain boundaries during the period of transition.
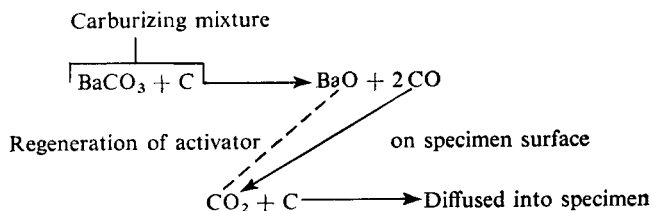
Case Hardening

Case hardening is a relatively simple operation which may be carried out in the laboratory or workshop. As the name implies, case hardening simply means surrounding or covering a component with a hard skin or "case", leaving the centre of the material in a soft state. Steels containing 0·15 to 0·25 per cent carbon are most suitable, and may be used for the production of light-duty cams. In this instance, the cam must have a very hard-working surface to resist wear, but must also be tough to withstand the rigours of service, such as impact. Steels of higher carbon content may be carburized but there is a danger of embrittlement due to excessive carbon in the case. The processes of case hardening fall into four categories; each will be discussed briefly.

CARBURIZING

Consider the component mentioned above, a steel cam. It is usually packed or buried in a compound consisting mostly of carbon, e.g. charcoal. It is heated to a high temperature (950 °C) for a period of time, up to 1–2 hours, and then quenched in water. During the course of heating, carbon penetrates from the compound into the surface of the component, usually initially at grain boundaries of the structure of the steel. As will be appreciated, the skin of the component is now higher in carbon content, and because it has been rapidly cooled (water quenched) the characteristics of the skin are similar to a high carbon steel which has undergone a similar heating and quenching process. Furthermore, because of the extra volume of material diffused into the surface layer, the process induces a state of residual compression in the surface of components which is beneficial in combating the effects of fatigue. However, it is important to realize that the charcoal, when used on its own, is unsatisfactory because it provides a slow penetration of the workpiece. By adding an energizer, such as barium carbonate, more rapid penetration to a greater depth will be obtained.

Therefore,

Carburizing mixture
$$BaCO_3 + C \longrightarrow BaO + 2CO$$

Regeneration of activator ⟋⟋ on specimen surface

$$CO_2 + C \longrightarrow \text{Diffused into specimen}$$

A second reaction product which is released from the carburizing compound containing barium carbonate is carbon monoxide gas. This is formed by a reaction between the charcoal and oxygen contained within the case-hardening box. Carbon from the carbon monoxide diffuses into the component, releasing oxygen to form carbon dioxide which in turn is reduced to carbon monoxide by contact with the hot charcoal. From this it may be seen that the additional quantity of carbon dioxide in the case-hardening box supplies more carbon monoxide, because of the reaction with the charcoal, thus increasing carburization.

A suitable case-hardening compound may be obtained from commercial sources or, if so desired, prepared in the laboratory. This may be carried out by mixing a 60/40 mixture of charcoal and barium carbonate in a mortar and pestle. This is especially useful for mixing purposes if the charcoal is not already in powder form. One application of the compound usually provides a "case" approximately 0·003–0·005 in. in thickness after heating for 15–20 minutes. However, if necessary this may be increased slightly by repeating the case-hardening operation.

Gas carburizing

Small components are often treated by the process of gas carburization—for example, gears as used in the automobile industry. The carburizing medium employed is usually methane or propane gas. The gas must be of a high purity to avoid depositing a film of soot on to the component. This is often the case

when other hydrocarbons are present. If it is desirable to provide a deep "case" on the component, this may be achieved by prolonging the heating period and subjecting it at the completion of the carburizing cycle to a gas mixture that is lower in carburizing power. This may be accomplished by regulating the gas supply.

CYANIDING

It is sometimes necessary to provide a thin "case" to the surface of a component in a short period of time. For this, use may be made of a cyanide bath. The bath usually consists of a mixture of sodium and potassium cyanide. It is of great importance to remember the following when using cyanide baths. **Cyanides are strong poisons.** Carelessness on the part of the operator will lead to **instant** death. Therefore if these baths are used in the laboratory, the cyanide furnace must be well ventilated, ensuring that the ventilation flue has a strong draught. Protective clothing **must** be worn at **all** times, and accidental splashes onto the person must be removed instantly.

The cyanide furnace is usually operated at a temperature between 750°C and 850°C. The component to be treated is immersed in the bath by suspending it on a wire hook. A quantity of components may be placed in wire baskets. Treatment time is usually in the order of 5–10 minutes though this time factor is rather flexible, depending on the depth of case required. The component is then removed from the furnace and quenched in oil or water, depending upon its composition. However, water quenching has the advantage of washing off the excess cyanide.

NITRIDING

A more up-to-date method of case hardening is the process of nitriding. Steels of certain compositions are heated to approximately 475°C in a stream of ammonia gas. The steels treated

91

this way are usually alloy steels. Steels containing aluminium, chromium or molybdenum are particularly suitable for this treatment, since these elements react with nitrogen to form hard nitride needles in the outer skin of the steel. There are other elements which are also used as alloying elements, namely silicon and tungsten, but in general use is made of aluminium.

The advantage of nitriding is that the complete operation requires heating the component in ammonia gas at a temperature of 475 °C and allowing it to cool slowly in the furnace. Surface contamination encountered in ordinary heat treatment is avoided. Hence components may be machined to final size and hardened by nitriding without any further operations. Also, because of the slow cooling, distortion is minimized. One disadvantage if this process is used in the laboratory is the presence of ammonia fumes. However, this should not become unbearable, providing an adequate ventilation system is available. There is also the danger of inducing temper brittleness (see also p. 88), and therefore steels containing nickel or molybdenum are best, since these elements resist the onset of embrittlement.

FLAME HARDENING

Flame hardening is a process usually associated with industrial concerns. However, simple hardening may be carried out in the laboratory, heat being supplied by an oxy-acetylene hand torch. The specimen is heated to a suitable temperature, and then quenched in the chosen quenching medium. If the area to be hardened is sufficiently small compared to the overall size of the component quenching is sometimes unnecessary since heat is conducted away from the area into the body of the metal so rapidly as to achieve a quench rate of cooling. Large components may be flame hardened. The process is useful because it is often unnecessary to harden the complete component, but only the surface or skin. An example of this is a large gear as used in heavy machinery. In the case of this type of component, the method of cooling is important. The teeth of the gear usually require

hardening; using an oxy-acetylene flame, it is played on to each tooth in turn. Usually either provision is made for the flame to move from tooth to tooth, or the gear is made to rotate slowly past the flame. Care should be exercised to avoid melting the surface in the extreme heat of the oxy-acetylene flame. Each tooth is quenched by water spray after it has passed the flame. This ensures rapid quenching. Also, on large-diameter gears the gear is made to rotate through a water bath. Thus the half of the component not being hardened, i.e. under the flame, is immersed in the water and so remains cool. Circular components such as piston pins, shafts, etc., are often mounted on centres and rotated in a machine. The flame is directed onto the component and, at the desired time, a water spray is operated from all sides of the component.

Controlled Atmospheres

It is frequently necessary in the laboratory to prevent reaction between the atmosphere and metals. Most metals are adequately stable in air at ambient temperatures, but almost all are vulnerable at heat-treatment temperatures. This is especially true where metals are being prepared for metallographic examination of the surface or for small-scale physical testing, when any appreciable degree of oxidation or other surface contamination could render the specimens completely useless.

The term "controlled atmosphere" can imply a wide variety of systems, varying from the simple but reasonably effective use of silica gel in storage cabinets to prevent rusting of steel specimens, to highly elaborate equipment in which contaminating impurities in the atmosphere are reduced to less than 1 ppm. The principal atmospheric contaminants are oxygen and water vapour; however, for certain metals and alloys nitrogen and/or carbon dioxide can also be harmful, and the technique of controlled atmospheres is based on taking a suitable inert gas for the metal concerned, say hydrogen or argon, and passing it over or bubbling it through a series of absorbing agents which will remove any harmful impurities down to an acceptable level.

TABLE 12. CONTROLLED ATMOSPHERES FOR COMMON METALS

Atmosphere	Iron	Steels	Copper	Brass	Aluminium	Tin	Lead	Chromium (incl. chr. plate)	Nickel	Comments
Vacuum	Suitable		Suitable	Tendency for the zinc to vaporize	Suitable		Suitable	Suitable	Suitable	
Hydrogen	Suitable	Wet hydrogen may cause decarburization	Suitable	May cause embrittlement					Suitable	Inflammable
Nitrogen		Suitable	Suitable	Suitable			Suitable			Copper and its alloys may suffer small traces
Argon	Suitable	Suitable	Suitable	Suitable	Suitable	Suitable	Suitable	Suitable	Suitable	
CO/CO_2 mixture		Adjust flow to give a neutral atmosphere to carbon in steel								Poisonous, depending on the CO concentration
NH_3	Suitable	Suitable	Suitable	Suitable		Suitable		Suitable	Suitable	

One of the first things which any laboratory worker must decide on is the appropriate atmosphere in which to carry out his work. This decision is usually arrived at in two stages: (a) which atmospheres are possible? and (b) of the possible atmospheres, which is preferable? For this reason it is necessary to treat controlled atmospheres, at least partially, on a comparative basis, and a rough attempt to summarize the merits, demerits, and possibilities of the commoner controlled atmospheres is made in Table 12. Obviously, such a table can make no claim to be exhaustive, and the final decision must depend on the particular conditions of the experiment.

Vacuum

The use of vacuum during heat treatments has certain points in its favour, providing that the system can be kept **simple** (i.e. ultra-high vacua are not necessary) and **small** (which depends largely on the size of the component treated). Given these two provisos, then it is an easy matter to connect up a rotary pump to a simple vacuum furnace according to the precepts set out in Chapter 6.

On the other hand, it must be remembered that a vacuum is a passively neutral atmosphere. That is to say, it protects merely by the absence of harmful gases and vapours rather than by any active chemical action which will remove impurities on the surface of the metal (as is the case, for example, if there are traces of oxide on a copper surface in a hydrogen atmosphere). Allied to this is the fact that any small leak results in an influx of air into the system and subsequent damage to the surface of the specimen, whereas if a gas is used for the controlled atmosphere, the pressure inside the system can be adjusted to be somewhat higher than atmospheric so that any leaks result in an egress of protective gas rather than an influx of harmful air.

Finally, if heat-treatment temperatures are high, many metals will be found to be surprisingly volatile at low pressures. Appreciable loss of material can occur in this way, and apart from

damage to the specimens, contamination of the vacuum system by condensed metal vapours is a nuisance and can take much time and trouble to clean up.

GASES

Inert gases may be obtained from commercial sources, supplied in metal cylinders of various capacities. The four common gases are hydrogen, nitrogen, argon and helium. Of these four gases, argon and helium are the only two that are inert to all metals. Hydrogen does not react with many metals, and may be purchased at low cost, which may make it attractive as against the more expensive noble gases.

HYDROGEN

Hydrogen is capable of reducing many oxides, being inert to metals which are clean and do not form hydrides. Metals often treated in an atmosphere of hydrogen to prevent oxidation are iron, molybdenum, tungsten, nickel and cobalt. As mentioned in Chapter 7, metal powders may be deoxidized by passing hydrogen over them whilst heating to below their sintering temperature. For many applications of heat treatment, it is desirable to use hydrogen which is very low in oxygen and water vapour content. It is important to remember that hydrogen, especially wet hydrogen, may in some instances actually remove constituents from a metal. In the case of steel, for example, carbon may be removed by the action of wet hydrogen. In the heat treatments discussed earlier, this would be most undesirable. Also, there is a risk of hydrogen absorption by copper specimens at a high temperature, although for most laboratory purposes this is not serious. One disadvantage of hydrogen is that combined with oxygen it forms a dangerous explosive mixture. Therefore if used in large quantities, there is the problem of its disposal. One method is to pass the hydrogen from the

exit end of the furnace through a gas train to remove any oxygen and then light the stream of hydrogen. Of course, care must be taken to maintain a very slow hydrogen flow to avoid the risk of any explosion. It is often advisable to test a small quantity of the hydrogen before applying a light, to determine whether any oxygen content is of a dangerous proportion.

NITROGEN

Nitrogen has a limited application as a protective atmosphere for metals that do not form nitrides, or dissolve nitrogen. Copper and its alloys, steel and certain alloys of iron may be subjected to nitrogen, providing that a small trace of nitrogen in the metal will not have adverse effects. Also it may be used for nickel, cobalt, zinc, lead and silver. However, nitrogen may react, although rather slowly, with such metals as iron, aluminium and tantalum.

Nitrogen may require purifying, especially if its application is critical. This may be carried out by passing the gas through a drying tower, to remove water, then over copper turnings at a temperature of approximately 600 °C to remove oxygen.

The process of nitriding may be carried out on some metals by a direct reaction with nitrogen gas, but metals such as iron are usually nitrided by ammonia (see section dealing with nitriding). It should be borne in mind, however, that nitrogen is a passive atmosphere. By this is meant that it will not remove oxide that is already on the surface of the metal.

ARGON

When it comes to the question of a truly inert gas, then the answer lies in the use of argon, though helium should also be considered. However, helium possesses approximately ten times the thermal conductivity of argon. In this instance, helium would conduct heat away from the component undergoing treatment

to the colder areas of the furnace more quickly than argon. Helium is an ideal gas to pass through a vacuum furnace in order to break the vacuum when the furnace must be cooled quickly. However, helium is rather costly to purchase.

Argon as supplied from commercial sources may require purifying. In this case, it may be passed over calcium or copper turnings in a tube furnace at a temperature of 600°C, then through another furnace at a temperature of 350°C. Both furnaces may be constructed from steel, which will prevent any reaction with ceramic materials. The first furnace will remove all impurities with the exception of hydrogen. The second furnace will remove the remaining hydrogen, because calcium hydride is stable at this temperature. This method involving two furnaces will provide inert gases with only very small amounts of oxygen, nitrogen and hydrogen present. It is important to remember that at a temperature higher than 600°C, the oxygen and nitrogen content will be reduced further. A temperature of 600°C is suitable because there is an increased rate of oxidation of iron by air at higher temperatures. Also oxygen will diffuse through iron more rapidly at high temperatures, thus causing a reaction with the calcium to form calcium oxide.

Chapter 4

Testing of Materials

Mechanical Testing

The fundamental mechanical properties of metals are ductility, malleability and toughness. Ductility may be described as the ability of a metal to undergo deformation under tension without breaking. If a metal is malleable, it is capable of withstanding deformation under compression. Whilst metals that are malleable may also be ductile, this factor cannot be assumed since a metal which is malleable may prove to be weak in tension. Malleability may be increased by raising the temperature of the metal (alloys may be hot rolled or hot forged) but the ductility of the same metal is usually reduced, since its strength is also reduced. Toughness refers to the ability of a metal to withstand bending or failure from fracture due to the application of shear stresses. It is important to remember that toughness should not be confused with either strength or hardness.

These fundamental mechanical properties cannot be expressed in numerical terms; hence it is necessary to subject the metal to certain mechanical tests. From the results obtained, specifications may be drawn up from which an engineer may base his designs.

Types of Test

Testing of materials is usually divided into three groups as follows: (a) tests that are designed to simulate conditions of service; (b) tests which have been designed to allow a study

of the behaviour of materials under controlled conditions, which are not necessarily related to service conditions. Later in this chapter the third group, methods of non-destructive testing, will be briefly outlined. The first of these groups is usually expensive, slow and generally requires elaborate equipment. It is the second and third groups with which we will be concerned, dealing in the first instance with the tensile, compression, torsion, impaction, creep, fatigue, and hardness comparison tests. These types of test have a disadvantage, however, in that they are destructive and, with the exception of some hardness tests, cannot be performed as a routine test on components going into service.

Behaviour of Metals under Stress

To begin to appreciate the significance of mechanical tests on metals, it is essential that the rudimentary principles of metallic deformation are understood. The reaction of any metal to a steadily applied stress may take one of two forms, depending on the magnitude of the stress. (The behaviour of metals under impact loading bears no relation to the principles outlined below; reference should be made to the section on impact testing.) Metals are crystalline substances, which means that their atoms are arranged in a regular array within each crystal. Any deformation of the metal by an applied stress must therefore occur by movement of the atoms relative to one another within the crystal. If the stress applied is low, then the metal extends by a stretching of the inter-atomic bonds in the direction of the stress, as is clearly illustrated in Fig. 4.1. If the stress is relieved, then the inter-atomic bonds immediately resume their normal equilibrium length, and the metal returns to its original dimensions. Such a stress, which has no permanent effect on the metal after its release, is termed an **elastic stress**, and the range of stress over which the metal reacts in this manner is termed the **elastic range** of the metal. As the stress is raised, there comes a point (the **elastic limit**) beyond which the metal commences to deform in another way as well. This process, known as **plastic**

deformation, occurs because the regular layers of atoms in the crystals slip over one another, as shown in Fig. 4.2, and any such slip is irreversible, conferring a permanent deformation
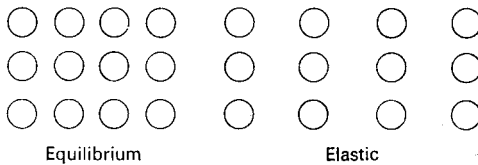

Equilibrium   Elastic

FIG. 4.1. Illustrates stretching of inter-atomic bonds in the direction of the stress.

or set of the metal after the stress is released. The "slipping" takes place on certain well-defined crystallographic planes, known as **slip planes.**

As soon as plastic deformation commences, another pheno-menon comes into play which is of vital importance in under-standing the deformation behaviour of metals, i.e. **work-hardening.** No metal crystal is ever perfect, however carefully
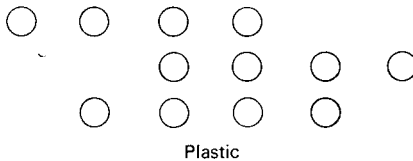

Plastic

FIG. 4.2. Shows how the regular layers of atoms in the crystals have slipped over one another due to plastic deformation.

prepared and purified; they all contain a number of regions of atomic misfit known as **dislocations.** In their normal amounts, the dislocations weaken the metal and aid deformation. (The actual strength of metals is many times less than their theo-retical strength, which is just as well; most of the common structural metals and alloys would be completely unworkable if their crystal structures were perfect.) Over the elastic range of

101

the metal, the dislocations play no part, since they do not affect the inter-atomic bonding appreciably, but when plastic deformation sets in they move along the slip planes in the metal crystals. Indeed, slip is not possible without the movement of dislocations. As deformation proceeds, however, the dislocations multiply rapidly within the crystal, and as their numbers increase, so the likelihood of dislocations interacting with one another increases also. The results of any such interaction almost invariably lead to loss of mobility of the dislocations concerned, thereby making slip along the planes of these particular dislocations more difficult, and therefore **increasing the resistance to further slip**. Thus, when a metal is plastically deformed, its resistance to the stress increases steadily as the deformation proceeds, and the metal is said to be **work-hardened**. The hardening of metals due to the process of work-hardening is illustrated by the popularity of the "bent-poker" act performed by strong men, for it is easier to bend the poker than to straighten it. With the exception of tin and lead, all common metals follow the behaviour described above at ambient temperatures. The practical application of these principles is best appreciated by considering the behaviour of a metal under increasing stress, and analysing the result in relation to the internal changes taking place.

The relationship between the typical curves obtained from a test and the internal changes in the metal being tested may be studied by careful analysis of an ideal tensile test. It is important to realize that, apart from the various numerical results which may be obtained from such a test, the curve is a **faithful representation of all the physical changes in the metal**—in other words, there is a physical reason for every detail of shape in the curve. This may be regarded as a truism, but it is surprising how often this is overlooked in practice, when so much information concerning the general character of the metal can be deduced from the shape of a tensile curve.

Let us analyse the conventional curve shown in Fig. 4.3 in relation to a metal. The curve shown may be referred to as either a load extension curve or a **conventional** stress–strain curve, de-

pending upon the units used along the axes. The qualification
"conventional" is important; it means that the stress is always
measured in relation to the **original** cross-section of the speci-
men, and is the reason why the stress apparently drops beyond
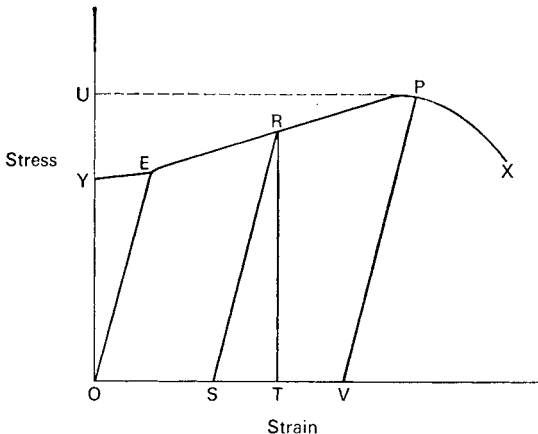the point *P*. Since the cross-section of the specimen decreases

FIG. 4.3. "Ideal" conventional stress–strain curve.

throughout the test, the **true** stress is always greater than the
conventional stress. However, true stress–strain curves are sel-
dom used; perhaps because, surprisingly, they yield less useful
practical information than the more scientifically inaccurate
"conventional" curves.

As the load is applied, the atomic bonds in the metal are
stretched elastically over the range *OE* (the **elastic range** of the
metal). This is the only internal effect which stressing produces,
since the stress required to cause slip has not yet been attained.
Slip starts to occur when the stress reaches the value *Y* and the
curve reaches *E* (the **yield point**). At this stage, the stress is
sufficiently great to cause slip, and the mechanism of defor-
mation over the range *ERP* may be considered stage by stage as
follows. As soon as the stress exceeds *Y*, some part of the metal
most suitably orientated starts to slip; this section then im-

103

mediately work-hardens, and as a result is strengthened suffi-
ciently to withstand the stress. The stress then increases, together
with the elastic deformation, until another section of the metal
slips and work-hardens. This process, of course, takes place
smoothly in practice with slip and work-hardening occurring
throughout the specimen continuously, together with a steady
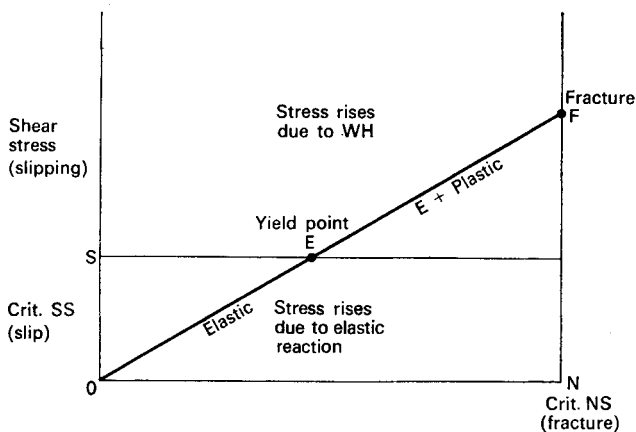

FIG. 4.4. Simultaneous occurrence of slip and elastic strain in a metal.

increase in elastic deformation. The simultaneous occurrence of
slip and elastic strain in a metal is shown in Fig. 4.4. As the stress
steadily rises, the critical shear stress (i.e. stress to cause slipping)
is first reached at the point E for almost all metals, and only
after a considerable further rise in stress is the critical normal
stress (i.e. cohesive strength) exceeded at F and fracture occurs.

Once slip has started, it is only the work-hardening which
prevents the metal failing without any further increase in load;
the gradient of ERP may be taken as an indication of the ability
of the metal to work-harden. If the stress is released over this
section at, for example, point R, then the metal contracts along
RS. This shows that the total strain OT is made up of two com-
ponents: OS, the plastic strain, which is permanent, and ST, the
elastic strain, which is recoverable. For the ideal curve SR is

104

parallel to *OE*, but in practice *OE* is seldom straight since some plastic yield, even in small amounts, occurs almost from the initial loading, especially in non-ferrous metals, as illustrated in Fig. 4.5. However, if a metal exhibiting no definite yield point (as shown in Fig. 4.5) is loaded and released along *ORS*, and
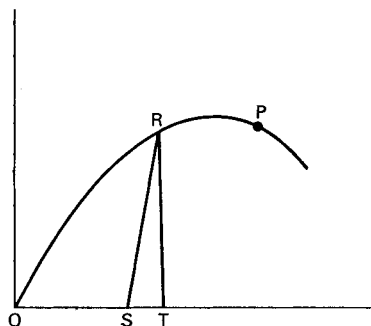

FIG. 4.5. Development of an elastic range in a non-ferrous metal in the work-hardened state.

then reloaded, it will be found that the metal follows the line *SRP*. That is to say, by loading and work-hardening the metal, it has been given a definite elastic range and yield point, which is of great value in the employment of non-ferrous metals. In effect, the plastic component *OS* of the strain *OT* has been eliminated by the process of working.

Tensile Test

TENSILE SPECIMENS

Although specimens may be obtained from commercial sources, they may be made, for class demonstration purposes, from stock materials, compressed and sintered metal powders, or from a component that has been in service. The cross-section of the specimen is usually circular or rectangular, and the di-

mensions must conform to the British Standard Specification for tensile specimens. This ensures that the metal is tested over a measured length of uniform cross-section, called the *gauge length*. Figure 4.6 illustrates this. The gauge length is usually determined from the relationship:

gauge length $= 4\sqrt{}$(cross-sectional area of the specimen).

When turning circular specimens on a lathe, a special turning tool should be used to produce shoulders with correct radii at the "heads" of the specimen. The heads should be larger in
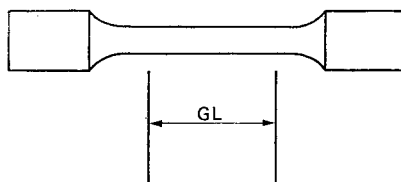

FIG. 4.6. Gauge length of a tensile specimen. This length must be parallel and free from tool marks, which act as stress raisers.

section than the gauge length to ensure that the fracture occurs in the correct region. The shoulders also provide a means of retaining the specimen in suitable chucks during the actual tensile test. Specimens may also have screwed ends for fixing into special chucks. A first-class finish is essential over the gauge length, especially at the shoulders; any tool marks or blemishes on the surface will undoubtedly result in an early fracture, and this will possibly occur in the wrong part of the specimen.

Some types of tensile testing machines use large test specimens which, even though made from stock material, present quite a different problem. This is especially true if a test specimen is required from a component that has been in service. It is quite possible that only a small amount of material would be available, and in this case, use may be made of a machine especially designed for testing small or miniature specimens. This is useful because when testing homogeneous materials the accuracy of the

106

test is not usually affected by the size of the specimen, provided the dimensions conform to the standard laid down. Hence accurate results may be obtained comparative to the larger specimens. Furthermore, the use of small specimens has an advantage over the conventional size. Should the material available be from, for example, a casting, any influences that may have been placed upon the mechanical properties of the material during casting are taken into account.

THE TESTING MACHINE

Basically, testing machines apply the load to the specimen by one of two methods: (a) hydraulic pressure applied by means of a motor-driven pump. In this method oil pressure operates a ram which is provided with grips or chucks. These hold the specimen firmly at one end, while at the other end it is fixed. The rate of loading may be set at any desired value by presetting a control valve. (b) The load is applied by means of a geared screw; the rate of loading in this instance is determined by a gear-box, which has provision for various gear combinations to allow a changeable rate of loading. The load applied to the specimen may be measured by one of the following methods. The load may be transmitted by way of levers to one side of a steelyard. A poise is moved manually on the other side of the steelyard until it is balanced. The arm is usually marked with an appropriate unit of load. Similarly, a pendulum may deflect, by way of suitable levers, a pointer which will give direct readings on a clock-type indicator. The Hounsfield Tensometer as illustrated in Fig. 4.7 measures the load by the deflexion of a steel beam, which is fixed at the ends. The deflexion of the beam moves a column of mercury along a glass tube of uniform bore. The position of the mercury indicates the load, which may be read off from a suitable scale fitted alongside the tube; the advance of the mercury may be followed along the tube, using a cursor. A sheet of graph paper is fitted around a geared recording drum, and is punctured at regular intervals by a needle fitted to the

107

cursor, thus producing a permanent record of the test in the form of a graph. The graph is in fact the stress–strain curve of the specimen under test. The rotation of the recording drum is proportional to the elongation of the test specimen, and depending upon the gear ratio employed to operate the drum, the elongation of the specimen may be magnified up to 16 times.

Methods of Testing the Tensile Strength of Metals

PROOF STRESS TEST

Modern engineering design requires knowledge of the exact stress at which a material reaches its elastic limit, and hence the proof stress, or yield point, is a matter of importance. During the initial part of a tensile test, a sensitive instrument called an **extensometer** is used to enable longitudinal strain readings to be obtained, and from these readings stress versus strain curves may be determined.

THE HOUNSFIELD TENSOMETER

The Hounsfield Tensometer may be attached directly to the specimen and will measure the degree of elongation accurately. It may be used for specimens up to $1\frac{1}{8}$ in. in diameter or rectangular section up to 1 in. square, provided the specimen has a parallel length of $2\frac{3}{4}$ in., and a true diameter. Some types of extensometer must be fixed to gauge length marks previously measured, using a ruler, and marked with a centre punch or by scribe lines. The Hounsfield Tensometer may be fixed at a precise gauge length of 2 in. without any prior marking of the specimen. Should it not be possible to use a specimen with a parallel length of $2\frac{3}{4}$ in. (as would be the case using small or miniature specimens), the specimen may be inserted into special proof stress chucks. For high-accuracy work on large specimens, a roller-type extensometer may be employed.

108

PROCEDURE FOR TESTING

The procedure for carrying out a test in this machine is as follows. The gauge length and diameter of the specimen should be measured before loading it into the special chucks. Once loaded, the pins on the chucks which locate the extensometer should be checked to ensure that they are at the correct distance apart (this should be 2 in.). The extensometer may be fitted to the chucks and the complete assembly arranged in the testing machine. The manual control operating the screw may be used to position the chuck attachment lugs, this facilitating the insertion of the pins which hold the extensometer in the machine. Before commencing the test, it is important to ensure that the correct spring beam and scale is in use, e.g. the $\frac{1}{2}$-ton beam for aluminium and annealed copper, and the 1-ton beam for mild steel or work-hardened copper. A sheet of graph paper, obtainable from the manufacturers of the machine, should be placed around the recording drum. At this stage the mercury column must be checked to ensure that it is free from air bubbles. This may be carried out by simply operating a mechanism which pushes the mercury along the tube, releasing any air that may have been trapped in the mercury, and returns it to the zero mark on the scale. It is important to zero the mercury correctly, otherwise inaccurate results will be obtained. As the operating screw is **slowly** turned to take up the slackness in the chuck and extensometer assembly, the mercury should move slightly, indicating that the slackness has been taken up sufficiently to start the test. At this stage the extensometer should be set to zero, and as the load is **slowly** applied to the specimen, readings may be taken at frequent intervals for loads up to approximately one-third of the full scale. Extra readings should then be taken back to zero.

MAXIMUM LOAD TEST

When the proof stress or yield point has been determined, the specimen may be subjected to a test to determine the maximum

109

load that it will endure. For this test the specimen **must** be removed from the special chucks and extensometer. This is very important; on no account should a maximum load test be conducted with the extensometer still in position, otherwise it will suffer damage. The specimen having been removed, it should be fitted into simple general-purpose chucks, replaced into the machine and retained in position with suitable pins normally supplied with the machine.

The actual test may now be carried out as follows. The slackness in the chuck assembly should first be accounted for as described earlier, and the mercury brought to the zero mark. It should then be ensured that the correct beam and scale is in position. The handle operating the screw mechanism should now be turned **slowly**, particularly as the curve recorded on the graph paper becomes horizontal. As the mercury rises it may be followed along the column, using the cursor and puncturing the graph paper at regular intervals. Once past the maximum load, the specimen will deform under **decreasing** loads, and prompt plotting and following of the mercury level are necessary if this part of the curve is to be recorded.

Throughout the test the specimen elongates due to the increase in the applied load. When a ductile material is undergoing test, this continual application of load causes elongation and also work-hardening, until a point is reached at which some section of the specimen is unable to strain-harden rapidly enough to support the load. Hence the particular section at which **all** the load is concentrated elongates very rapidly and therefore decreases in cross-sectional area due to localized deformation known as "necking". A point is reached at which further increases in load causes fracture as illustrated in Fig. 4.8. Should "necking" occur near either end of the specimen, the values obtained from the test would be low, due to the support of the ends of the specimen, which are of larger cross-section. In this case, another test would have to be carried out. When flat strip is tested, the specimen being of wider section may show a lower reduction and elongation.

Before calculating the results from the tests, it is important to

110

plot the appropriate correction curve on the graph for the spring beam used. The correction value may be obtained from the manufacturers' literature supplied with the testing machine.
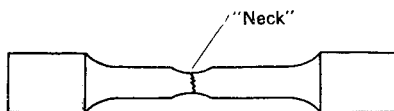

FIG. 4.8. Necking and fracture of a tensile specimen.

AVERY TENSILE MACHINE

The Avery machine, which is self-indicating, applies the load hydraulically. It is important that the correct grips are used to hold the specimen. Wedge grips are commonly used for round specimens, and vary in size according to the diameter of the specimen. When testing specimens of thin sheet or strip metal, special auxiliary holders are usually available. Similarly, when there is insufficient material for gripping, as in the case of short specimens, the ends of the specimens may be threaded and fitted into special holders.
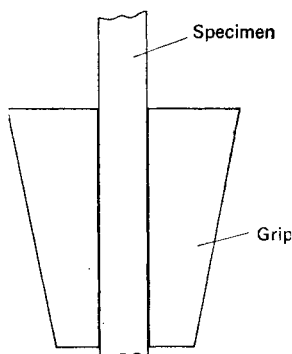

FIG. 4.9. Diagram showing the specimen correctly engaged in the grips for tensile testing.

111

Before commencing the test, it is important to ensure that the specimen is correctly engaged into the grips or holders, as illustrated in Fig. 4.9. The machine should be set to the correct loading range, e.g. 6-ton range for $\frac{1}{2}$ in. diameter mild steel, and the indicator pointers, which indicate the applied load, should be zeroed. The machine has two pointers, one of which is known as the "tell-tale" pointer. If a large number of identical specimens are to be tested, a constant rate of straining may be selected by way of a suitable control valve. Hence all the specimens are strained at the same rate. Within fairly broad limits, the rate of straining has no effect upon the tensile properties of the metal undergoing test. Once the initial strain is applied to the specimen, the rate of loading is carried out automatically, but if it is so desired, the specimen may be held under load at any given point (to take extensometer readings) by simply operating a suitable lever.

When the specimen reaches its yield point, the pointer indicating the applied load stops momentarily, and then continues to rise slowly throughout the plastic range of the specimen. The pointer eventually becomes stationary as the maximum load is reached, and once "necking" commences, it drops slowly back. When the specimen fractures, the pointer will return to zero. The "tell-tale" pointer remains in position, indicating the maximum load that the specimen has endured.

LAMB'S ROLLER EXTENSOMETER

The extensometer consists of two members which are clamped on opposite sides of the test specimen. Each member is in fact an extensometer, and may be used separately; but by using both together as a complete unit, extremely minute extensions of the specimen may be accurately measured. Also an average reading is ensured covering any possible unequal strains due to non-axial loading or lack of homogeneity of the specimen. Errors due to movement of the specimen in the testing machine are also eliminated.

Measurement is made by using a projection lamp or a tele-scope. The latter is useful for measuring very accurately strains of a small magnitude. The lamp is arranged in conjunction with mirrors attached to the extensometer to throw a beam of light
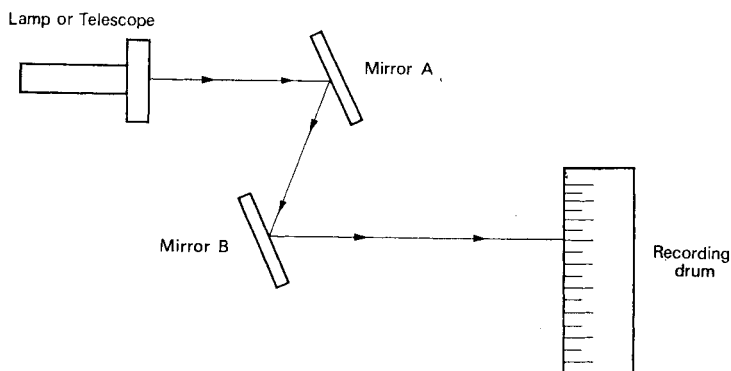

Fɪɢ. 4.10. Lamb's extensometer simplified.

onto a piece of graph paper fixed to a recording drum, as illus-trated in Fig. 4.10. The lamp must be correctly focused until the cross-wires, incorporated within the lamp, are clearly defined on the drum.

Pʀᴏᴄᴇᴅᴜʀᴇ ꜰᴏʀ ᴛᴇsᴛɪɴɢ

The extensometer should be arranged on the specimen, and a light load applied to give an initial reading. This reading is recorded by focusing the cross-wires of the lamp directly onto the graph paper on the recording drum. As the load is applied to the specimen, the drum will revolve to bring an appropriate line on the graph paper into a relative position. The increase in strain moves the projected image of the cross-wires parallel to the axis of the drum. The mirrors attached to the extensometer move in relation to the extension of the specimen. As the load is increased, points should be plotted on the graph paper at suitable intervals (e.g. every 2000 lb. for specimens of 1 in. in diameter)

until the specimen yields. The curve should then be plotted under decreasing loads back to zero. The specimen may be subjected to a maximum load test with the extensometer retained in position if so desired.

<div align="center">TEST RESULTS</div>

For tests on tensile specimens the following measurements are generally made.

(a) The maximum load attained during the test. This is generally transformed into Ultimate Tensile Stress, which may be defined as:

$$\text{U.T.S.} = \frac{\text{maximum load}}{\text{original cross-sectional area}}.$$

From the graph obtained from the test, the area under the curve equals the work done to fracture; for example, a small area would denote little work done as in the case of a brittle material, whilst a ductile material may show a large area under the curve, and hence more work done, as illustrated in Fig. 4.11.

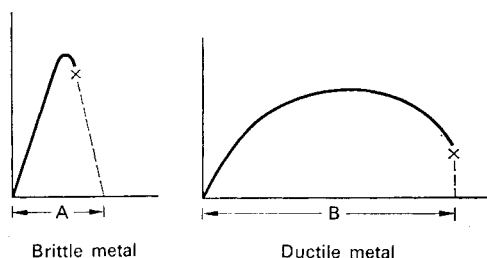

Brittle metal   Ductile metal

FIG. 4.11. Curve showing work done to fracture. A and B represent the work done.

(b) Percentage elongation at the fracture point. This is obtained by fitting the broken halves of the specimen together after the test has been completed and measuring the gauge length.

<div align="center">114</div>

Thus percentage elongation equals

$$\frac{\text{extension of gauge length} \times 100 \text{ per cent}}{\text{original gauge length}}$$

(c) Percentage reduction in area. If the initial cross-sectional area of the specimen is A_0 and the final smallest cross-sectional area is A_1, then the percentage reduction in area is given by

$$\left(\frac{A_0 - A_1}{A_0}\right) \times 100 \text{ per cent}.$$

(d) Young's Modulus of Elasticity (E). This is given by the slope of the initial elastic portion of the stress–strain diagram, and is defined as

$$E = \frac{\text{stress}}{\text{strain}}.$$

It must be remembered that Young's Modulus refers only to the elastic portion of the test specimen, where any deformation is recoverable.

(e) The elastic limit. This is defined as the stress at which appreciable plastic deformation begins. It is indicated by the bending away of the load–elongation curve from the initial linear portion corresponding to the elastic state of the specimen. In mild steels this point is well defined, and the stress may be recorded; but in most non-ferrous materials this bending away is usually very gradual, and the stress used is the **proof stress,** which corresponds to a given small amount of permanent deformation, usually 0·1 or 0·2 per cent of the gauge length.

Compression Test

The compression test is not carried out as frequently as the tensile test, but it still has a place in engineering science, since compressional properties of metals cannot always be deduced accurately from a tensile test. Therefore tensile testing machines

usually have provision for suitable attachments to enable materials to be subjected to compressive loads.

The preparation of test specimens is very important. To determine the general compressive properties of the material, a specimen is usually prepared in which the length is approximately twice its diameter. The end faces of cylindrical specimens should be smooth and preferably polished, to eliminate to some extent friction between the face of the specimen and the face of the pressure block on the testing machine. Specimens must be carefully aligned between the pressure blocks, and should there be a tendency for the material under test to buckle under load, or where excessive deformation of the specimen may impose lateral strain to the testing machine, it is advisable to employ some form of jig to give support to the specimen. These jigs will vary according to the type and size of specimen undergoing test, being usually designed to suit individual requirements. Therefore each test should be considered prior to its commencement.

The direction of the force of load is reversed in comparison to the tensile test, the majority of tests being usually carried out to obtain a stress–strain curve for a particular material which may be compared with the stress–strain curve for the same material in tension. As the load is applied to the specimen, there is an increase in the cross-sectional area of the specimen because of Poisson's ratio effect **below** the elastic limit, and because of plastic yield **beyond** the elastic limit. If the specimen under test is ductile, there will be a pronounced bulge in the middle section because of friction to the faces of the specimen. This bulge is known as "barrelling", and if a lubricant is applied to these faces, this will contribute in reducing friction, and subsequently to a reduction in the "barrelling" effect. Because of this effect, the values usually gained from a test equivalent to percentage elongation and percentage reduction in area cannot be determined, as they can with the results from the tensile test. Furthermore, ultimate compressive strength has no particular meaning, as the increase in cross-section gives a reduction in the **true** stress, as against **applied** stress from the application of the load. A typical curve for a ductile material is illustrated in Fig. 4.12.

116

As the load is applied, the barrelling effect as well as the work-hardening of the specimen serve to raise the load required for further deformation. Thus there is no compressive equivalent to the Ultimate Tensile Stress of a material. Except where fracture occurs, the applied load rises with increasing rapidity, and the limit is set by the load capacity of the testing machine rather than by any property of the specimen. Hence there is a possibility that the testing machine may suffer damage if the test is carried too far. Failure of most specimens under test is usually by shear
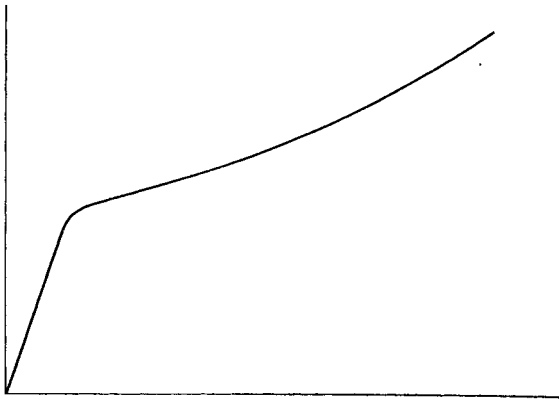

FIG. 4.12. Example of a typical compression curve.

at approximately 45° to the axis of the load. With brittle materials in particular, sudden fracture may cause the broken specimen to be ejected from the testing machine. To avoid injury, therefore, the operator should not place himself directly in line with the specimen, but in a safe position where injury will be avoided.

The conclusions made from test readings of a brittle material decide the maximum load the specimen will endure. When the maximum load figure is divided by the original cross-sectional area of the specimen, the compressive strength is obtained. It should also be remembered that during the early part of the test, strain should be measured between gauge points on the specimen, and not between the pressure blocks of the machine.

Torsion Test

The torsion test is usually used for quality tests on metals for such components as axles, shafts, twist drills, and on materials at elevated temperatures to determine forgeability.

The machine used to apply the load consists basically of a rotating head, to which is attached one end of the specimen, gripped in a suitable chuck. The chuck supplies the necessary twisting motion by way of an electric motor. The other end of the specimen is also gripped in a chuck, which has provision for carrying weights, and for measuring the applied torque. The measurement of specimen deformation is made by an instrument called a Troptometer, which indicates the displacement of a pointer at one end of the specimen in relation to a similar pointer at the opposite end. The specimen must be arranged in the machine so that its axis coincides with the axis of the rotation of the machine.

The test specimen is usually circular in cross-section. Thin-walled tube is preferable because it provides a more uniform stress distribution throughout its cross-section. The cross-section does not change during the test; thus the properties of the material rather than the actual thickness are subjected to the test. If it is too thin, there is a possibility that the tube may collapse under the applied load, instead of failing through direct torsion. The test specimen should have a portion machined to a smaller diameter than the ends of the specimen that are gripped in the chucks.

From the results gathered from the test, the modulus of elasticity in shear and the shearing yield strength may be determined, but it is the ultimate shearing strength of the material that is usually considered. The curve plotted from the test usually closely resembles the familiar stress–strain curve, but the test in general accentuates the ductile characteristics of a metal.

The Notched-bar Impact Test

When any mechanical test is performed upon a metal, the behaviour of the metal will depend to some extent on the speed of testing. Under slow deformation, e.g. the tensile test, a metal may show considerable elasticity, whilst a sudden shock will produce a brittle fracture. It is also true to say that although a metal may exhibit a satisfactory elongation and strength in the tensile test, it need not show these qualities under conditions of impact, where the loading is very fast and where, in addition, the specimen has a stress-raiser in the form of a notch cut into it. The purpose of the notch is to introduce a high state of localized deformation in the specimen and accentuate any brittle potentiality of the material. Despite the fact that the notch is an artificial addition, notches of some sort frequently occur by design or accident in components in service, so the provision of the notch can give valuable (though approximate) indications of service behaviour. Although the notched-bar impact test does not give the fundamental information that can be obtained from the tensile or compression test, it serves a vital need in indicating the probable behaviour of a metal under shock conditions. Materials used for important parts of machinery such as shafts and axles in motor vehicles are usually subjected to this test. Failure of these parts while they are in service may cause loss of life.

This test can only be expected to indicate whether a particular metal is brittle or not at the temperature at which the test is carried out and with a notch of certain dimensions. Within these limitations it gives a useful basis for comparison, being particularly valuable in the selection of metals for use at low temperatures. Under these conditions of testing, temperature is one of the most important variables. Some metals exhibit a sudden change from a tough behaviour to a brittle behaviour as the temperature is lowered. The transformation temperature varies considerably from metal to metal and may be influenced considerably by the presence of alloying elements, or by the

119

heat treatment of the metal prior to testing and this applies particularly to steels. If successive specimens are broken at different temperatures, and a graph of energy versus temperature is plotted, as illustrated in Fig. 4.13, it is quite possible to determine that a transition temperature range often exists above which a metal is ductile and below which a brittle fracture will occur.
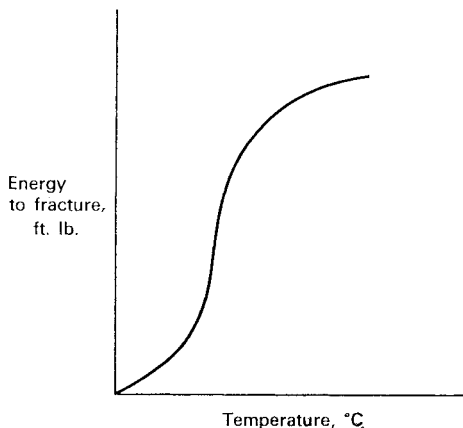

FIG. 4.13. Typical impact test curve.

The notched-bar impact test consists of bending a test specimen through a right angle in a suitable machine. The two most common tests are the Charpy and the Izod, both of which use a weighted swinging pendulum to fracture the notched specimen. The energy absorbed by the specimen during fracture is registered by a suitable instrument contained within the testing machine. There is a third test, which is being used more frequently, known as the V-notch Charpy test. Let us now consider the basic principles of each test.

CHARPY TEST

In the Charpy test the specimen is supported at both ends, as a beam against an anvil. It is broken by a single blow from a free-

falling pendulum, which strikes the middle of the specimen. As the pendulum completes its swing, the energy absorbed by the specimen is indicated. Usually the specimen breaks at the notch and the two halves will tend to be ejected from the machine. In Fig. 4.14 is illustrated the specimen used in this test, the dimen-
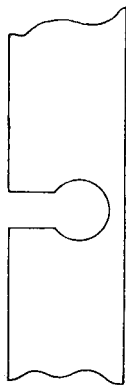

FIG. 4.14. The Charpy impact specimen. Note the shape of the notch.

sions of which must conform to the specification laid down for impact specimens. (This applies equally for all types of impact specimens.) The notch may be formed by accurately drilling a hole in the appropriate part of the specimen, and then by carefully cutting a slot to meet the hole, using a suitable cutter in a milling machine.

IZOD TEST

The machine used in the Izod test is very similar to the Charpy. In this test the specimen is clamped in a vice in a horizontal position with the centre of the notch in line with the upper face of the vice jaws and only one-half of the specimen is broken by the action of the pendulum. Figure 4.15 illustrates the principle of the machine. The notch may be machined in a similar manner to that formed in the Charpy test specimen.
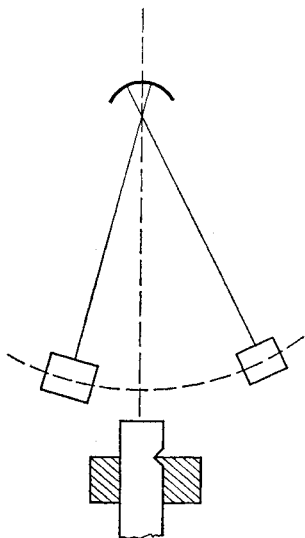
121

FIG. 4.15. Principle of the Izod impact test machine. The weighted pendulum swings from the raised position and strikes the notched specimen held in a vice. The energy absorbed to fracture the specimen is recorded on a scale.

HOUNSFIELD BALANCED IMPACT MACHINE

This type of machine also uses a weighted pendulum, which is divided into two, as shown in Fig. 4.16. One part of the pendulum moves in the opposite direction to the other. The test specimen,

FIG. 4.17. Hounsfield impact specimen.

illustrated in Fig. 4.17, is supported in the centre of one of the pendulums. When the pendulums are released from the raised position, the specimen is broken against two anvils which are

fitted to the second pendulum. The energy absorbed in fracturing the specimen is registered on an indicator, and if the value indicated is multiplied by 2·4, the result obtained will compare favourably with an equivalent Izod test result. The specimen used in this machine may be "notched" using a special notching machine supplied by the manufacturers.

It is important to remember when preparing test specimens of either of the types illustrated that if the notch is not cut to the correct depth, the energy absorbed by the specimen may be unusually high, and the test ceases to be comparative. This is because the shallow notch has restrained the specimen in fracturing, and in the case of the last two tests described above, the specimen will be driven violently through the anvils. Always ensure that the correct notched specimen is used for the particular test employed. Also care must be taken when placing the specimen in the machine. It is probably true to say that negative results can usually be attributed to the neglect of one or both of these factors.

Prior to commencing the actual test, certain safety precautions should be observed. Specimens that have been subjected to high or low temperatures should be transferred to the testing machine as quickly as possible, and must be handled with a pair of tongs or a gloved hand. Should an operator during a transfer operation drop a specimen that has previously been placed in a container of liquid air to reduce its temperature, it must not be picked up with the fingers or severe burns may be caused.

The testing machine should never be tampered with or used without first considering and understanding its method of operation. Accidents can usually be attributed to carelessness on the part of the operator, who has not really understood how the machine should be operated. Vessels containing liquids in which the specimens are placed to change their temperature from room temperature may contain solid CO_2 and acetone ($-83\,°C$) or liquid air ($-183\,°C$) and should never be placed in such a position that they may be broken by the action of the weighted pendulum. Similarly, if the impact machine is used on a bench in the laboratory, and the test is being carried out by students as a

group practical experiment, great care should be taken. It is possible that persons engaged in writing up notes relating to the test may be standing at a part of the bench directly in the path of the swinging pendulum, and serious injuries may result.

Creep Test

The mechanical tests dealt with up to now have given information as to the behaviour of a metal which has undergone testing for a short duration of time. If a load is applied to a metal at various temperatures, and is retained for a long period of time, the metal may show evidence of having undergone a gradual extension. This phenomenon is known as creep, and by this process a metal or component in service may fail under a particular stress which is well below its ultimate tensile stress.

The ability of a metal to creep could present a difficult problem, particularly when considering the design of a chemical plant or furnace equipment which operates at elevated temperatures. Soft metals such as lead will even creep at room temperature. An example of the ability of lead to creep becomes apparent when lead sheeting that has been used as a protection for roofs of buildings over a period of many years is examined. Because the lead has literally crept under its own weight it will be found to have become thicker at the edge of the roof than at the top.

The specimen used in the creep test is similar to the tensile test specimen, but usually with a much greater gauge length. Long gauge lengths are preferable, because very small percentages of strain may be measured. Of course, there is a limit as to the length of the specimen, this usually being governed by the size of the furnace equipment available. Consideration must also be given to temperature control. The furnace may be required to operate up to a temperature in the region of 900 °C in certain instances, and it must be capable of being controlled very accurately (to within $\pm\frac{1}{2}$°C) for long periods of time. The time factor may extend in some cases to several months. For details regarding the construction of a suitable furnace which may be built into

testing apparatus, reference should be made to Chapter 2. Difficulty may be experienced in maintaining a constant uniform temperature over the gauge length of the specimen, hence quite elaborate precautions are necessary for accurate creep work. It is preferable to have the testing laboratory completely free from draughts, and the laboratory temperature should be thermostatically controlled. The equipment used to control both the laboratory and the furnace temperature must be of a high quality and absolutely reliable in operation.

The limit of creep at any particular temperature is usually governed by the stress below which no **measurable** creep takes place. To measure creep of such a low magnitude, an extremely sensitive recording apparatus is required, and because measurement is solely dependent on the degree of sensitivity, there is no absolute guarantee that creep will not take place at even lower stresses. The load may be applied to the specimen by way of a suitable weighted bar which has provision for carrying extra weights. Measurement of the extension of the specimen may be made by using an extensometer, and the optical type, which allows very accurate measurements to be made, is generally used. In the Barr–Bardgett test, the load is applied to the specimen by a weight bar and a screw mechanism. The stress, as applied to the specimen, is measured on a dial gauge, and as the metal creeps, the relaxation in the strain is recorded. By performing several tests at different temperatures and constant stress, and extrapolating the results back to zero relaxation, the maximum permissible temperature for a given stress may be found. Alternatively by using various stresses at a constant temperature, the maximum load for a given temperature is determined. This technique is very useful for relatively short laboratory tests.

From the results obtained during a test, a graph as shown in Fig. 4.18 may be plotted which may be analysed as follows. The initial stage of Decreasing Strain Rate is due principally to the normal action of stress on a metal, i.e. elongation accompanied by work-hardening, until the latter balances the load. The second (and by far the longest) stage of Constant Strain Rate is due

125

to the viscous flow of the metal. The grains very slowly slip over or past one another, causing a slow overall extension. Such motion, which is entirely specific to creep at high temperatures, is permitted by a broadening of the boundaries between the grains within the metal. The third stage of Increasing Strain Rate,
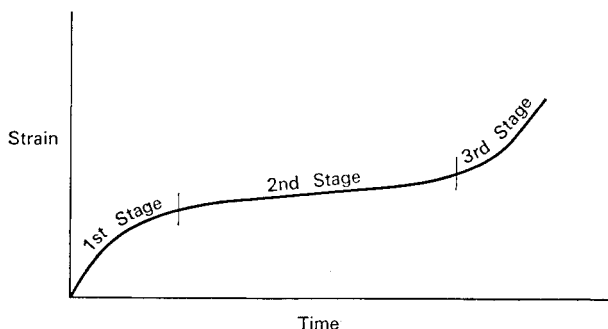

FIG. 4.18. Typical graph of creep test results.

leading to fracture, is thought to be due to voids opening up in the grain boundaries after an excessive amount of viscous flow. Such voids act as stress raisers and in addition decrease the effective cross-section of the specimen, so that the process "snowballs"; the increased load results in faster creep and more voids form, which in turn still further increases the load. This process continues until fracture occurs.

Fatigue Test

A material that is subjected to repeated alternating stresses may fail under a considerably lower load than normal through a process known as fatigue. The greatest number of failures are attributable to the mechanism of fatigue. The danger of fatigue is that there is no prior warning or indication that a component is about to fail. Cracks which ultimately lead to a fracture are fine and extremely difficult to detect, but as they grow in pro-

portion they spread quite rapidly through the component, causing failure in a relatively short time. The cracks usually start at the surface and progress steadily and undetected through the metal. Fatigue cracks will also propagate from regions of high stress concentration, which may be caused by a keyway, a hole, a groove, very bad tool marks, a defect in the microstructure of the metal, or a change in the diameter of a shaft. Hence it is of the utmost importance when designing components to ensure that if, for example, there is to be a change in diameter of a shaft, there is an adequate fillet which is not too sharp in the appropriate place.

The fatigue limit of a material is determined as the maximum range of stress that it will endure, without fracturing, after an infinite number of reversals. In practice, 10^7 reversals is taken as a working interpretation of "infinite"; if a specimen can withstand this number of reversals without fracture, then, except in special circumstances, it is considered to be "safe". Testing machines may subject a material to continuous vibrations of varying intensities, to determine the fatigue limit, the stress involved being less than that required to cause failure from a single, steady pull. In determining this limit, a number of specimens are tested by repeated cycles or vibrations at a known range of loads (if possible the load should be similar to that which the component may have to endure in service), until eventually fracture occurs or the specimen attains a "life" without fracturing. From the results gathered from these tests a curve may be plotted for stress versus the number of reversals or vibrations that the specimen has endured. These results are only an approximation, and should only be used as a guide to determine a safety factor for the material. For ferrous metals, the curve usually becomes horizontal at one particular stress, which is referred to as the fatigue or endurance limit; there is no fatigue limit for non-ferrous metals. Typical examples of fatigue curves for a ferrous and a non-ferrous metal are shown in Fig. 4.19.

It is **vitally important** that test specimens should have a surface finish that is **absolutely free** from blemishes and machine tool marks; such imperfections act as stress-raisers and drastically reduce or completely obscure any fatigue limit the metal may

127

possess. For the same reason any risk of corrosion, which results in surface pitting and irregularities, should be rigorously excluded from the specimens. Specimens are usually attached to the testing machine by suitable chucks, or they may be threaded at each end for fixing purposes. To prevent possible failure at that part
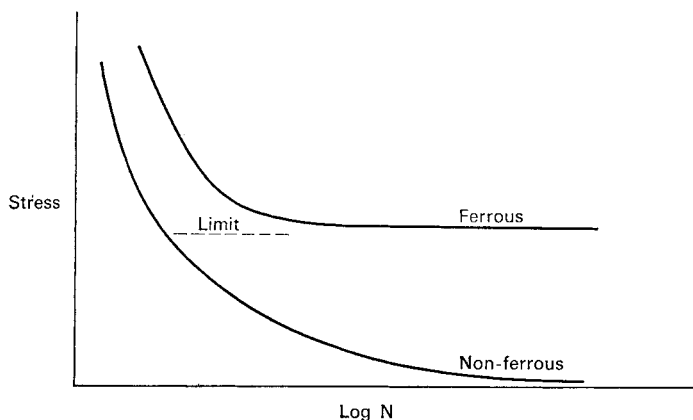

Fɪɢ. 4.19. Examples of fatigue curves for a ferrous and a non-ferrous metal.

of the specimen attached to the machine, it should be machined with a larger section than the portion under test. To reduce stress concentrations between the ends and the test portion, large radii should be provided. Figure 4.20 illustrates the general shape of the specimens used in the Wöhler and Haigh machines. In the latter case, the diameter of the parallel section should not be less than $\frac{1}{4}$ in. to prevent possible distortion or buckling of the specimen when it becomes loaded in a compressive state. Wherever possible it is desirable to test actual components. There are many such components that may be used, namely springs, shafts, bolts and components that have riveted or welded joints. Suitable fixtures will be required to attach them to the machine.

There are various types of testing machines available for use in the laboratory. In some instances special apparatus may be
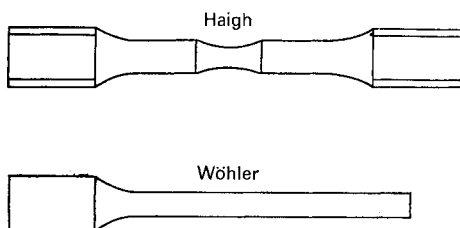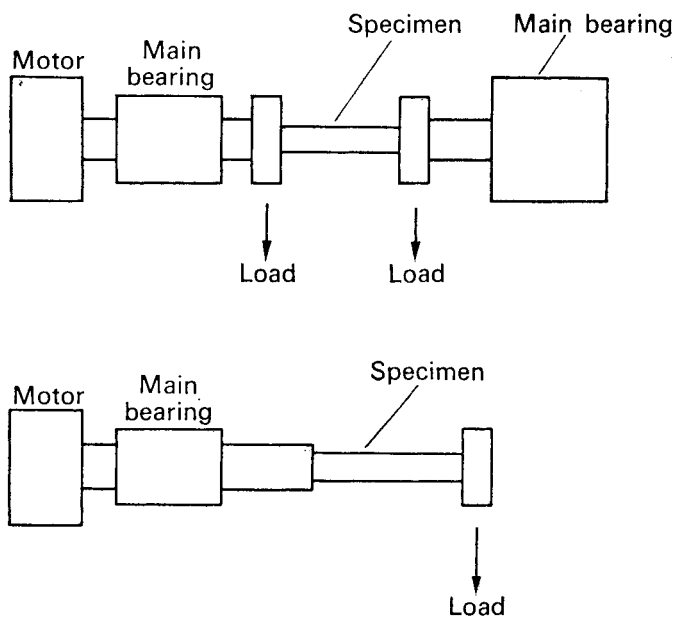
FIG. 4.20. Haigh and Wöhler test specimens.

FIG. 4.21. Shows the Wöhler fatigue machine in diagrammatic form.

constructed, in which a component or specimen may be sub-
jected to fatigue stresses. An example of this is the type of
apparatus constructed a number of years ago to solve fatigue
problems that were occurring in aircraft at that time. In this
instance, a section of an aircraft was arranged in a test rig which
simulated the stresses and strains encountered during actual
flight conditions. After testing over a period of time, part of the
aircraft structure suffered a fatigue failure. For general laboratory
applications, a common type of testing machine is the Wöhler
machine. This machine uses a revolving specimen, in the form of
a cantilever, and carries a load which acts through a ballrace in
one direction, and is shown in Fig. 4.21. The direction of stress
in the specimen is reversed each time the specimen is turned
through 180°. Hence its outer surface is in tension in one instant,
then in compression. A counter is used to register the number of
reversals that the specimen has endured. This type of test has the
disadvantage that the stress varies along the length of the speci-
men, and must be calculated by formula. By supporting the
specimen at both ends and hanging the weight from two points
in the middle, the stress between these points may be taken as
constant to a first approximation.

HAIGH TESTING MACHINE

The Haigh testing machine is operated electromagnetically.
Two electromagnets are supplied with an alternating current
which is 90° out of phase. This produces an alternating force on
an armature, supported between two magnets, which is trans-
mitted to the lower end of the specimen. The upper end of the
specimen is held in a "head" which is bolted to the main frame
of the machine. One end of the armature is also connected to the
main frame by way of springs which are used to apply the load
to the specimen. The stiffness of the springs may be adjusted so
that the resonant frequency will correspond with the frequency
of the load cycles. Hence any inertia forces which would other-
wise affect the calibration of the machine are eliminated. The

load is measured by the voltage which is induced in the secondary coils of the armature, and the voltage reading is calibrated by making a comparison with a measured deflexion of a weightbar, which is substituted in place of the test specimen.

A specimen that has fractured through fatigue has a characteristic type of surface which consists of two definite areas, as illustrated in Fig. 4.22. One is relatively smooth and somewhat
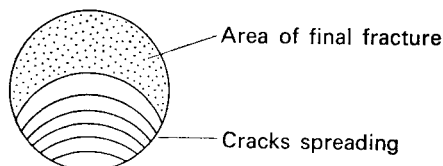

Fig. 4.22. A typical fatigue fracture.

burnished in appearance, showing fine, ripple-like marks which tend to travel out from the centre of any crack formation. These markings trace the growth of the fatigue crack throughout the life of the specimen. The other area is rather coarse and crystalline in appearance, and indicates the final fracture of the remaining cross-sectional area, which was unable to withstand the varying stresses.

CORROSION FATIGUE

If a component fails through a corrosive action at the same time as a fatigue stress, the mechanism is known as **corrosion fatigue.** Common types of steels usually corrode rapidly unless suitably protected, e.g. heavily greased, painted, or alloyed. Some metals, however, have a certain amount of natural resistance to corrosion, but should the metal be subjected to corrosive atmospheres, corrosion pits may form, usually over a period of time. The action of the metal under alternating stresses may break down this natural resistance at individual pits and hence corrosion will continue unimpeded, and even deeper pits will be formed. As a result the metal is weakened in some regions, and intense stresses are produced at these places.

131

Other Tests

THE ERICHSEN CUPPING TEST

This test will determine whether a sheet of metal is sufficiently ductile to be satisfactorily drawn or pressed. Ductility is usually measured by determining the depth to which a round-headed plunger can deform the metal, held in suitable clamps, before it tears. The appearance of the dome or cup produced is important. A rough surface indicates that the metal has a large grain size. If cracks occur in one direction only, then a variation in ductility is indicated.

THE SIMPLE BEND TEST

This test may be performed on wire, tube or sheet to determine ductility. The test usually consists of recording the number of reverse bends the metal will endure before fracturing.

THE TRANSVERSE TEST

A transverse test is usually applied to cast iron specimens. A standard specimen, supported at each end, is loaded in its middle portion until it fractures. The load required to fracture the specimen and its maximum deflexion under load are measured. The result, known as the transverse rupture stress, may be determined from the expression

$$\frac{WL}{0 \cdot 4d^2}$$

where W = the load required to break the specimen, L = the length that is supported, and d = the diameter of the specimen. For further information regarding the above tests, reference should be made to such works of reference as the *ASM Metals Handbook*.

Hardness Test

Hardness tests are usually carried out on metals because they are simple tests, are virtually non-destructive and also because they may be closely correlated with the tensile strength and yield strength of the metal. In steels the tensile strength and yield strength are directly proportional to the hardness of the metal. Hence these properties may be quickly assessed by this type of test.

A hardness test is usually performed by pressing an indenter into the surface of a specimen under the application of a known load. The indenter is usually of a fixed and known geometry and the load is applied to the specimen either directly or by way of a system of levers. The indentation produced in the surface of the specimen is measured after either partial or the complete load has been removed. Thus elastic recovery is permitted; it should be mentioned that measurement of the indentation does not represent its actual size under the applied load, but a measurement of the recovered indentation. Depending upon the type of test employed, the hardness value is expressed by a number which is either proportional to the depth of the indentation for specific load or indenter, or proportional to a mean load over the area of the indentation. For testing hard materials, a diamond indenter is preferable because it does not become deformed, whereas steel indenters are apt to deform themselves under high loadings on hard specimens. Such deformations lead to inaccuracies in the calibration of the hardness tester.

BRINELL HARDNESS TEST

The Brinell hardness-testing machine normally consists of a hand-operated hydraulic press which has been designed to force a small hardened steel ball into the surface of the specimen under a predetermined load. Standard test procedure requires that the test should be made using a ball 10 mm in diameter, under a

133

load of 3000 kg for ferrous metals, or 500 kg for non-ferrous metals. When testing ferrous metals, the loaded ball should be pressed into the surface of the specimen for 15 seconds, while for non-ferrous metals the time factor is 30 seconds. The diameter of the impression produced is measured by using a microscope containing an ocular scale, which is usually graduated in tenths of a millimetre. To ensure that the desired load is not exceeded, the maximum pressure may be controlled by way of a weighted yoke. Hence should the maximum load be 3000 kg, weights to this value may be placed equally on the two pans which hang on either side of the yoke. Once the maximum load has been attained by way of the hydraulic pump, a piston raises the weighted yoke, which then "floats". All that remains is to keep the piston in the raised position by occasionally operating the pump. Once the test has been completed, the hydraulic pressure may be released by operating a pressure release valve.

The reading obtained from a test is converted to a Brinell hardness number (Bhn) which has been previously defined as the ratio of the load, in kilograms, to the area of the impression in square millimetres. This definition may be calculated from the formula

$$\text{Bhn} = \frac{P}{(\pi D/2)\,[D - \sqrt{(D^2 - d^2)}]}$$

where P = load applied in kilograms, D = the diameter of the ball in millimetres, d = the diameter of the impression in millimetres. Calculations using this formula are usually unnecessary for each test because tables are available which allow the reading obtained for the depth of the impression to be directly converted into a Brinell hardness number. The use of two loads for one ball diameter limits this test to relatively large specimens. Should a test be carried out on small specimens, then a ball of smaller diameter should be used in conjunction with a lighter load. The manufacturers of the testing machine usually supply tables which give the various combinations of ball diameters and corresponding loads that should be applied.

When the indenter is forced into the surface of the specimen,

the ball usually deforms slightly. The amount of deformation is dependent upon the hardness of the metal and the magnitude of the applied load. A Brinell hardness number of approximately 525 is the upper limit that may be used with an ordinary steel ball, but by using a Hultgen ball, the hardness number may be increased to 600. By using a tungsten ball the hardness number may be increased to 725.

<div align="center">ROCKWELL TEST</div>

In the standard Rockwell test, which originated in the United States of America, the specimen is placed surface uppermost on an anvil which is slowly raised by way of a handwheel until the surface of the specimen just touches an indenter. By raising the anvil still further, a **minor** load is applied, causing an indentation in the specimen. This load of approximately 10 kg is indicated on a dial indicator, and also sets the dial to zero. Whilst this minor load is in operation, it is augmented by a **major** load by operating a suitable lever which will slowly move in a downwards direction. This operates the mechanism in the machine and increases the depth of penetration of the indenter. The rate of loading is controlled by a dashpot, and when the lever applying the major load has completely descended, the reading on the indicator becomes steady and indicates the total load that has been applied to the specimen. The major load is then removed, still retaining the minor load as illustrated in Fig. 4.23. Hence the
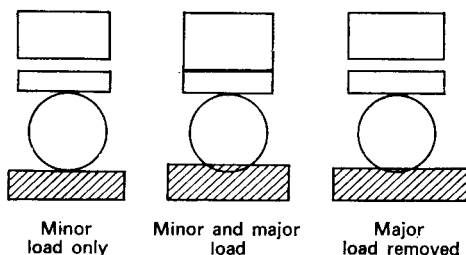

Minor load only   Minor and major load   Major load removed

FIG. 4.23. Demonstrates the principle of the Rockwell hardness test.

<div align="center">135</div>

specimen is allowed to recover elastically. The relevant hardness number is read directly on the dial indicator, having been derived from the measurement of the depth of the impression by the formula $H_R = E - e$, where e is the difference between the depth of penetration before and after the application of the major load and whilst the minor load is in operation. This value e is expressed in units of 0·002 mm. E is an arbitrary constant; its value will depend upon the type of indenter used (reference should be made to Table 13) whilst H_R is an abbreviation for Rockwell hardness.

The types of indenters which may be used are a steel ball of $\frac{1}{16}$ in. in diameter or a diamond cone with a point, rounded to a radius of 0·2 mm. The various loadings are 60, 100 and 150 kg. The most common combinations used in the standard test, which serve to measure the hardness of all the common metals, are the $\frac{1}{16}$ in. diameter ball used with a minor load of 10 kg and a major load of 90 kg, making a total of 100 kg, or the diamond cone used with a minor load of 10 kg and a major load of 140 kg, making a total load of 150 kg. Typical combinations are illustrated in Tables 13 and 14. Because there is more than one combination available, the dial indicator is divided into two scales designated B and C, whilst in some instances there may be a third scale available. Should the accuracy of the test be impaired by the fact that the specimen has been case hardened, having an underlying softer material, then use may be made of this third scale, designated A. These three scales cover the requirements for the standard test; however, in some instances it may be desirable to use some other scale. Hence additional scales designated D, E, F, G and H are available, and may be used with the combinations of indenters and loadings as given in Table 13. If the diamond cone was used to produce the impression, the direct reading of the hardness of the specimen would be referred to as, for example, H_R C60. Should the $\frac{1}{16}$ in. diameter steel ball be used, then the reading would be, for example, H_R B75.

TABLE 13

Scale	Indenter	Minor load (kg)	Major load (kg)	Total load (kg)	Value of E
A	Diamond cone	10	50	60	100
B	$\frac{1}{16}$ in. dia. steel ball	10	90	100	130
C	Diamond cone	10	140	150	100
D	Diamond cone	10	90	100	100
E	$\frac{1}{8}$ in. dia. steel ball	10	10	100	130
F	$\frac{1}{16}$ in. dia. steel ball	10	50	60	130
G	$\frac{1}{16}$ in. dia. steel ball	10	140	150	130
H	$\frac{1}{8}$ in. dia. steel ball	10	50	60	130

TABLE 14

Scale	Indenter	Total load (kg)	Material
A	Diamond cone	60	Thin hardened steel strip, extremely hard materials when small impressions are required
B	$\frac{1}{16}$ in. dia. steel ball	100	All mild steel and medium carbon steels, soft materials
C	Diamond cone	150	Hardened steels, hardened and tempered steels, alloy steels

VICKERS PYRAMID HARDNESS TEST

The Vickers pyramid hardness-testing machine, illustrated in Fig. 4.24, is suitable for measuring the hardness of all types of materials, from thin sheet to very large sections of metal or complete components. This is because of the wide range of loads that may be applied and the shape of the indenter, which in this case is a diamond pyramid that has an included angle of 136° between opposite faces. The machine is semi-automatic in operation, because after the surface of the specimen or component has been brought into contact with the indenter, the preset load is applied for a definite period of time and is then automatically removed.

Operation. A weight carrier is loaded with any of the weights selected from those which are supplied with the machine to suit test requirements. For most tests, a load of 30 or 50 kg is usually applied, but in certain instances, for example when testing non-ferrous metals, it is often necessary to use a lighter load. The specimen should be placed on the table of the machine, which may be raised by way of a handwheel, until the surface of the specimen is just clear of the point of the indenter. A lamp incorporated in the machine may be used to facilitate this adjustment. At this stage a pedal may be depressed in order to load the machine. If the operator has left too wide a gap between the specimen and the indenter, the test would not proceed satisfactorily. Therefore a small buzzer automatically comes into operation to indicate this error. A small lever fitted to the machine should be depressed to release the loading mechanism, which then operates automatically; an auxiliary weight and cam action allows the load to be applied slowly. Hence any errors that may arise from an inertia effect or any sudden impact from the application of the load are eliminated. An oil-dashpot controls the duration of the load as applied to the specimen, which may vary between 10 and 30 seconds. An audible "click" will eventually be detected which indicates that the test has been completed.

The impression in the surface of the specimen is measured by either (a) swinging a microscope over the specimen to measure across the diagonal corners of the square impression, or (b) the machine may be equipped with a sliding table, which may be moved sideways into position under the microscope. The impression produced by the indenter in the surface of the specimen
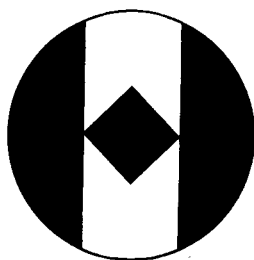

FIG. 4.25. Impression of Vickers pyramid indenter as seen through the microscope.

may then be observed through the microscope. By means of a knurled thumbscrew, a knife edge is adjusted to correspond with the left-hand corner of the impression. A micrometer screw connected to a counter adjusts a second knife edge to correspond with the right-hand corner of the impression. With both adjustments completed, the impression as viewed through the microscope should appear as shown in Fig. 4.25. The figures shown on the counter are converted directly into Vickers pyramid numbers by consulting the tables supplied by the manufacturers of the testing machine.

There are, however, advantages and disadvantages concerning these common types of hardness tests. The Brinell test is an established test which is generally limited to testing large specimens. The large indentation produced from the 10 mm ball with a load of 3000 kg is an advantage when the material under test is not uniform. Hence should the indenter penetrate an inclusion, it will still be possible to obtain an accurate measurement of hardness, because the indentation covers a reasonably large

139

area. In the case of the diamond pyramid indenter, the material needs to be more uniform because of the small size of the indenter and consequently the smaller indentation produced. Thus if the indenter penetrates an inclusion, an inaccurate measurement will be obtained, because the indentation produced covers a very smaller area.

The Shore Scleroscope test

The Shore Scleroscope consists of an instrument which measures the hardness of a material by measuring the rebound of a diamond-tipped plunger, which is allowed to fall under its own weight and strike the surface of the specimen or component. The plunger operates within a vertical glass tube, which has a suitable scale engraved upon it for measuring the height of the rebound. A dial gauge may be used to measure and record the height of rebound if so desired.

This test is especially suitable for measuring the hardness of smooth or precision surfaces that must not be marked by any indentation, however minute, as would be the case with some types of hardness test. Should this test be carried out on a specimen or component which has a rough surface, then low readings will be obtained. Hence the surface condition of the specimen or component is very important when this test is employed.

Methods of Non-destructive Testing

It is not the intention of the author to describe in a book of this length all the facts and techniques related to the testing of metals by non-destructive methods. For those sufficiently interested in this vast subject, further reading of suitable literature is recommended, and reference may be made to the Bibliography at the end of this book. However, the more common types of test will be briefly outlined below. These tests are useful for the examination of such components as castings, forgings or welded

joints, to detect such imperfections as inclusions, cracks, or holes. Even though mechanical tests may indicate some possible defects by giving unusual test readings, the exact location or nature of the defect will not necessarily be easily obtained.

FLUORESCENT TEST

This test is very useful for testing non-ferrous metals. A fluorescent substance is usually painted or sprayed onto the component, whilst in some instances the component may be completely immersed into the substance. It is important to remember that any possible cracks must absorb the fluorescent substance; none must be left on the surface of the component. The surface may be cleaned by a water spray, having allowed time for the fluorescent substance to penetrate, and then thoroughly dried prior to actual inspection. The component is subjected to inspection under an ultra-violet light, and any cracks that are present are revealed by the glowing of the fluorescent substance.

MAGNETIC TEST

Magnetic tests may be carried out on ferrous metals. The component is first magnetized, and should there be any cracks present, then the two sides of the crack become opposite magnetic poles. When fine iron powder mixed with paraffin is poured over the surface of the component, the iron powder will cling to the edges of the crack or cracks, forming an easily recognizable dark line. This dark line indicates and determines the extent of the crack. Any non-magnetic inclusions in a component may also be revealed by this method. After the test has been completed and conclusions have been made as to the extent of any crack formation, the component may be demagnetized.

X-ray test

In this test a beam of X-rays is passed through the metal under test. The metal is then either (a) shown as a shadow on a photographic material or (b) shown on a fluorescent screen. If the structure of the metal is uniform and free from flaws or imperfections, the X-rays will be absorbed evenly by the metal. Hence the shadow or picture will reveal a perfectly satisfactory metal. However, should there be any imperfections in the metal, then these are revealed because the X-rays are not evenly absorbed, and consequently the imperfections are represented in the resulting picture as light or dark areas, according to the nature of the imperfection. This type of test is especially useful for such components as castings or welded parts. Also it may be used solely as a means of inspection for finished components because it is 100 per cent non-destructive.

For anyone engaged on this method of testing, stringent safeguards are essential. The effects of X-rays are cumulative, hence the operator should never be exposed to the main beam of rays, or be subjected to prolonged exposure of scattered radiation. The rules and regulations laid down for operators of X-ray machines must be adhered to at **all** times.

Ultrasonic test

In this test, inaudible sound waves of very short wavelength are sent through the metal which is under test. They are reflected back, or become altered by defects in the metal, and are recorded by a suitable instrument, e.g. a cathode-ray tube. Even though this type of testing has not been fully developed, it has been established that the position of defects can be estimated with reasonable accuracy and fine cracks can be detected better than by using X-rays.

Analytical Testing

Analysis as carried out in a metallurgical laboratory is a specialized branch of general chemical analysis; therefore most details can be found in general chemical text books on the subject, as well as in books written purely for the metallurgist. Analysis can take place at three different levels:

(a) Spot tests. These are designed to indicate the presence of *one* particular element. (See Bibliography.)

(b) Qualitative analysis. This should reveal all the elements present, but not their relative amounts.

(c) Quantitative analysis. This is used to find out accurately the amount of or all of the elements present in the alloy. Since the methods used in quantitative work may vary depending on other elements present, qualitative analysis is frequently necessary before quantitative estimations can begin.

Qualitative metallurgical analysis is often aided by certain safe assumptions, and by the appearance of the specimen, either under the naked eye or through the microscope. For instance, with experience, the carbon content of an annealed steel can be estimated almost quantitatively from microscopical examination, and on a qualitative basis it is safe to assume that any ordinary grade carbon steel has in addition a quantity of silicon, manganese, sulphur and phosphorus. The colour and density of a specimen are also valuable pointers, especially in conjunction with metallographic examination; bearing metals, many brasses, and some light alloys can be roughly identified in this way.

On the other hand, quantitative techniques are often lengthy and elaborate, partly because the analyst is frequently concerned in estimating very small amounts of elements, e.g. sulphur or phosphorus in steels, or lead in brasses.

This short chapter gives a simple concise introduction to analytical chemical techniques which, since the equipment required is simple and readily obtainable, are still the most widely used. The section deals with the practical aspects and further information concerning the theoretical background can be ob-

143

tained from more extensive works such as Vogel and Naish, Chemell and Kingsford (see Bibliography).

There are many sophisticated analytical instruments now available, including the potentiostat, the absorptiometer, and the vastly expensive electron probe analyser. These all involve highly specialized techniques, each with their own literature, which could not possibly be considered in adequate detail here.

Qualitative Analysis

DISSOLVING THE ALLOY

1. Alloys which are attacked by dilute hydrochloric or nitric acid. Dissolve 1–2 grams of the sample, preferably as turnings, in a few c.c. of the liquid. Add a little concentrated hydrochloric acid and evaporate until dry. Add a little more of the concentrated hydrochloric acid, and again evaporate to dryness. Redissolve in water and heat to boiling point. Transfer to a test tube and filter whilst the solution is still hot. The residue may contain silver, or the chlorides of silver. The warm liquid should be transferred to another test tube, and agitated with a glass rod. If a white precipitate forms on cooling, this may indicate chloride. Therefore the precipitate should be filtered and retained for further examination.

2. Alloys which are NOT appreciably soluble in dilute acids. Initially determine the effect of concentrated acids on the alloy. Should a residue remain on the completion of this treatment, then separate the residue, and boil it in a little aqua regia. Aqua regia is a mixture, in the ratio of 2 to 1, of concentrated hydrochloric and concentrated nitric acid. Having boiled the residue, evaporate until dry and if necessary repeat the treatment with the aqua regia. Add a few drops of concentrated hydrochloric acid to the residue and evaporate until it is dry. Place the residue into a little boiling water and filter whilst it is still hot. Examine residues according to Group I table.

144

PRIMARY SEPARATION INTO ANALYTICAL GROUPS*

Pour a few drops of dilute hydrochloric acid into about 5 ml. of the cold solution in a test tube. If no precipitate is formed, rub gently with a glass rod, or vigorously shake the test tube.

RESIDUE	SOLUTION						
The residue may contain chlorides of lead, silver and mercury. Examine for this from the table for Group I.	Dilute the solution to about 10 ml., heat until it just boils, saturate with hydrogen sulphide, filter, dilute, pass hydrogen sulphide again and filter.						
	RESIDUE	SOLUTION					

	The residue may contain the sulphides of mercury (lead), bismuth, copper, cadmium, arsenic, antimony and tin.
	    Examine by the table for Group II.
	    Bi, As are unlikely in most alloys.
		Boil off the hydrogen sulphide, add about 10 c.c. of bromine water and evaporate to about 5 ml. Add one-sixth of the bulk of solid ammonium chloride and concentrated ammonium hydroxide in drops until it is present slightly in excess.					

		RESIDUE	SOLUTION				
		The residue may contain the hydroxides of iron, aluminium and chromium. Examine for these from the table for Group III.	Add a few drops of concentrated ammonium hydroxide, warm and then saturate with hydrogen sulphide. Then filter.				
			RESIDUE	SOLUTION†			
			The residue may contain the sulphides of zinc, manganese, nickel and cobalt. Examine for these by the table for Group IV.	Boil off the hydrogen sulphide. Add a few drops of dilute ammonium hydroxide and sodium phosphate solution. Gently rub with a glass rod. The solution may contain salts of magnesium. A white precipitate indicates the presence of magnesium.			

* N.B. "Groups" in the analytical sense have no relation to the groups in the periodic classification of the elements.

† In standard tables or Qualitative Analysis, a fifth group containing the metals barium, calcium, and strontium is included at this point, whilst magnesium occurs in Group VI. However, owing to the extreme improbability of finding these metals in alloys, they have been omitted from the present table.

Testing of Materials

GROUP I METALS
(Lead, Silver, Mercury)

A few drops of water should be added to the residue, which is then broken up and heated to boiling point. Filter.

RESIDUE	SOLUTION
About 2 ml. of dilute ammonium hydroxide is added, and it is then warmed. A blackening of the residue indicates the presence of mercurous mercury. Filter, then add dilute nitric acid. A rather curdy white ppt. indicates the presence of silver. However, if there is an absence of mercurous mercury, the residue should completely dissolve in ammonium hydroxide, and be reformed by the addition of nitric acid.	Transfer a little of the solution to a test tube and allow it to cool. A white ppt. will indicate the presence of lead. To confirm this result, add a little potassium chromate solution, whereupon a yellow ppt. should form.

Note. Mercury may occur in either Group I or Group II.

IONS WHICH ARE SEPARATED AS SULPHIDES FROM AN ACID SOLUTION
(Group II)

About 2 ml. of yellow ammonium sulphide is added to the residue. It should be broken up, gently warmed and filtered.

RESIDUE	SOLUTION

The residue may contain the sulphides of mercuric mercury (lead), bismuth, copper, and cadmium.
    Therefore examine by the table for Group IIa.
	The solution may contain traces of arsenic, antimony, and tin.
	    Therefore examine by the table for Group IIb.

GROUP IIa METALS

(Mercury, Bismuth, Copper, Cadmium)

A few drops of water and an equal amount of concentrated nitric acid should be added to the residue, which is then broken up. Gently warm and filter.

RESIDUE	SOLUTION	
To the residue add a few drops of hydro-chloric acid, and a little concentrated nitric acid. The residue should be broken up, heated carefully and the solution diluted with a little water. Add a small crystal of potassium iodide to a small quantity of the solution. The resulting crystal should be scarlet in colour, which indicates the presence of mercuric mercury.	Dilute the solution with an equal amount of water. Add concentrated ammonium hydroxide until the solution is strongly alkaline. Filter.	
	RESIDUE	SOLUTION

	Dissolve in a very small amount of dilute hydrochloric acid, and then pour it into a beaker containing approximately 10–20 ml. of water.
	    A white turbidity indicates the presence of bismuth.
		If the colour of the solution is blue, then copper is present. Transfer a little of the solution to a crucible, add a small amount of dilute acetic acid, followed by a little potassium ferrocyanide solution. At this stage, a red/brown colour should form. If there is no copper present, the remainder of the solution should be treated with hydrogen sulphide. A yellow precipitate will indicate cadmium.
		    However, if copper is present in the solution, mix a little of the solution with some potassium cyanide solution. The blue colour should disappear. The test for cadmium may be made with hydrogen sulphide.

147

Metallurgical Laboratory Techniques

Concentrated hydrochloric acid should be added to the ammonium sulphide extract. Filter. Wash the precipitate and add a little concentrated hydrochloric acid and a few drops of concentrated nitric acid. Dissolve by warming. Particles of sulphur should be removed, and the remaining solution evaporated to half its quantity. It should then be cooled and diluted. Test for arsenic, antimony, and tin as follows.

Arsenic

Pour a little of the solution into a test tube, evaporate until it is almost dry and cool. Add a little ammonium molybdate solution and gently apply heat. A light yellow precipitate, usually left as a yellow stain, indicates the presence of arsenic.

Antimony

Add some sodium sulphide to the solution until the effervescence which occurs stops. Gently warm, and blow air through the solution. If a white precipitate forms immediately, or after dilution, then antimony is indicated.

Tin

Pour a little of the solution into a test tube and add a few milligrams of iron wire or turnings. Heat for about 1 minute, cool, filter off the liquid, boil, allow to cool and then add to approximately one-half of it a few drops of mercuric chloride solution. If a white precipitate immediately forms, then tin is indicated.

To the remaining half add a very small amount of cacotheline solution. If a violet colour results, then tin is indicated.

GROUP III METALS

(Iron, Chromium, Aluminium)

To the residue add a few drops of sodium hydroxide solution and some hydrogen peroxide solution. Break up the residue, warm and filter.

RESIDUE	SOLUTION
Dissolve the residue in a few drops of dilute hydrochloric acid. Pour a little into a test tube and add a little potassium ferrocyanide solution. If a blue colour results, this indicates the presence of iron. However, should ammonium thiocyanate solution be used instead of ferrocyanide, the solution should colour red, which indicates the presence of iron.	If the solution is yellow in colour, then chromium may be present. Divide the solution into portions and test as follows: (1) Pour a few drops into a test tube, evaporate until almost dry then dilute with a few drops of dilute acetic acid. Also add a little lead acetate solution. A yellow ppt. indicates the presence of chromium. (2) Pour a few drops of the solution into a test tube, evaporate until it is dry and pour in a few drops of dilute hydrochloric acid. Add a very small amount of litmus solution and a few drops of dilute ammonium hydroxide. A blue lake formation indicates aluminium. (3) Proceed as above, but take up in a few drops of water. Add a little alizarin red solution and acidify with very dilute acetic acid. The formation of a bright red lake indicates aluminium.

149

GROUP IV METALS
(Cobalt, Manganese, Nickel, Zinc)

To the residue add a few drops of water and an equal amount of dilute hydrochloric acid. Stir well and filter.

RESIDUE	SOLUTION
To the residue add a few drops of concentrated nitric acid and warm until it dissolves. Dilute with water, and divide into portions. Test these as follows: (1) Pour a small amount into a test tube and add a small crystal of each of the following: sodium acetate, sodium nitrite and ammonium chloride. A bright yellow ppt. indicates cobalt. (2) As an alternative to the above, pour a few drops into a test tube, add solid sodium bicarbonate in excess, followed by a few drops of hydrogen peroxide solution. A bright green colour indicates cobalt. (3) Place a small amount onto a filter paper and add a spot of dilute ammonium hydroxide and a drop of dimethylglyoxime solution. If the original spot of the solution is red in colour, then nickel is indicated.	Test the portions as follows: (1) Pour a little of the solution into a test tube and evaporate until it is just dry. Pour in a few drops of concentrated nitric acid and evaporate again to dryness. Add a little dilute nitric acid and a small piece of sodium bismuthate. A violet colour indicates the presenc of manganese. (2) Warm a few drops of the solution and aerate to expel hydrogen sulphide. Cool, and add some $0 \cdot 005$ M copper sulphate solution, a few drops of mercuric chloride solution and some ammonium thiocyanate solution. Agitate gently. A violet ppt. indicates the presence of zinc.

150

Quantitative Analysis

In the metallurgical laboratory, quantitative analysis is usually carried out to determine the carbon content in steels, rather than to determine the constituents of alloys. Therefore this section deals with just two methods of carbon determination.

CARBON IN IRON AND STEEL

The method usually employed to determine the total amount of carbon in a specimen of steel is by direct combustion. This may be carried out as follows. The steel should be in the form of turnings, and is burned in a refractory boat in an abundant stream of oxygen within a furnace. The tube furnace is especially suited for this purpose. Under these conditions, all the carbon present in the steel is oxidized to carbon dioxide, which is absorbed in a weighed tube containing soda lime, soda asbestos or similar absorbent. Before the oxygen enters the furnace, it is passed through a 50 per cent sulphuric acid solution in which chromic acid has been dissolved. It is then passed through soda lime or soda asbestos before it finally enters the furnace, to remove any traces of carbon dioxide. As an additional safeguard, in the event of the carbon being oxidized to carbon monoxide, and because the sulphur is only burnt to sulphur dioxide, the exit end of the furnace is usually plugged with platinized asbestos. This acts as a catalyst, thus completing the process of oxidation. The gases are then passed through one, or even two, gas-washing bottles containing the sulphuric acid/chromic acid solution, before absorption in the soda lime.

PROCEDURE FOR DETERMINATION

A known weight of the metal turnings should be placed in the combustion boat and covered with a layer of red lead. The boat should be preheated to remove any carbonaceous matter. The

151

boat is then placed in the hot zone of the furnace (approximately 1100°C). This may be accomplished by placing it in the open end of the furnace tube and pushing it along the tube with a metal rod or wire (e.g. stainless steel). The furnace tube is then closed by a fitting which has provision, by way of a small-bore pipe, for supplying the oxygen. Having connected the weighed soda lime tube, oxygen is passed in a slow stream for approximately 15 to 20 seconds, after which the supply is turned off. When combustion has been observed to have commenced, the oxygen supply is recommenced, thereby maintaining a slow bubbling through the gas bottles. The flow of gas will increase to a rush, at which stage the supply of oxygen is gradually reduced until a steady flow is indicated. The rush of gas indicates that the combustion is completed. However, the oxygen is continued to be passed for a further 15–20 minutes. At this stage the oxygen supply is turned off and the soda lime tubes are disconnected and re-weighed. The increase in weight, in milligrams, multiplied by a factor of 10 equals the percentage of carbon in the original known weight of the sample of metal turnings.

CARBON DETERMINATION
USING THE STROHLEIN APPARATUS

Using this apparatus, a sample of steel, usually in the form of turnings (approximately 1 gram), is burnt in a stream of oxygen in a tube furnace at a temperature of 1100–1200°C. The carbon thus burnt forms carbon dioxide. The gas mixture then passes through an absorption vessel which has been filled with caustic potash, thus absorbing all the carbon dioxide. The remaining gas returns to a burette, and the carbon content can be read off from a scale fitted to the burette. A correction table is used to obtain a correction according to the barometric pressure and the temperature of the gas measured.

DESCRIPTION

The apparatus illustrated in Fig. 4.26 consists basically of
the following. An absorption vessel (1) is filled with caustic
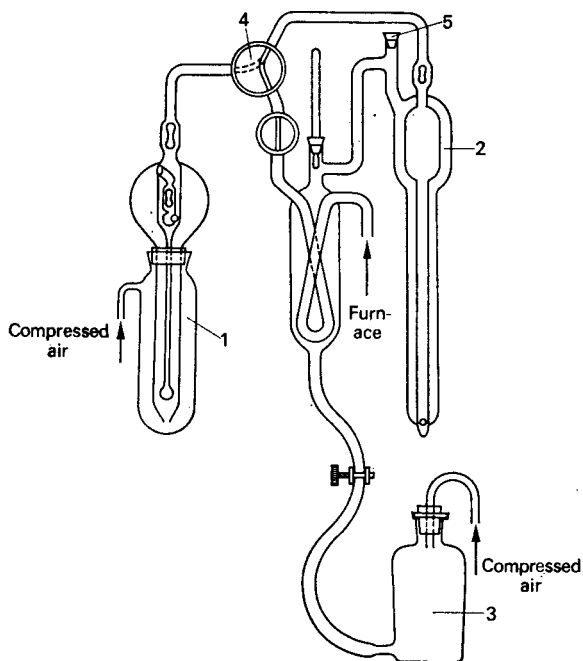potash or, if preferred, 50 per cent solution of potassium

FIG. 4.26. The Strohlein carbon analysis apparatus in diagrammatic
form.

hydroxide. The burette (2) should be filled with a barrier liquid,
usually a 20 per cent solution of sodium chloride to which a few
drops of concentrated sulphuric acid and some methyl orange
are added. The latter enables the level in the burette to be easily
seen. The levelling bottle (3) is filled and connected to an air
supply. By operating suitable controls, the barrier liquid will

rise in a cooling coil, having made sure that the stopcock (4) has been positioned to allow the air to pass freely. As the barrier liquid reaches the burette its flow must be slowed, by using a simple clip, to allow time for air to escape through (5). Carry on filling the burette, and when the liquid reaches the neck of the burette, insert a rubber bung into (5) and the stopcock (4) is then positioned to connect with the absorption vessel. At this stage the clip is removed. The barrier liquid will flow back into the levelling bottle and suck the potassium hydroxide solution into the upper part of the absorption vessel. It should be ensured that the tube at the air control valve is open at this stage. As soon as the liquid has finished flowing, the stopcock (4) is moved to the vertical position and the tube to the right of the absorption vessel is closed until the burette is full. Stopcock (4) is repositioned in its original position and more air is removed from the absorption vessel as the liquid returns to the levelling bottle. It is finally filled by operating the air control valve, closing the tube to the left-hand side, thus allowing the air to force up the potassium hydroxide solution.

The oxygen is usually passed through three wash bottles prior to entering the furnace. Number (1) may be filled with caustic potash solution, (2) with dilute sulphuric acid and (3) with water. Finally the oxygen supply is connected to (1) and the exit of (3) is connected to the furnace. The exit from the furnace is connected to the cooling coil.

OPERATION

The burette and the absorption vessel are filled to capacity, making sure that no air bubbles are present. The stopcock (4) is moved to the closed postion and the lower stopcock is turned to the open position. A refractory boat containing the sample is inserted into the centre of the tube furnace, and the furnace is closed with a stopper or bung. Having allowed sufficient time for the boat to reach furnace temperature, the stopcock to the cooling coil is opened slightly. The level of the liquid in the burette will slowly fall. When combustion has commenced, the stopcock

is opened further to permit more oxygen to be drawn into the furnace to ensure complete combustion. Complete combustion may be observed by noting the reduction in the flow of bubbles emitted from the tube in the third wash bottle. It is necessary to ensure that combustion has taken place before the liquid in the bulb of the burette has been reduced by approximately one-third of the original volume, otherwise the results of the analysis will be too low.

The stopcock (4) is operated, thus connecting the absorption vessel with the burette. The gas mixture is passed twice through the caustic potash solution by operating the air control valve, and is returned to the burette by reversing the operation. As soon as the gas has been returned to the burette, the stopcock (4) is closed, and the levelling bottle is raised to level the liquids. When the liquid has run down the burette, the reading is taken from the scale.

The reading must be corrected for atmospheric pressure and gas temperature, and the result is the percentage of carbon for the original 1 gram sample.

The burette usually has a scale reading of 0–1·5 per cent carbon. This is suitable for 1 gram of the sample. However, if the carbon content of the sample is suspected as being high, then a smaller amount of the sample is used. If the sample is very low in carbon content, then a micro burette may be used.

Chapter 5

Joining of Metals

BASICALLY the joining of metals may be achieved in four different ways. They may be joined by a purely mechanical device, such as a rivet or a nut and bolt, or they may be joined physically using (a) the same metal as a joining medium (welding), (b) a different metal (brazing and soldering), (c) a non-metallic adhesive, usually a type of resin. Although these processes may lie more in the realm of the workshop, there are many instances where the laboratory worker may be required to join metals in connection with the construction of laboratory apparatus, or to repair existing equipment. Therefore let us consider these processes, dealing in the first instance with mechanical methods.

Riveting

The joining of metals by riveting is one of the oldest methods used; the procedure to effect a satisfactory joint has no doubt been the experience of most laboratory workers.

The main types of rivet may be classified into two groups. The first group consists of solid rivets, namely the snap head, countersunk, and dome or mushroom-headed rivet. In the second group, we are concerned with tubular rivets, of which there are but two common types, known by the trade name, the "tucker pop" and the "chobert" rivet. Figure 5.1 illustrates the different types of rivet. Whilst solid rivets may be used for most applications, requiring only simple hand tools to effect the forming of a joint, for example a hammer and a suitable punch or "snap" to form the head of the rivet, the tubular rivet is formed by using a special

tool. These types of rivet are usually used where the under side of the metal to be joined is inaccessible (a closed box-like construction), or too thin in cross-section to produce a satisfactory joint using solid rivets. The rivets are obtainable commercially,
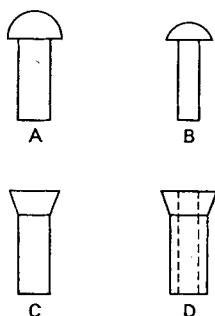

FIG. 5.1. Different types of rivet: A, snap head; B, mushroom head; C, countersunk; D, tubular countersunk.

fixed to a mandrel ready for use. When fitted into their respective applicators and placed into a hole drilled through the metal to be joined, the mandrel will pull through the rivet when the special tool is operated, thus expanding it in the hole. The main difference between these two types of rivet is that in the case of the tucker pop rivet, the mandrel breaks off, leaving part of it in the rivet as a plug. However, in the case of the chobert rivet, the mandrel breaks off at the small head used to retain the rivet on its mandrel. This small head will fall away, therefore the chobert rivet should not be used where these small heads are likely to cause damage, for example to mechanism within the riveted construction. The action of the mandrel breaking off will be determined by the construction and the action of the special tool, thus leaving the rivet in its correct position.

There are two important points to remember during any riveting operation. The rivet must not be formed in shear, as illustrated in Fig. 5.2. If this is the case, then the joint of the parent metals will be weakened. Secondly, the head of the rivet must

157

be flush with the surface of the parent metals; this applies equally to the head that is formed and the head that is already existing on the rivet. Great care must be taken to ensure that there is no gap between the surface of the metal and the under side of the
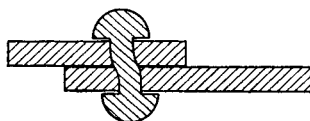

FIG. 5.2. Rivet formed in shear. This is usually caused by misuse of the riveting tools or failure to line up the holes drilled in the parent metals correctly.

head, otherwise these faults will provide an area where corrosion may attack and so progress into the structure of the rivet and parent metals.

Welding

Welding consists primarily of joining similar metals only, using a filler or joining metal of the same composition as the parent metals. It may also be carried out without the use of a filler metal at all, but these instances (spot welding is the most important) are relatively less frequent in the laboratory. The "ideal" welded joint may be defined as follows. When two pieces of metal are joined together so that the original interfacial division between them can no longer be seen, even with the aid of a microscope, they are said to be welded together. However, in practice this is by no means true, since from a metallurgical point of view there is bound to be an interfacial division (which may be minute, but is more frequently clearly visible to the naked eye), owing to small differences in composition between the parent metals and the filler metal, and also because of the effect of the heat on the microstructure of the metals. Thus, the "invisible interface" is an ideal towards which all welding should strive, but which, alas, is never attainable in practice. The term "welded" is a general term usually associated with one of the processes described below.

FUSION WELDING

In fusion welding, the metals that may be joined are usually common steels and aluminium. They may be held together in a vice or by using suitable jigs, clamps or fixtures. The interface of the metals is melted so that the metals blend together. As they cool, the joint solidifies and the parts are now held together by an area of cast metal. However, to provide extra strength at the joint, it is necessary to add metal by using a filler metal which is of a similar composition to the parent metals.

A mixture of oxygen and acetylene gas is usually used as a source of heat, which will provide temperatures up to 3000°C. A temperature of this magnitude will be sufficient to melt all the ordinary steels and alloys, and is necessary when joining sheet metal or a large mass of metal. This is because of the tendency for the heat to be rapidly conducted away from the joint. Also rapid heating tends to localize the heat over a smaller area, thus reducing the tendency of the metals to become distorted, as is often the case when an area of the metal is exposed to prolonged high temperatures.

During the welding operation, the oxy-acetylene torch should be held at approximately 30° to the axis of the parent metals and usually precedes the filler rod, which is hand fed onto the joint by the operator. The operator should slowly progress along the joint, ensuring that the filler metal when molten flows evenly into the joint. It is important to build up the filler metal above the parent metals, to allow for subsequent cleaning by way of machining or hand filing. When using an oxy-acetylene hand torch, it is important to ensure that the correct nozzle or jet is used. The nozzle selected must suit the particular welding operation, taking into account the type of metal being welded, its cross-section, and whether any fixing or clamping devices are likely to conduct heat away from the joint. Nozzles are obtainable commercially as a set. The manufacturers usually stamp a number or letter on them, and by consulting a chart supplied, the correct nozzle may be selected according to the thickness or

gauge of the metals to be joined. Also the chart will indicate the recommended gas pressures to be used with any particular nozzle.

FLAME CHARACTERISTICS

The oxy-acetylene flame consists of burning acetylene in a stream of oxygen. If too much oxygen is used, the torch flame is said to be an **oxidizing** flame. When insufficient oxygen is used
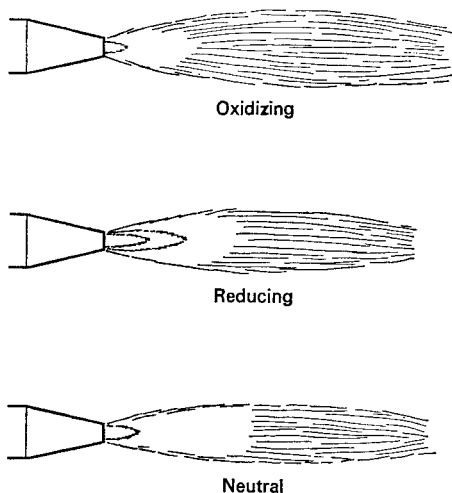

Oxidizing

Reducing

Neutral

FIG. 5.3. Torch flame characteristics.

it is a **reducing** flame. However, the correct mixture of oxygen and acetylene will produce a **neutral** flame. These types of flame are illustrated in Fig. 5.3.

To adjust the hand torch to give a neutral flame, the acetylene is turned on, by way of the control on the torch, and is lit. The flame will be smoky and yellow in colour. The acetylene pressure is increased by manipulating the control on the torch until the smokiness disappears, leaving a bright flame. This indicates that

160

the correct amount of acetylene is being used, in conjunction with the nozzle in use. At this stage, the oxygen should be turned on, again by operating the appropriate control on the torch, and its pressure should be increased until the flame ceases to be luminous. At the tip of the torch nozzle, an inner blue luminous cone will be observed, surrounded by a white plume. This condition indicates an excess of acetylene, producing the reducing flame, which will disperse as more oxygen is supplied until the flame is a clear blue cone without the white plume. This type of flame is the neutral flame, and is the type normally employed for most welding operations. Should the oxygen supply be increased still further, the inner blue cone becomes somewhat smaller and thinner in shape, and the outer envelope becomes streaky. This type of flame is the oxidizing flame. Because the oxidizing flame is difficult to determine, it is convenient to commence the adjustment of the torch with an excess of acetylene, and then increase the oxygen supply until a neutral flame is obtained, sooner than try to obtain the desired neutral flame directly from an oxidizing flame. This applies equally when joining metals by brazing or silver soldering, which will be discussed later in this chapter.

It is essential that the operator wears a face mask or protective goggles when welding is being carried out to protect the eyes from the brilliant glare of the oxy-acetylene flame. Failure to do so may result in severe damage being caused to the eyes.

FLUXES

A flux is essential for all types of welding operation, to protect the parent metals from oxidation, and to assist the molten filler metal to flow at the joint. The joint may be painted with the flux prior to heating, but it is usually common practice to dip the filler rod into the flux prior to melting it into the joint. (The action of fluxes is dealt with more fully under the section on brazing and soldering.) Suitable fluxes and filler metals may be obtained commercially, and it is necessary to ensure that the correct com-

bination of flux and filler metal is used for joining the metals. For example, it would be quite useless to attempt to weld mild steel using aluminium as a filler metal with its corresponding flux, or vice versa.

ELECTRIC ARC WELDING

In this method of welding, an electric arc is struck between two carbon or tungsten electrodes, or between the metal to be welded and one electrode. Similarly, a rod of filler metal may be used as one electrode, whilst the metal being joined forms the other. With this method the filler rod melts when the arc is struck, and is deposited into the joint of the parent metals. At the same time, the heat generated melts the edges of the joint; together they form a continuous weld. The filler rod is coated with a suitable flux which, apart from supplying the weld and thus safeguarding the joint from oxidation, assists in stabilizing the arc. As with other forms of welding, the correct filler metal must be used corresponding to the composition of the parent metals. Electric arc welding may be used to join all the common steels. However, if tungsten electrodes are used to produce the necessary welding conditions, an atmosphere of argon gas is usually maintained around the arc, this process being known as **argon-arc** welding. The inert atmosphere reduces the tendency for the parent metals and the electrodes to oxidize and become contaminated, thus inducing greater electrical resistance between the electrode and the parent metals. The application of argon-arc welding is usually reserved for welding metals which readily form tenacious oxide films on their surfaces, such as stainless steel, aluminium and its alloys, and titanium and its alloys.

The most difficult factor concerning successful arc welding is to make the initial contact between the electrode or filler rod and the parent metals, hence "striking" and maintaining the arc. This can only be successfully accomplished with practice, and once the arc has been "struck", no attempt should be made to rush the welding operation. The operator should progress slowly along the joint, maintaining a suitable distance between the work and the tip of the electrode or filler rod to maintain the arc. There

are two methods of "striking" the arc; in the first instance the tip of the filler rod or electrode may be jabbed onto the joint and then lifted slightly. However, the operator may find that the filler rod or electrode sticks as it is jabbed onto the joint. This is caused by the welding current melting the end of the filler rod or electrode and welding it to the joint. If this is the case, it may be freed by twisting it sharply, or, failing that, it should be removed from its holder and pulled away from the joint with the aid of a pair of pliers.

The second method involves drawing the filler rod or electrode across the joint with a slight circular motion, so that the arc is struck as the filler rod passes through the lowest point of the circle, and as the motion is completed the arc is drawn to the required length, thus effecting a satisfactory welding condition.

A face mask must be worn at all times to protect the operator's face from the glare of the arc.

PRESSURE WELDING

This method of welding originated in the blacksmith's shop, and was generally used for welding wrought iron and steel by hammering the metals on an anvil. The methods employed in modern industries are more refined; however, pressure welding may be carried out in the workshop, if so required, using a hammer and an anvil.

Welding is carried out by heating the metals to be joined to a high temperature, which is just below the point of actual fusion, and hammering them firmly together. During the course of hammering recrystallization occurs at the interfaces, and the crystals, which make up the structure of the metal, grow across the joint, thus producing a fine-grained structure. This method of hammering the weld may also be carried out on the completion of a fusion weld to produce the same result, where it has the additional advantage of removing the solidified flux on the surface of the weld. An advantage of pressure welding is the complete absence of a cast structure and of the defects usually associated with other types of weld.

163

Spot welding

Spot welding is a form of pressure welding, and consists of gripping the metals to be joined between copper electrodes as illustrated in Fig. 5.4. The parent metals are usually slightly overlapped, and actual welding is carried out by operating a suitable
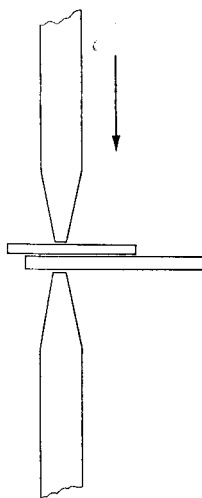

FIG. 5.4. Electrodes gripping the metal for spot welding. Pressure is applied by the upper electrode in the direction of the arrow, thus lightly gripping the metal.

switch (usually a foot-operated switch) which controls the welding mechanism, thus passing an electric current through the electrodes, and hence through the metals to be joined. Intense heating occurs at the interface between the metals (where the electrical resistance is highest), and the result is a very localized pressure weld. If the current passed is high enough, localized fusion may occur at the joint, but this is not necessarily an advantage, since the extra heat involved may cause distortion or buckling of the parent metals.

164

This method of welding may be used in the laboratory for joining thin sheet alloys, forming the hot junction of thermocouples, attaching thermocouples to specimens and to produce temporary joints prior to further working, e.g. brazing or silver soldering. Of course, there are limitations regarding the possible applications of spot welding in the laboratory, these being somewhat dependent upon the type and size of the equipment available. For example, it would be impossible to spot weld heavy sections, as used in the automobile industry, using a spot-welding machine designed to weld the hot junction of a standard thermocouple.

Faults in Welding

Failure to produce a successfully welded joint may be attributed to one of the following defects:

(a) Incorrect fusion; this may be caused by failing to heat both of the parent metals to the correct temperature, which must be sufficient to melt the filler metal by the action of heat transfer from the parent metals.

(b) Using a filler metal of a vastly different composition to the parent metals.

(c) Failure to apply the correct or insufficient flux, thereby allowing the surfaces of the parent metals to oxidize, and restraining the molten filler metal from penetrating the joint.

(d) Cracks, which may occur at the joint, caused by welding metals *in situ* which may have been in strain. Also incomplete penetration of the filler metal may produce a crack directly adjacent to the joint. Therefore should the parent metals be placed under a load in service, or suffer stresses, the crack may propagate through the weld, causing complete failure.

(e) Slag inclusions; during the course of welding the joint, the gas flame may blow particles of slag, which forms on the weld from oxidation caused by absorption of oxygen from the atmosphere, into the adjacent molten filler metal. Hence when solidification of the filler metal occurs, the slag inclusion will be retained within the weld. Should the work being welded be a

165

container, e.g. a container to hold a liquid, then the liquid may soften the inclusion and thus permit the liquid to seep through the weld.

(f) If the joint takes too long to form, the prolonged heating of the metal in the vicinity of the joint may result in a loss of strength due to either annealing, grain growth, or possibly decarborization of the steel. This effect must be remembered, and allowance made for it when work-hardened components are welded.

(g) It must always be remembered that welding will undo the effects of quenching and tempering a steel, leaving a weaker, softer normalized structure. In consequence, either allowance must be made for this weakening if tempered components are to be welded, or the welded assembly must be reheat-treated, an operation which might prove difficult. For these reasons, tempered structures are not suitable for welding, although there is no particular difficulty in actually forming the weld.

The necessary skill required to produce a perfectly satisfactory joint, irrespective of the method used to effect the joint, cannot be obtained from a textbook. This skill is acquired by practical experience, over a long period of time, by welding all types of metals and common and complicated joints. It cannot be too strongly emphasized that in the final analysis, practice and practice alone (leading to experience) is the only way to make a proficient welder.

Soldering

The methods of welding described previously involved heating the parent metals to temperatures equal to, or at any rate approaching, their melting points. In some instances, however, it is undesirable or impractical to raise the temperature of the parent metals to the vicinity of their melting point, or to apply pressure, as required for pressure welding. This is particularly so when joining small sections or components made of non-ferrous materials. High temperatures and pressure may cause these materials and components to distort or even melt. Therefore an alternative method is necessary to raise the temperature of the

parent metals up to, or beyond, the melting point of a filler metal that is of a different composition to the parent metals. Hence a satisfactory joint may be produced by causing the lower melting-point filler metal to bond to the parent metals. This method is called **soldering**; however, soldering covers more than one process, namely soft soldering, silver soldering and brazing. Therefore it is necessary to differentiate between them.

SOFT SOLDERING

Soft soldering employs a filler metal to penetrate capillary gaps between the metals being joined. The filler metal used is composed of metals which have a relatively low melting point, normally an alloy of tin and lead.

A soldering iron, blow-lamp, or a gas flame, e.g. a bunsen burner, is quite adequate to raise the temperature of the parent metals sufficiently to effect a satisfactory joint. One of the main causes of failure in producing a satisfactory joint is the inability of the solder to bond the parent metals because of the presence of dirt or oxide on one or both of the interfaces of the metals. These surfaces should therefore be thoroughly cleaned prior to soldering, using a file or emery paper.

As the parent metals are heated, and before the solder can be applied, a film of oxide will have been produced on the surface of the joint, thus contaminating it and preventing the solder from bonding the metals. To prevent oxidation a flux is used (as is the case for all types of soldering) whose function is to protect the surface of the joint and act as a wetting agent to assist the molten filler metal to flow into the joint. Fluxes are of two types, inorganic and organic. The inorganic variety clean the joint more efficiently (thus making a good join easier), but are dangerous if not washed off carefully; organic fluxes are less searching cleansers, but are relatively harmless if left on the completed joint. For this reason, they are often preferred for joints in electrical apparatus where moisture is undesirable, or where the location of the joint makes satisfactory cleaning difficult.

167

Soft solder is usually obtainable combined with an organic flux, in which the centre or "core" contains the flux. Hence the common term "multicore" solder. Of course, ordinary soft solder may be used, which is obtainable in the form of sticks and usually has a high lead content. When this type of solder is used, the stick may be dipped into a container of liquid flux and then applied to the joint.

There are various types of solder which are obtainable in the form of sticks. The first of these, plumber's solder, contains approximately 67 per cent lead. When the solder cools from the molten state, there is a wide temperature range over which solidification takes place and therefore over which the solder remains in a pliable state. This is an advantage for the plumber, because this allows time in which a joint may be "wiped", whereas this is not possible with a solder which melts and solidifies over a small temperature range. Another solder used in stick form is tinman's solder, which contains 62 per cent tin and possesses the advantage that it solidifies quickly at one temperature (like a pure metal) without becoming pliable. Therefore if the joint is disturbed after the application of the solder, it will not break. This type of tinman's solder is used for most applications in the laboratory. The high tin content is an aid to good bonding between the solder and the components to be joined, but also makes the solder more expensive than plumber's grade. Coarse tinman's solder contains 50 per cent tin and is a cheaper grade than the more tin rich tinman's solder. However, it possesses a definite, albeit small, temperature range of solidification (similar to plumber's solder) over which range care must be taken not to disturb the joint.

Once soldering has been successfully accomplished, it is very important to thoroughly clean the joint under hot running water, and by scrubbing with a wire brush, to remove residues of flux. Flux left on the joint may produce corrosive conditions which will lead to staining, weakening and often joint failure.

CAPILLARY GAPS

Earlier, reference has been made to capillary gaps between the metals being joined, and it is important to understand what is meant by a capillary gap. To illustrate this point, let us consider a piece of clean plain glass sheet held horizontally with a small pool of water on it, as shown in Fig. 5.5. If another piece of
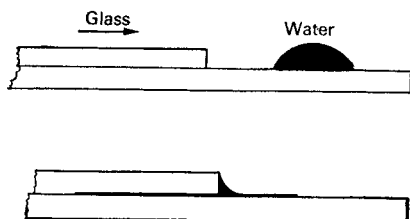

FIG. 5.5. Diagram illustrating capillary attraction. From the diagram it will be seen that water penetrates deeply between the interfaces of the plates by capillary attraction. In the same way a penetration of a thin layer of solder binds together two metal surfaces.

clean plain glass is laid on the first, and if its edge is brought into contact with the pool of water, some of the water will immediately flow into the narrow gap between the glass surfaces for an appreciable distance. The water is propelled by **capillary attraction,** and the gap penetrated is therefore a **capillary gap.** If the gap is made too wide, for example by placing a spacer between the glass sheets, the water will penetrate for only a little way or not at all. Therefore if the gap is purposely kept small, the capillary effect will be obtained even when the glass sheets are vertical and the water will not fall out unless they are separated. Also it is extremely difficult to pull the glass sheets apart because of the bonding effect. Exactly the same effect occurs when a joint is soldered or brazed, except that instead of glass sheets there are the opposing surfaces of the parent metals, and instead of water there is molten solder. Thus consideration of the thickness of the film of water that has been

169

propelled between the plates by capillary attraction will indicate that optimum penetration and maximum strength at a soldered joint will be obtained by ensuring that a minimum thickness of solder flows into the joint.

Silver soldering and brazing

Whilst soft soldering requires the application of heat up to a temperature of approximately 185 °C, silver soldering is usually carried out by heating the parent metals to a temperature in the range of 600–800 °C. As the name implies, silver soldering requires the use of a filler metal containing silver. In some instances this method of joining metals is referred to as hard soldering. Metals whose melting point is above 800 °C, e.g. steels and copper, may be joined by a process known as **brazing** in which the filler metal is ordinary brass rod of extra-high zinc content.

Filler metals

Filler metals may be obtained commercially in the form of rod, strip or foil; also in various lengths, widths and thicknesses. Although the majority of soldered or brazed joints are made by applying the filler metal to the joint by hand, it is often an advantage to use the filler metal as a preplaced insert. Filler metal formed into a circular ring is the more common type employed. Silver soldering or brazing strip may, if so desired, be cut into small segments and placed around the joint. This technique and the use of inserts is very useful if the joint to be soldered or brazed is partially or completely inaccessible. Brass and copper fittings used in the construction of vacuum apparatus are provided with an insert of soft solder. However, it is often practical to form a fillet at the joint, using silver or soft solder, in case the insert does not completely make the joint vacuum tight. The location for inserts is at a shoulder machined on the component or metals to be joined, or in a groove for internal applications.

170

Silver soldering alloys are more expensive than ordinary brazing metals because of the cost of the silver used in their manufacture. The quality of the alloy varies directly with the silver content (which strengthens it and assists bonding with the components); hence using silver solder we have the not uncommon question of compromise between cost and quality. Therefore, it is advisable to use silver solder rather sparingly, provided an adequate joint is made. Table 15 illustrates the various compositions of silver solders and brazing alloys, and gives a selection of their general applications. It is important to ensure that the correct flux is used, which will depend upon the type of metals to be joined. Reference to suitable literature is recommended (see Bibliography) should there be any doubt as to the correct flux to use for a particular brazing or soldering operation.

TABLE 15. SILVER SOLDERING AND BRAZING ALLOYS

Composition (%)					Melting range (°C)	Application
Cu	Zn	Ag	Cd	Sn		
16	4	80			740–795	For use on copper,
20	15	65			645–725	brass and stain-
15	17	50	18		625–635	less steel
50	50				870–880	Ordinary brazing of
50	45			5	750	ferrous materials

FLUXES

As mentioned earlier in this chapter, a flux is essential if good results are to be obtained from metal-joining processes. A flux must be molten, completely fluid and chemically active at approximately 50 °C below the temperature at which the filler metal melts. It must also remain stable up to the maximum temperature of the soldering or brazing operation, and must continue to carry out its functions until after the filler metal has solidified. The flux should be prepared as a paste, and applied

171

to the joint of the metal or component. The flux should not easily be displaced by the pressure of the torch flame, but at the same time it must be fluid enough to be easily displaced from the joint by the molten filler metal. It may be applied by means of a brush or, alternatively, the parent metals or components may be dipped into the paste, if this is practicable. Flux correctly applied before the application of heat will allow the molten filler metal to penetrate the joint gaps to a greater extent than if it is applied after heating of the parent metals has begun. However, if prolonged heating vaporizes the flux, additional flux may be applied as a paste or in powder form by dipping the filler metal into the flux and applying it to the joint.

Fluxes may give rise to corrosive conditions if the irresidues are left in the vicinity of the joint. Quenching the joint in warm water very soon after brazing or soldering may cause the skin of the flux to crack, after which it may be removed by chipping it off.

Joint Design

If a soldered or brazed joint is to be successful, some degree of consideration must be given during the early stages of the design of apparatus to the method of soldering and the filler metal to be used. Whilst a filler metal tends to flow into the joint by capillary attraction, it is important to vent totally enclosed joints, so as to allow air to escape. Also consideration must be given to joint strength. If the clearance allowed at the joint is too small, the filler metal may not penetrate completely into the joint. However, if the clearance is too large, the filler metal will run out of the joint; hence a weak joint will be obtained in both instances. Opinions may differ regarding the importance of clearance in relation to the strength of the joint; in the case of non-ferrous metals clearance generally varies between 0·0015 in. and 0·005 in. Also thermal expansion of the parent metals must be taken into account when considering joint clearance. Table 16 illustrates the change in length of the more common metals for a temperature change of 700 °C.

TABLE 16. CHANGE IN LENGTH PER INCH FOR
A TEMPERATURE CHANGE OF 700°C

Metal	Change in length in inches
Copper	0·011
Iron	0·008
Mild steel	0·008
Stainless steel	0·013
Phosphor bronze	0·013
Brass	0·013

Differential expansion due to heating dissimilar metals may be counteracted by unequal heating, e.g. when joining mild steel to brass the mild steel may be heated to temperature in the first instance, and then the brass. This will ensure that both metals are at a suitable temperature to melt the filler metal, but the temperature will not be so high that the brass melts.

The easiest joints to braze or solder are those where the parent metals are self-locating and need no external jigs to hold them in their correct position in relation to one another. Figure 5.6 illustrates how standard parts may be arranged to enable a satisfactory joint to be made. However, where it is impossible or inconvenient to use these methods, other devices such as jigs may be employed. Jigs are often inconvenient because of the restraint they place on the natural expansion of the parent metals. Also they absorb a large amount of heat, thereby adversely creating a chilling effect on some portion of the parent metals. Therefore Fig. 5.7 illustrates some of the many methods available for holding together parts that are not self-locating.

When the filler metal is hand fed by the operator, provision must be allowed for this during the construction of the assembly to be brazed or soldered. This may be carried out by providing a point at which the filler metal may be aimed, for example by providing an external ledge or corner at the joint. This will also facilitate visual inspection of the joint to determine if it has been brazed or soldered satisfactorily.
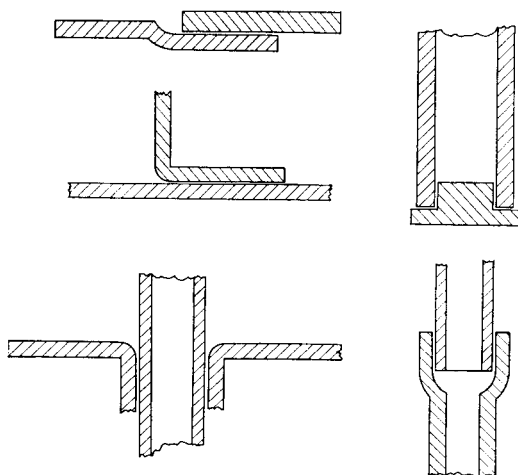
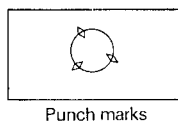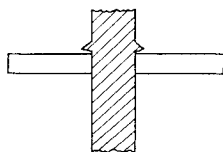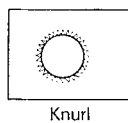FIG. 5.6. Arrangement of standard parts for brazing and silver soldering.

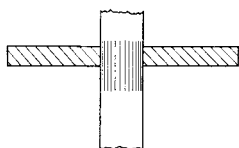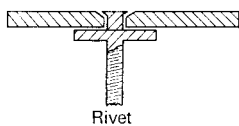

Spot weld

Rivet

Knurl

Punch marks

FIG. 5.7. Methods of holding parts that are not self-locating.

174

Method of Applying Heat

The hand gas torch is the traditional method of applying heat to braze or solder joints, in spite of the fact that there is an increasing tendency, mostly in industry, to mechanize these processes. There are various designs of gas torch available; ideally the torch should be light in construction, simple to operate, and easily used with the minimum of fatigue. The gas leads should also be light in construction, and should not be allowed to impede the movement of the hand torch. Usually it is possible to control the gas mixture by manipulating suitable control taps or levers fitted to the torch, so enabling variations to be made in the size of the flame, and in particular to its characteristics as described in the section on fusion welding. These adjustments will enable the flame to be adjusted to be oxidizing, reducing or neutral according to requirements.

Of the selection of hand torches available, the type used will depend, to a great extent, upon the size of the work being brazed or soldered and the temperature to be attained. For relatively small delicate work, a torch which produces a small flame, or may be adjusted to give such a flame, is used, provided it will heat the parent metals sufficiently.

For brazing, the hand torch flame should have a cone, or an envelope as it is commonly called, of a size that is sufficient to heat the joint in a reasonable length of time. If the flame is too large, then an area will be heated larger than is necessary. Similarly, if the flame is too small, it will probably be difficult to heat the metal to the correct temperature and ensure that both pieces of metal reach this temperature at the same time. Hence it is true to say that experience will determine the choice of torch and the method by which it is used.

The majority of brazing or soldering operations require a torch which is adjusted to give a reducing flame, since this helps to prevent rapid oxidation of the surface of the parent metals. The time taken for a particular type of flame to heat the metals to brazing temperature is dependent upon the heat content of the gas mixture, the velocity of the gas and the type of torch

used. The torch chosen must suit the particular gas mixture, e.g. should the gas mixture be oxy-acetylene, then an oxy-acetylene hand torch must be used. There are various gas mixtures which are suitable for brazing and soldering; the common mixtures in order of increasing heat content are listed in Table 17.

TABLE 17. COMMON GAS MIXTURES

Compressed air and coal gas
Oxygen and coal gas
Oxygen and propane
Oxygen and hydrogen
Oxygen and acetylene

Of the above mixtures, oxygen/acetylene and compressed air/coal gas are probably more widely used in the laboratory or workshop. However, the final choice of gas usually depends upon the type and size of the metals to be joined and the ease with which circumstances allow the flame to be seen by the operator. For example, an oxygen/hydrogen flame is difficult to see; therefore, because of this factor the workpiece or surrounding area may suffer damage. More important, the operator may receive a self-inflicted injury.

Brazing and soldering should, where possible, be carried out at a bench which is of a convenient height. The bench should be faced with a refractory material such as firebricks or soft asbestos. Also, suitable refractory materials may be used to protect the work from currents of air, which can be the cause of uneven heating. Therefore it is often advisable to arrange the refractory material so as to reflect heat from the torch back onto the work. Protective goggles should be worn, especially when an oxy-acetylene torch is used, to protect the operator's eyes from the glare of the flame.

One of the important factors in producing a satisfactory joint is to ensure that the work is heated correctly by using an even heating pattern. Heating may be commenced by holding the torch so that its nozzle is several inches from the work, and so that the flame envelope is spread over a large area of the joint. Even heating is obtained by keeping the torch on the move,

176

and if the metals to be joined are of different section or if they are of different compositions, then the flame should be directed more on the large section of higher-melting-point metal. The ideal heat pattern should be such that the area or point at which the filler metal is to be applied is the last to reach the brazing or soldering temperature. Hence the molten filler metal will flow through the joint to the hottest part.

As heating continues, the flux will commence to bubble and then become a clear thin liquid. This is a good indication that the work is reaching the correct temperature for applying the filler metal. The filler metal should be placed in contact with the joint and fed into the joint, slightly overfilling to allow for shrinkage on solidification. It is very important to remember that the filler metal must be melted by the action of heat transfer from the work, and not from direct contact with the torch flame. When the work has reached the brazing or soldering temperature, the filler metal will melt and immediately wet the surfaces of the joint, flowing into and through the joint. If the filler metal is melted by the torch flame, it will remain on the work like "rock cake" until the work reaches temperature. As a result, the work will require heating to a much higher temperature before the joint can be made, because the lower melting constituents of the filler metal become vaporized, leaving an alloy of a higher melting point at the joint.

Having produced a satisfactory joint, the work may be allowed to cool before removing flux residues. Alternatively the work may be quenched in hot water, unless the metals joined have different coefficients of expansion, or are of different sections. Careful (and possibly controlled) cooling is as important as controlled heating if distortion is to be avoided.

Adhesives

The oldest form of adhesive is fish or bone glue, which has the merits of being cheap and easy to apply, but lacks strength and is susceptible to moisture. However, present-day adhesives,

177

especially those used for joining metals, are based on epoxy resins and possess exceptional strength. These types of adhesive may be used to join most materials, and are useful in the laboratory for joining light components or parts of exceptionally small cross-section. The "setting" of some types of epoxy resin adhesives may be influenced by the application of heat and pressure. However, stress concentrations may develop within the adhesive, due to contraction during the "setting" of the adhesive.

It is important to realize that adhesives are mostly specific to certain types of surface if they are to develop their optimum properties. This is because the strength of the joint depends essentially on the strength of the metal—adhesive bonding. As might be expected, therefore, this bond strength can vary very considerably, depending on the nature of the surface and the nature of adhesive used. For example, it would be useless to use a wood glue to join metals. Therefore suitable literature should be consulted prior to joining the metals, to determine that the adhesive used will give satisfaction.

SURFACE PREPARATION

Although adhesives will adhere to most materials, a greater bonding strength will be obtained if the surfaces of the metals to be joined are adequately prepared. Surfaces are normally prepared by degreasing, but if necessary abrasion followed by degreasing may be employed.

DEGREASING

All traces of oil and grease must be removed from the surfaces of the metals to ensure that adequate bonding between the metals and the adhesive is obtained. This may be carried out by wiping the surfaces with a clean cloth soaked in trichlorethylene. It should be remembered that the solvent is toxic, as a liquid and in the form of vapours. Therefore the laboratory or work

area should be well ventilated if prolonged usage of this solvent is envisaged. Also it is inadvisable to smoke whilst this method of degreasing is being carried out.

Surfaces may also be degreased by washing them in a solution of detergent such as Teepol. The soap solution may be removed by washing the metals or components with clean hot water. It is important to dry the metals thoroughly if this method has been employed by placing them in an oven for a period of time. Otherwise the surfaces may corrode, especially in the case of steel components, where the formation of rust may cause complete failure of the joint and further damage to the component.

ABRASION

Surfaces that have been lightly abraded will produce a better "keying" effect for the adhesive than a highly polished surface. Abrading may be carried out by using a wire brush or a piece of emery paper. However, after abrasion, the surfaces of the metals must be degreased to remove loose particles of metal and abrasive compound.

Painted surfaces should be stripped of paint prior to applying the adhesive, otherwise the strength of the joint will be limited by the comparatively low adhesion of the paint to the metal. Having removed all traces of paint from the surfaces of the joint, it may be abraded and degreased prior to applying the adhesive.

After either of the degreasing operations has been carried out, the surfaces of the metals should not be touched with the fingers, otherwise the surfaces may become contaminated and will have to pass through the degreasing process again.

METHOD OF APPLICATION

A thin coating of adhesive should be applied to the surface of the joint, using a small brush or any suitable applicator. The components or metals are then pressed together, and if necessary

retained in position using string or wire. Any residues of adhesive that may have been extruded from the joint may be carefully removed, using a piece of clean cloth or tissue. This is desirable from the point of view of appearance, but it is particularly important if the components have been designed to fit existing apparatus. Failure to remove the excess adhesive may create difficulty in the final stages of assembly if, as a result of this failure, part of the component is now oversize.

Most adhesives are cured at room temperature, and during the "curing" period, the components or metals must not be disturbed, otherwise misalignment or weakening of the joint is inevitable. Depending upon the type of adhesive, however, it is possible to speed up the curing process without adverse effects upon the strength of the joint by placing the components or metals in an oven for a short period of time. On the other hand, there are certain adhesives which may only be cured at elevated temperatures. Therefore the manufacturer's literature should always be consulted prior to using the adhesive, to determine the correct curing procedure.

Chapter 6

Vacuum Techniques

VACUUM techniques are the basis of much modern scientific research work. In industry they are essential for the manufacture of many items of everyday use, such as electric light bulbs, neon signs, fluorescent lights, radio valves, and cathode-ray tubes. They have also allowed the manufacturer to produce high-accuracy mirrors, micro-circuits and anti-reflection films on lenses by means of vacuum deposition of thin films.

The twentieth century has witnessed many important advances in the field of vacuum technology, beginning with Gaede's rotary oil vacuum pump (1907) and his diffusion pump (1915). On the basis of McLeod's vacuum gauge (1874), Pirani's produced their direct-reading gauges (1906); Buckley's ionization gauge appeared in 1916 and in 1937 Penning's cold-cathode ionization gauge.

Vacuum techniques have their place in metallurgy. For example, holding molten metals and alloys under vacuum serves to de-gas them, and may also prevent the formation of solid impurities (e.g. oxides, nitrides) on cooling. Although this technique is limited to small amounts of alloy for ultra-high purity requirements, or for research work in the laboratory, the result is a truly homogeneous product without flaws at the crystal interfaces. Although problems of oxidation have been overcome by the introduction of controlled atmospheres in furnaces, the same cannot be said of the problems of occlusions in metals except by methods employing vacuum techniques.

The design of suitable laboratory vacuum apparatus depends upon individual requirements. For ease of working, it should be kept to the simplest possible form, such as the layout illustrated

in Fig. 6.1. Assuming that this type of simple standard system is required (and this is the basis of all vacuum systems), each item of equipment in turn will be discussed, commencing with the vacuum pump.
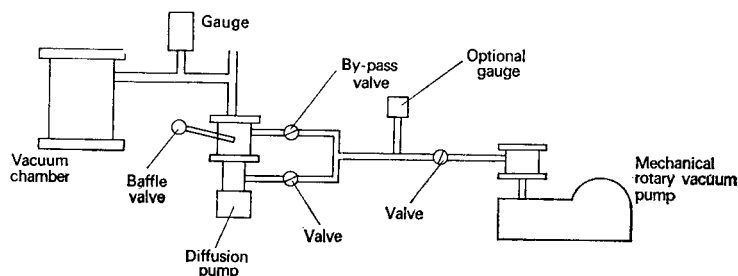

FIG. 6.1. Layout of a simple vacuum system.

Vacuum Pumps

Depending upon the degree of vacuum required, a vacuum system may require a rotary pump or a rotary pump plus a diffusion pump to enable the system to operate satisfactorily. The rotary pump is used initially to remove air from the system, and operates in the pressure range from atmosphere to 10^{-2} mm Hg. At this stage (if it is desirable) the diffusion pump may be brought into operation, enabling the system to be pumped down to a pressure of 10^{-4}–10^{-10} mm Hg. Since diffusion pumps can only operate at low pressures, they require a rotary pump to act as a backing pump. The lower points of these ranges are not achieved abruptly, but reach their ultimate low pressure gradually. The ultimate vacuum that may be obtained depends upon the speed of the pump, usually measured in litres per unit of time, and on the evolution of gas or vapour in the system. Hence when the speed of the pump and the evolution of gas or vapour are equal, the ultimate vacuum is reached. If it is possible to decrease or retard outgassing, the effective speed of the pump will be increased. Therefore it is important to

182

maintain the vacuum chamber and all ancillary fittings incorporated in the system free from volatile greases and similar contaminants. One of the most common and by far the worst contaminants found in vacuum systems is water vapour; performance of the system is greatly improved and contamination of pump oil is avoided by introducing some powerful drying agent such as phosphorus pentoxide or magnesium perchlorate into the system. Provision is usually made for this in a container adjacent to the rotary pump. Water vapour is absorbed by the pentoxide and the time taken to "pump down" the system will be greatly reduced. It also serves to prevent contamination of the oil in the rotary pump. However, phosphorus pentoxide has certain disadvantages. In the first instance it is a very powdery substance, and there is a considerable possibility that it will be sucked into the vacuum pump, thus contaminating the oil and possibly causing corrosion of the pump interior. Also, as the pentoxide takes up moisture, a hard crust or skin is formed and it therefore becomes self-sealing. Hence it must be replaced frequently and usually before it is fully hydrated, which is wasteful. On these grounds, although it is more expensive, magnesium perchlorate is preferable. As an alternative drying agent, use may be made of molecular sieves (highly porous dehydrated crystalline zeolites), which may be obtained from commercial sources and which have the advantage (unlike the other agents mentioned) that when saturated with moisture they may be dried out by heating under vacuum and used again.

Oil-sealed rotary pump

The oil-sealed rotary pump, illustrated in Fig. 6.2, is a mechanical pump which operates in the following manner. A rotor within the body of the pump is turned eccentrically about its axis by belt drive from an electric motor. Gas is drawn into the pump at a low pressure, compressed to atmospheric pressure and expelled through a pressure-operated valve. To ensure that the gas does not leak past the rotor, the entire mechanism is im-

mersed in an oil of low vapour pressure. However, if water vapour is being pumped from the system, the vapour will condense during the stage of compression and mix with the oil, recirculating in the vacuum system and contaminating it. This eventually affects the performance of the pump. It is possible, however, to allow a controlled quantity of air to mix with the volume of gas or vapour under compression, and atmospheric pressure is reached before the partial pressure of the vapour at pump temperature has reached a state of saturation. This is known as air-ballasting, and is extremely useful if a system is liable to be "dirty"; however, the ultimate vacuum attainable is rather less, although not sufficiently so to affect performance as a backing pump. As mentioned earlier, a tray of drying agent placed in a container, which is normally fitted to the pump, may be used to remove water vapour. Experience is usually sufficient to indicate when the oil in the rotary pump is saturated with water vapour, because a pump under normal conditions will operate smoothly; but it will give out a noisy "gurgling" sound when the oil is badly contaminated. Another obvious indication is the inability of the pump to produce the required vacuum in a reasonable length of time.

When using a rotary pump, it is important to remember to open to the atmosphere that part of the system adjacent to the rotary pump prior to turning the pumps off. This will ensure that when the pump is at rest, the oil is not sucked into the system, which at the best is messy, and at worst can be disaster if the oil is sucked back into a furnace; in any case it will involve stripping and thoroughly cleaning the entire system. It is often advantageous to fit to the pump some form of valve or cut-out mechanism which will release the system to the atmosphere in the event of the pump failing for any reason. These devices are available from commercial sources, and provide an added safeguard should the electrical supply fail for any reason.
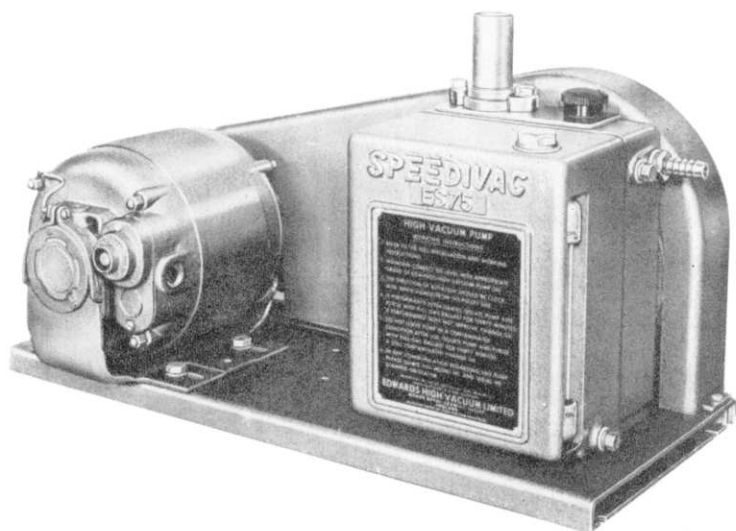
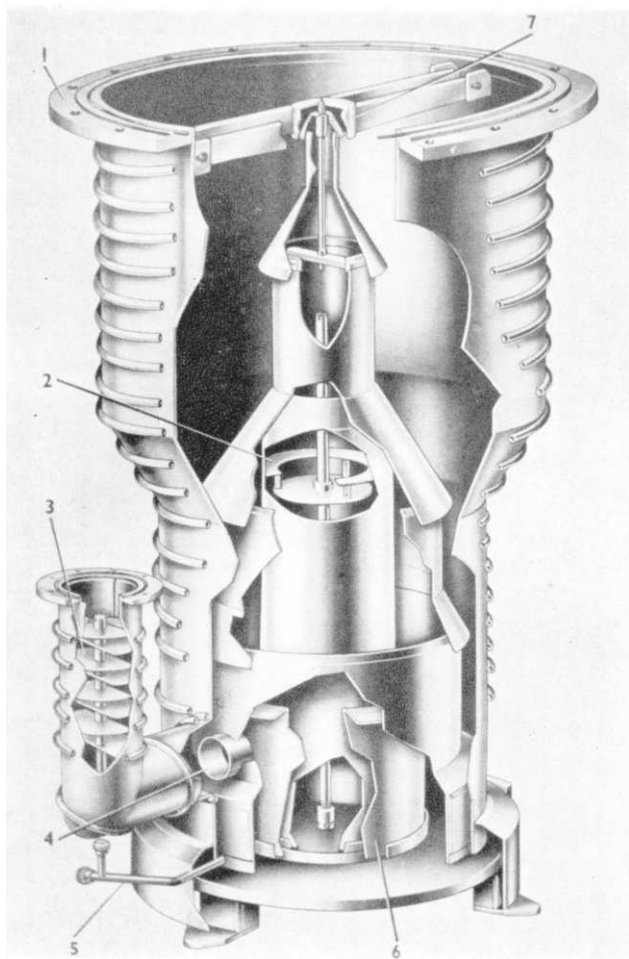FIG. 6.2. Oil-sealed rotary pump. (Courtesy of Edwards High Vacuum Ltd.)

FIG. 6.3. Jet arrangement in an oil diffusion pump. (Courtesy of Edwards High Vacuum Ltd.)

VAPOUR DIFFUSION PUMP

The early type of diffusion pump was usually constructed from glass, and mercury was used as the pumping medium. Although this type of pump may, in some instances, still be used today it has been largely superseded by the oil diffusion pump. The advantage of the oil diffusion pump over the mercury pump is that there is no necessity to use a vapour trap, even though a baffle is often used to prevent oil vapour from entering the vacuum chamber or condensing around the entrance of the diffusion pump. When mercury is used as the pumping medium, a cold trap is essential to prevent mercury vapour from diffusing throughout the system.

The oil diffusion pump uses a high boiling-point silicone oil as the pumping medium. It is contained in a boiler at the bottom of the pump, evaporated and discharged through a jet or series of jets arranged at different heights within the pump (see Fig. 6.3). Molecules of gas from the system which diffuse into the stream of oil move along with it. It condenses on to the walls of the pump, which are water-cooled, and is deflected downwards. In the course of its downward travel the vapour collects molecules of air which are ejected towards the bottom of the pump. From this position they are eventually removed by way of the mechanical pump.

In certain instances, usually where extremely high vacuum is required, a fractionating pump, containing more than one jet, is employed. In this case only the high boiling-point constituents of the oil are admitted to the jet, which is situated at the highest position in the pump, where they will not affect the ultimate vacuum. The constituents of lower boiling point are directed to the lower jet. Hence the constituents of low boiling point and vapour pressure are condensed in close proximity to the high vacuum side of the pump. Ultimate vacuum pressure is therefore determined by the vapour pressure of the condensed oil or fluid nearest to the vacuum side.

185

VAPOUR BOOSTER PUMP

The principles of operation are basically the same as the oil vapour pump. Booster pumps have a high pumping speed and can therefore handle quite large quantities of gas at pressures that are usually too much for a diffusion pump. However, they are used principally in large-scale applications, such as industrial vacuum melting by induction or arc processes, and for vacuum sintering, under which conditions there is considerable gas development. Their use in laboratories is infrequent.

Pumping Lines

Copper and glass are the two commonest materials used for the pipes in a vacuum system; both have advantages and disadvantages which are briefly considered below. Whatever the material used, however, the diameter of the pipe is important. If it is too small difficulty will be experienced in producing a good vacuum. The pipe diameter actually used must be decided upon by the worker, but the following points should be carefully noted. The vacuum chamber is connected to the pump by a pipe, and the ultimate vacuum attainable (in the absence of leaks) is limited by the pipe dimensions, irrespective of the size or speed of the pump. The conductance of the pipe (i.e. the ability to maintain a vacuum in the chamber) is proportional to D^3/L, where D and L are the diameter and length of the pipe respectively. Thus it is desirable to use the largest convenient diameter piping and to arrange the shortest possible distance between the chamber and pump. In practice this means the use of piping of the same diameter as the input orifice on the pump; the length of pipe is usually dictated by experimental layout rather than by the best possible vacuum conditions. When making connections, it is important to ensure that the joints are vacuum tight. Simple elbow-shaped pieces, "T" pieces and reducers are available commercially, usually provided with a soft solder insert which when heated with a blow-lamp

186

or bunsen burner flame will produce an effective seal (see Chapter 5). The use of glass for pumping lines has certain advantages. In the first instance, excellent joints are possible because special tapered cone and socket fixtures, which have precision ground surfaces, are available commercially in a wide range of sizes and shapes. They should be lightly coated with vacuum grease prior to assembly to ensure a perfectly vacuum-tight joint. Similarly, cocks or taps are available, made with the same precision. The glass vacuum system is easily dismantled for cleaning purposes and is more easy to clean than metal. The main disadvantage of glass is the ever-present danger of breakage by accident or faulty design of the apparatus, which may place undue strain on the components. This is especially so during assembly, and great care is essential when lining up the components.

Whatever material is used for the pumping line, it is beneficial to keep the joints to the absolute minimum (even though the system is easier to clean if it can be dismantled into small units). An excessive number of joints makes it more possible for the system to leak. It is also convenient to standardize the size and nature of the joints and valves as far as possible, so that units of the system can be interchanged for leak testing or replacement.

The Vacuum Chamber

Even though early experiments were carried out using chambers made of glass, the majority in use today are made from metal. Common materials are mild steel (although this may corrode), stainless steel or brass. Glass, however, is still used extensively for pumping lines, control valves and traps, since these can be designed for a more or less standard layout. In these circumstances it has certain advantages over metal. (See section dealing with pumping lines.) Vacuum chambers, however, are highly individual, depending on the exact requirements, and, as such, frequently need modifications and additions. Such alterations are easier to carry out in metal than in glass, which

187

needs the services of a highly skilled glassblower; the attainment of vacuum-proof joints in metal is not such an exacting technique.

Both size and shape of the chamber must be governed by the purposes for which it is to be used. However, it is important to keep it to the minimum practicable size and volume, otherwise pumping rates are slower, and the ultimate attainable vacuum may be lower. It is also worth remembering that curved surfaces (convex on the atmosphere side) are more stable mechanically than flat surfaces, and therefore less prone to leaking from distortion under high vacua. The larger the chamber, the more important this is, and the optimum shape of the chamber is thus cylindrical. If the ends of the chamber are to be dismountable (this is usually desirable for the purpose of cleaning), then flanges must be welded or, in the case of brass tube, silver soldered to the open ends of the tube. Great care must be taken in preparing the metals and making the joint. This is very important since residual stresses may be set up around the joint which, together with the pressures set up under vacuum conditions, may be sufficient to cause cracking in the thin-walled apparatus. The flanges should have a groove machined in them to accommodate a sealing ring. (See section on seals.) The depth of the groove is usually half the thickness of the sealing ring. During machining care must be taken to ensure that the lathe tool does not produce scores or "chatter" marks in the face of the groove; an imperfect surface will not allow the seal to "sit down" properly and therefore it will not function correctly.

Metal covers or end plates are used to close the chamber, and these may also be machined to correspond with the groove in the flanges. Under normal conditions, however, the second groove may not be necessary since the cover is retained in position by the vacuum in the chamber. Provision should be made, however, for bolts to pass through the cover and flanges as a locating fixture for sections of the system. This also provides insurance against slightly untrue surfaces, and prevents the plate dropping off when the vacuum is released.

The thickness of the walls of the chamber should be sufficient to ensure that no distortion occurs whilst the chamber is "under vacuum". For example, if a 1-gallon petrol can is evacuated it readily collapses, since the walls are unable to withstand the pressure (approximately 14 lb./in.^{2}) of the atmosphere. Therefore consideration must be given, when designing laboratory chambers, to their wall thickness, which is usually $\frac{1}{4}$–$\frac{1}{2}$ in.

Vacuum Seals

The most common method for making a seal between demountable joints such as valves and pipework, or between the vacuum chamber and end plates, is to use an "O" ring. The rings are made of plasticized rubber and are obtainable commercially; their practical use is illustrated in Fig. 6.4, which shows the ring being used to seal the end plates of a vacuum chamber.
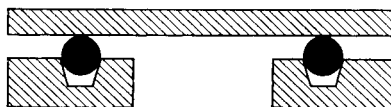

Fig. 6.4. Use of an "O" ring for sealing the end plates of a vacuum chamber.

Figure 6.5 illustrates the use of the ring when connecting a valve or pressure gauge to the pipework of the system. When the system is under vacuum, the reduction in pressure within the system places the ring in compression, thus producing an effective seal. The ring may be lightly greased, using a low vapour pressure grease, to assist sealing. However, there are disadvantages in the use of the "O" ring. In the first place, "O" rings are normally made from neoprene rubber, which is only efficient up to a maximum temperature of approximately 120°C. Above this temperature the rubber decomposes, and the joint will leak to the atmosphere. Therefore they should not be used in the vicinity of furnaces, as employed in high-temperature work.

For moderately high temperatures, large flanges with water cooling are useful, as illustrated in Fig. 6.6, and silicone rings
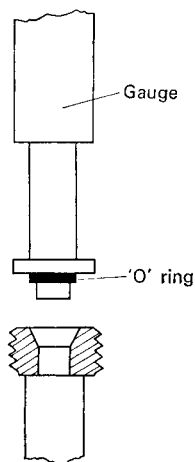

FIG. 6.5. Use of an "O" ring for sealing a vacuum gauge to the pipework.
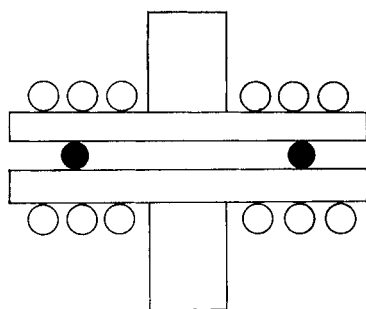

FIG. 6.6. Water-cooled flanges for use in moderately high temperatures.

which are stable at high temperatures (100°C) may be employed. Also, should "O" rings be used in ultra-high vacuum systems, the rather volatile constituents of the neoprene ring may cause

190

contamination of the system. Alternatively, special hollow metal seals are often employed; copper and aluminium components of this nature are widely used as an effective semi-permanent seal, being able to withstand wide extremes of temperature.

Another type of seal which may be obtained commercially and is useful for certain applications is the Wilson seal, the use of which is illustrated in Fig. 6.7. Sealing is effected by the difference in pressure between the inside and outside of the vacuum
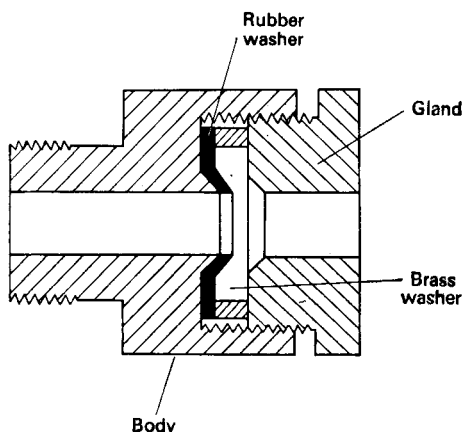

FIG. 6.7. General principle of a Wilson seal.

chamber. Wilson seals may be used to provide a means for introducing rotational or sliding movements via a metal rod into the vacuum chamber, to operate simple mechanisms inside the chamber. The rod does not have to have a perfect surface, but deep scratches should be removed by lightly abrading the rod with emery paper. This will avoid possible damage to the seal, and allow the neoprene washer to form an effective joint around the rod. The rod should be greased rather sparingly with vacuum grease, to allow the rod to slide or rotate through the seal. The body of the seal may be silver soldered into the chamber at a convenient working position.

191

It may be necessary to make fittings to suit a particular vacuum system, or according to experimental needs. If "O" rings are to be used as a sealing device, it is important to ensure that the components are machined accurately according to the dimensions of the "O" ring. Otherwise, when the vacuum system is in operation, the ring will be sucked out of position, thus producing a leak. Similarly the recess should not be too small. In this instance, the ring will tend to be forced into an irregular shape, and again will produce a defective joint. Suitable dimensions for grooves, related to the gauge of the "O" ring, are usually recommended by the firms manufacturing the rings.

Vacuum Valves

A vacuum valve is used to isolate a section from the remainder of the system, for example to allow specimens to be inserted or withdrawn from the chamber. When the chamber is open to the atmosphere, the valve permits the remainder of the system to be kept under vacuum. Contamination of the vacuum line is thus prevented and this saves time in "pumping down" the complete system when it is operated again. In some instances, especially if the vacuum system is large, the time required to pump it down to the required pressure may take a number of days. Valves are also invaluable for isolating sections for leak testing.

Types of valve

The more common types of valve employed are the baffle, diaphragm and needle valves; the latter is usually used to admit small quantities of gas into the system. If the use of the needle valve is envisaged, it is important to mount it into the system so that the atmospheric pressure pushes the needle down onto its seat, and not vice versa.

THE BAFFLE VALVE

The baffle valve is usually incorporated into a diffusion pump. Hence its application is twofold. Apart from isolating the diffusion pump from the chamber, it also condenses, by way of water-cooling, any vapour which may tend to pass into the system. Figure 6.8 illustrates the working of the valve.
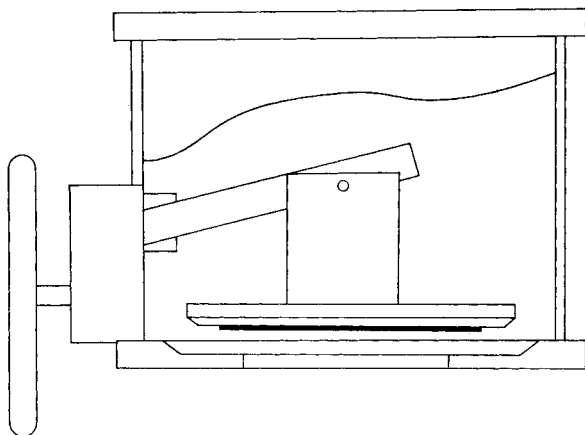

FIG. 6.8. A simple baffle valve.

THE DIAPHRAGM VALVE

The last of the common types of valve is the diaphragm valve. In this instance a diaphragm or disc is forced onto a seat machined in the body of the valve when it is in the closed position. The sizes of the valves vary from approximately $\frac{1}{4}$ in. diameter upwards, though 2 in. diameter is as large as most laboratory apparatus requires.

In the average system a combination of all three types of valve is often used. The baffle valve serves to isolate a diffusion pump from the chamber, the needle valve to allow the intro-

duction of an inert gas into the system and the diaphragm valve to isolate parts of the system as required, as in the by-pass arrangement between a diffusion pump and its backing pump.

Traps

Traps are an important item in a vacuum system to assist in preventing unwanted vapour from entering the vacuum chamber. When vapour diffusion pumps are used, a metal baffle may be used to prevent oil vapour from entering the vacuum chamber.
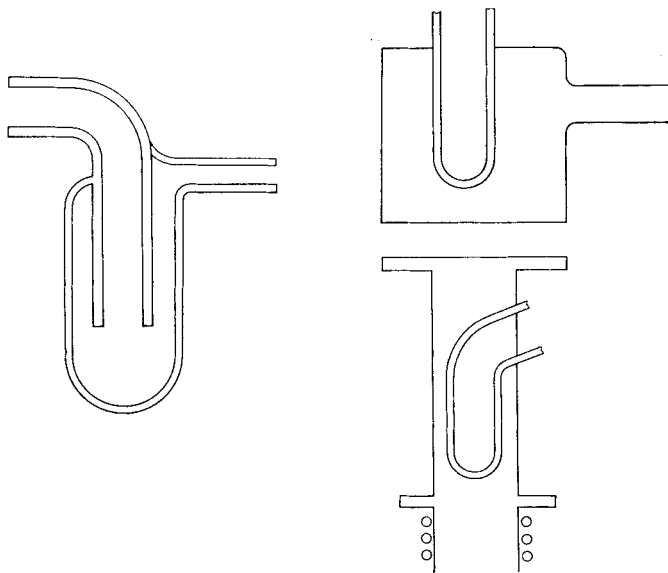

FIG. 6.9. Examples of common types of cold trap.

Diffusion pump baffles are often water-cooled on the outside to ensure the condensation of any oil vapour escaping from the main body of the pump. A vacuum valve placed between the diffusion pump and the chamber will act as a baffle, but it may

194

not be so efficient. In systems used in conjunction with vacuum melting furnaces, traps may be employed to prevent dust particles from refractory materials entering the pumping system. However, if the dust concentration is relatively low, then the baffle in the diffusion pump acts as a trap and prevents dust from reaching the mechanical pump. Cold traps may also be used to remove some forms of condensable gases, such as traces of organic vapours from cleaning agents, and water vapour. In this instance the trap may consist of a "cold finger", which is basically a tube inserted into the vacuum chamber, into which liquid air or liquid nitrogen is poured, thus freezing out the water vapour. It is also possible to use a diffusion pump which is fitted with a suitable cold finger, and to insert traps at strategic points in the vacuum line. Examples are shown in Fig. 6.9.

Gauges

Pressure gauges are used to indicate the degree of vacuum within the chamber and system. As it is the vacuum in the chamber that we are ultimately concerned with, gauges should be located as close as possible to the chamber. Further gauges may, if so desired, be situated at various positions in the system, for example to measure the backing pressure between a diffusion pump and a mechanical pump. For reasons of economy, it is best to arrange a series of dismountable joints, isolated by valves from the main system, to which the gauge can be attached in turn, according to the section in which it is desired to read the pressure. In this way, a large system can be covered quite adequately by about four gauge heads, which can be moved about for purposes of leak detection.

MᴄLᴇᴏᴅ ɢᴀᴜɢᴇ

The McLeod gauge was developed from the simple U-tube manometer and operates down to 10^{-6} mm Hg (see Fig. 6.10). The gauge relies on trapping a known volume of gas from the

vacuum system, compressing the gas to a known pressure and measuring the volume thus occupied. The introduction of air at the surface of a reservoir of mercury drives the gas up a closed capillary tube and also up an adjacent open capillary tube which is connected with the vacuum system. By comparing
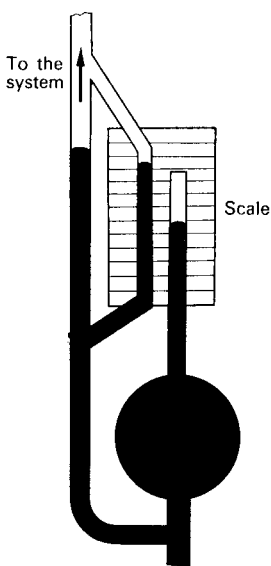

FIG. 6.10. McLeod gauge.

the levels of the mercury against a scale, the pressure is determined. The advantage of the McLeod gauge is that it is the only vacuum gauge which actually measures the pressure of the vacuum, rather than the effect of the pressure upon some secondary variable. It is therefore the basic reference source from which all the more sophisticated electrical vacuum gauges are calibrated. One of the disadvantages of this type of gauge is that, because of its mechanism (see Fig. 6.10), it will not give a continuous reading of pressure.

196

PIRANI GAUGE

The Pirani gauge and similar thermal type gauges make use of the fact that the thermal conductivity of a gas varies with pressure. The gauge is operated by the change in temperature of an electrically heated filament, arising from variations in pressure in the surrounding gas. The filament, made from tungsten, has a large temperature coefficient of resistance, and therefore any change in temperature results in a considerable change in resistance. The tungsten filament is connected to the vacuum system and arranged within the gauge as one arm of a Wheatstone bridge, the other arm being exposed to the atmosphere or to gas at a standard pressure. The change in resistance thus produces a current which is out of balance, the shift in balance being proportional to the difference in gas pressure between the two arms of the bridge. This current is measured on a meter suitably calibrated to indicate pressure.

The useful working range of the Pirani gauge is from atmosphere to 10^{-3} mm Hg, but it will not remain accurate when used in a system in the presence of vapours, for example a system involving a vacuum melting furnace. This is because, apart from residual air, moisture and oil vapour present in the system, metal vapours are also encountered. Hence the wire within the gauge whose resistance is being measured will become contaminated, thus changing its resistance and producing an inaccurate pressure reading. For a similar reason, the gauge should not be used when the vacuum system is open to the atmosphere, and should be switched off before the vacuum is released.

Pirani gauges are very sensitive to hydrogen (due to the greater thermal conductivity of hydrogen) and, if used in conjunction with a hydrogen probe, are extremely useful as a leak detector (see section dealing with leak detection). It only requires a very small quantity of hydrogen to enter the vacuum system through a leak for the gauge to show an increase in pressure.

IONIZATION GAUGE

The ionization gauge measures the ion source which is produced in the gas, depending upon its pressure. The most frequently encountered gauge of this type is the triode or hot cathode gauge. The gauge consists of a cathode, an anode and a grid, the cathode being a filament which is heated by means of an alternating current. It is controlled to supply a constant stream of electrons, which are deflected between the filament and the anode to a positive grid. As the electrons move to the grid the atoms of gas in the ion tube of the gauge are ionized as they collide with the electrons. Therefore the ions produced between the grid and the anode travel to the anode, thus producing an electric current, which is measured on a suitable meter, e.g. a microammeter. From this it can be seen that the greater the amount of gas, the greater is the degree of ionization and therefore the greater the current.

This type of gauge is very sensitive and may be used to measure vacua down to 10^{-8} mm Hg. However, the length of the "life" of the gauge depends largely upon the "life" of the filament; under ideal conditions in which all forms of contamination are avoided, for example moisture, the filament will not last more than a few thousand hours of continuous operation. Also the filament will burn out if the gauge is used above 10^{-2} mm Hg.

Leak Detection

It is probably true to say that after completing a vacuum system, the laboratory worker will spend a great deal of time detecting and rectifying leaks. However, if a systematic approach is made to the problem, the task of leak hunting should not prove particularly onerous. There are several techniques available, some of them requiring the use of quite elaborate equipment; in this instance we are only concerned with the simple, straightforward techniques which may be carried out without dismantling the apparatus. Further information on the more elaborate techniques can be obtained from the Bibliography.

If more than one gauge is employed in the system, it is possible to determine whether the leak is in the pumping line of the vacuum chamber by simply closing down, by way of suitable valves, first the system and then the chamber. A leak will be detected by observing whether the gauge indicates a low-pressure reading. However, extra gauges may not be available, or may not have been combined in the apparatus. In this instance a technique may be used which, although it may seem crude, is perfectly simple and reliable. The gas pressure in the system should be reduced, or returned to atmospheric pressure. Air should be admitted so as to pressurize the system to approximately 2–5 lb/in². A soap solution may be applied around the joints, especially movable or demountable joints, and a careful watch made for the appearance of bubbles. Usually this method will reveal the presence of a leak very quickly. However, if the leak is large, it should be rectified before continuing with the search. It is important to remember that where large leaks prevail, the soap bubbles may be blown away. Therefore it is necessary to observe the soap solution in the crevice of the joint closely, to determine whether the film of soap solution has remained unbroken. If the sound level in the vicinity of the apparatus is low, it is sometimes possible to hear the escape of air from a large leak.

Another method of leak detection, assuming that a Pirani or thermocouple gauge is employed, is to apply a rubber tube to each suspect region in turn and allow hydrogen gas to stream slowly over the area. The gauge should be checked to detect any fluctuation in pressure reading. If a leak is apparent, there will be a rapid increase in pressure due to the high thermal conductivity of the hydrogen gas, as compared to air. It is sometimes advisable to enlist the help of a second person to observe the gauge, whilst the other applies the rubber tube to the joints. This technique is very effective, and only minute leaks escape detection. It is necessary to pass the rubber tube **slowly** over the joints, to allow time for the hydrogen gas to diffuse throughout the system. Also this method of detection may be carried out whilst the system is fully operative.

199

Chapter 7

Powder Metallurgy

POWDER metallurgy is a relatively young and very important branch of general metallurgy, and for that matter of engineering as a whole. From its modest beginnings in 1829 it has expanded rapidly and now plays a vital part in the manufacture of small engineering components, including many which are not available by any other metallurgical techniques. For example, the production of refractory metals, such as tungsten, tantalum and molybdenum, has always posed a problem because of the high temperatures required for melting operations; by using powder metallurgical techniques, melting is avoided completely, and with it the problem of finding refractories capable of containing molten metals at extreme temperatures. Similarly in the manufacture of cutting tools, powder metallurgy affords a means of making up and forming excessively hard composite alloys based on tungsten and titanium carbides and cobalt metal which could not be achieved by any other methods. These alloys are used in the production of machine tools, thus increasing the speed at which metal may be removed in machining operations and giving a longer tool life. Materials may be produced containing mixtures of non-metallic and metallic phases, and also porous materials in which the porosity can be controlled to fine limits. Such alloys are unrealizable by the applications of conventional melting and casting techniques, as is the degree of purity obtained.

Another application of powder metallurgy is in the production of small components on an industrial scale in a finished state, requiring no further machining. This is especially beneficial for mass-production purposes, where the use of metal powders

200

enables the output of certain articles to be increased and thus involves lower labour costs. Also the accumulation of large quantities of scrap metal, which can seriously raise the cost of an industrial process, is overcome.

To offset these considerable advantages, powder metallurgy has also some serious drawbacks which still limit its applications to a relatively narrow field. Relatively speaking, metal powders are usually more expensive than other forms of metal; they are difficult to keep pure without the extensive use of controlled atmospheres for handling, and, especially when the particle size is very small, they may be dangerous. This applies particularly to reactive metals such as magnesium and aluminium, but even iron becomes pyrophoric when sufficiently finely divided. The quality of the product is dependent upon maintaining a very high degree of purity in the powders. The products of powder metallurgy are limited to small and fairly simple shapes, because of the difficulty of obtaining a uniform density in the pressing. Furthermore, sintering leads to dimensional changes in the compact which must be allowed for, and which can be quite unpredictable if uniform density has not been achieved. From the point of view of production, the initial cost may be quite high, because of the expense involved in obtaining suitable dies, punches and presses required for compacting the powders into their final shapes. This may, however, be justified by the demand of present-day markets for articles which may be produced cheaply on a large scale by powder technology. Despite all the above difficulties, many of which are still to be solved satisfactorily, the list of products is a formidable one, and ranges from everyday commodities such as filaments for electric lamps and radio valves, gears for light machinery, bearings, filters, automobile components, magnet alloys and components used in domestic appliances to the complex range of components used in the aircraft industry,— for example components for ancillary parts for engines and friction discs used in brake systems.

Preparation of Metal Powders

The description in detail of all the various methods employed in the usage of metal powders and their preparation is beyond the scope of this book. Powders cannot easily be prepared in the general laboratory but are obtainable from many commercial sources. The main methods of production are worthy of a brief discussion both from the point of view of general interest, and because the physical properties of the powders are to some extent determined by their method of preparation.

COMMINUTION

Comminution is a mechanical process used to reduce metals to particles by grinding (ball milling) or machining. In some instances crushing techniques may also be employed. Ball milling is mostly used for reducing iron and steel alloys, whilst ductile metals such as aluminium, lead and in some instances copper, may be comminuted by hammering in a hammer mill. The process of hammering produces particles which do not sinter as reliably as particles produced by other methods. However, they are often used as pigments in paints, since the flake form of the particles allows them to cover a maximum area for a given weight of powder.

BALL MILLING

Ball milling may be carried out in the laboratory, and consists of rolling a drum or cylindrical container, made of steel or rubber-coated steel, on motorized rollers as illustrated in Fig. 7.1. The drum is filled with a quantity of balls (approximately one-third of the capacity of the drum) and the metal to be ground. The quantity of metal is usually 25 per cent of the volume of the balls. After closing the drum, it is rolled on the rollers or in some instances in a cradle for several hours. The

202

design of ball mills may make provision for rolling several drums at a time. The speed at which the drum revolves is important, as it affects the milling action of the balls. If the drum is rotated at a high speed, then centrifugal force causes the balls
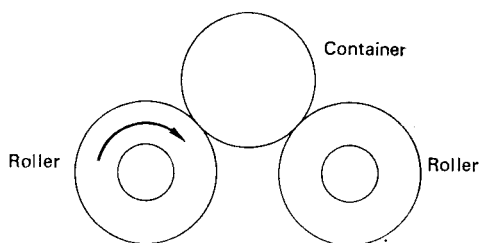

FIG. 7.1. Simple ball mill.

to cling to the walls of the drum and the milling is not carried out correctly because the balls do not come into contact with the metal to be ground. This also applies if the speed is too slow. In this instance the balls move over the lower part of the drum, and the action is insufficient to grind the metal. When the drum is rotated at the correct speed, the balls are lifted up and allowed to fall onto the metal and this continuous movement produces the desired grinding action. The speed of rotation is usually determined experimentally, but in the case of commercially manufactured mills data may be provided indicating milling speeds for various metals.

It is important to ensure that the balls used are suitable for milling a particular metal, otherwise the powder produced will become contaminated with particles of metal ground from the balls. Similarly, contamination can occur from particles that are worn away from the interior surface of the walls of the drum. Therefore if, for example, a steel-lined drum is used to grind hard steel alloys, the drum must be lined with a hardened-steel liner, and furthermore, hardened-steel balls must also be used. In general ball mills are better employed on brittle materials, because ductile materials tend to become flattened by the action of the balls rather than be broken into small particles.

During the course of milling, the particles may suffer from oxidation. This may be reduced by carrying out the milling operation in an inert atmosphere. A process known as wet grinding is sometimes used, by placing a quantity of some protective non-corrosive liquid, for example alcohol, in the drum. Although this method has been used to prevent oxidation of pyrophoric powders such as magnesium, it is also used when milling other metals as it is possible to produce a different size of particle than that produced by dry milling.

Powders produced by mechanical methods will have undergone some degree of work-hardening. Therefore it is important to anneal the metal powder in the final stages of preparation.

COMMINUTION BY MACHINING

Comminution by machining is expensive compared with other methods, and is not used as the final stage of comminution. One interesting powder that is produced partially by this method is probably familiar to us all. The metal powder referred to is the dental alloy. In this case cost is not considered to be excessive when taking into account the cost of the alloy, which usually contains up to 70 per cent silver among its constituents.

The constituents of the alloy are cast to form an ingot, approximately 12 in. in length and 2 in. in diameter. The ingot is reduced to particles by placing it in a lathe and positioning a lathe tool against its face. Whilst the lathe is operating, the tool is automatically "fed" into the ingot. The particles of alloy are allowed to fall into a tray placed beneath the lathe chuck. They are separated into fine and coarse grades; the fine grade undergoes further comminution in a ball mill. Finally it is annealed and deoxidized. The coarse grade, however, only requires sieving, grading and annealing. The annealing operation is very important, and the facts concerning annealing are closely guarded secrets of the manufacturers. This is because the annealing operation determines the final quality of the alloy.

Magnesium powder for pyrotechnic purposes has in the past been produced by machining. Usually it is carried out in an inert atmosphere because of the explosive natue of the powder when exposed to oxygen. The atmosphere under which the powder is produced may contain sufficient oxygen to afford a protective coating of oxide, but care must be taken to ensure that the oxygen is not of such a quantity to cause an explosion.

ATOMIZATION

Atomization is the technique used to produce non-ferrous metals such as aluminium, lead, tin, zinc and copper in powder form. The metal, in a liquid state, is poured from a crucible by way of a nozzle against a jet or stream of air, water, or inert gas, which rapidly breaks up the stream of metal into droplets. However, it is the design and positioning of the nozzle combined with the velocity at which the air, water, or gas strikes the stream of metal which finally determines the shape of the particles of powder produced. The chilling effect of the atomizing agent also prevents any risk of the powder self-sintering immediately after formation and before it would have had time to cool. Powders produced by atomization usually conform to the following particle shapes: tear-drop, irregular, and almost perfectly round.

OXIDE REDUCTION

Metal oxides are frequently brittle and susceptible to powdering by comminution and ball milling. An oxide powder of the requisite size having thus been obtained, it may then be reduced chemically to give the pure metal powder. Reduction is carried out by passing hydrogen or carbon monoxide over the metallic oxide at a suitably elevated temperature. This method enables powders to be produced with a wide range of characteristics regarding the size, shape and porosity. Also because comparatively low temperatures are employed, excessive sintering of the

products is avoided. Most powders, especially in large quantities, are produced by this method, for example tungsten, molybdenum, cobalt and iron. The process results usually in irregularly shaped particles which are sometimes porous.

ELECTROLYSIS

Metals may be deposited electrolytically in powder form by employing a higher current density than is normally encountered in electroplating. However, some alteration must be made to the ordinary electrolyte used in electroplating, and to the cell arrangement. The electrolyte used is of a low metal ion concentration and high acid concentration. Also a high current density is maintained at the cathode. Metal powders produced by electrolysis are iron, copper, zinc, cadmium, nickel, tin and lead. The deposit may be removed from the cathode at regular intervals, otherwise the build-up of metal deposited onto the cathode will increase its effective area, thus decreasing the current density. (Therefore effective working of the electrolytic bath will become impossible.) The deposit is usually removed by scraping it away from the cathode.

The actual powder produced by electrolysis requires no further treatment besides washing and screening, by way of sieves, to final size and condition. The washing operation must be carried out thoroughly. Should a very small amount of electrolyte remain within the collected metal powder, rapid oxidation will occur. The shape of particle produced is dendritic (irregular, tree or fern shape). Therefore the particles tend to compact well, because of the tendency for the particles to readily interlock. However, if iron or nickel is reduced by electrolysis, a hard, brittle substance will be deposited, which requires some degree of mechanical breakdown, e.g. ball milling. Therefore, because of the mechanical working of the particles, they will require deoxidizing and annealing before the powder is in a suitable condition for compacting. Powders produced by the process of electrolysis are usually in a high state of purity.

Sieving Powders

In any powder metallurgical operation it is important to know the size of the individual powder particles; further, it is important to know what range of sizes occur in any batch of powder. Particle size and size distribution may play a large part in determining the physical properties of the compact. For example, in order to
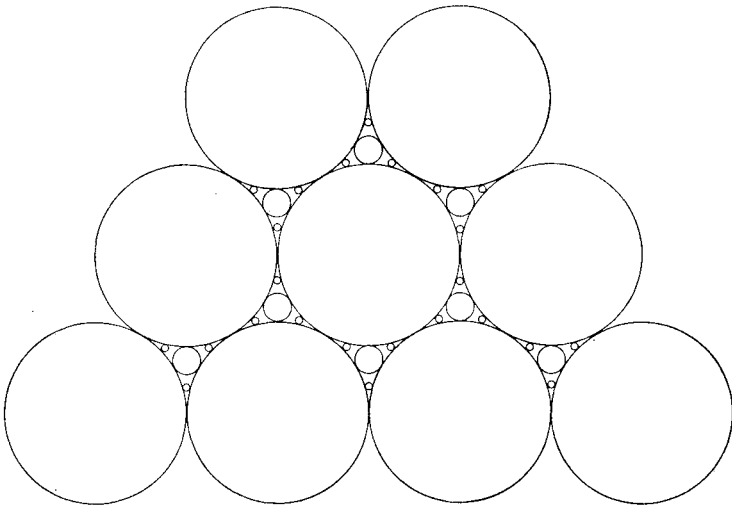

Fig. 7.2. Schematic showing particle size and distribution.

obtain maximum density in compacts, it is necessary to have a distribution of particle size according to Fig. 7.2; in practice this distribution can obviously only be approximated to, but it illustrates the need for knowledge as to the size of the powder.

Grading of powders is carried out by passing them through sieves of increasingly fine mesh. Table 18 lists the British Standard mesh numbers and their equivalent size in inches. Obviously, even this method of grading is approximate since it grades powders according to their maximum dimensions only; thus particles of very different mesh may be graded to the same sieve

207

TABLE 18. BRITISH STANDARD MESH
NUMBERS AND SIZES IN INCHES

Sieve number	Size of mesh
100	0·0060
120	0·0049
150	0·0041
170	0·0035
200	0·0030
240	0·0026

number. The weight of powder initially in the sieve is important.
When sieving irregular-shaped particles, the possibility of a
particle being in the correct position to fall through the sieve is
increased as the total number of particles in the sieve decreases.
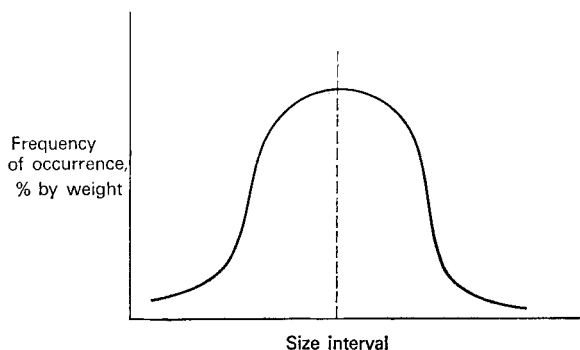
Frequency
of occurrence,
% by weight

Size interval

FIG. 7.3. Illustrating normal distribution.

Furthermore, in any sieving operation it takes **time** for all the
fine particles to work their way through the sieve, and therefore
sieving should be carried out for a standard time. The result will
not give 100 per cent separation of the coarse from the fine par-
ticles, but it should give a standard distribution (see Fig. 7.3).

Sieving is usually carried out on a machine which automati-

cally shakes the sieves. The method employed is called "stack sieving", in which the coarsest sieve is placed at the top and intermediate grades are placed underneath, down to the finest grade required. The range of particle sizes produced from the various sieves is usually indicated as, for example, $-100 + 150$. This shows that the particles have passed through the 100 mesh, but are too large to pass through the 150 mesh.

MIXING OF POWDERS

If powders of different metals are mixed to form an alloy powder, the process must be controlled to ensure uniformity in the density and composition of the finished compact. Mixing is affected by the size variation of the particles of powder, density variation between particles (if more than one metal is present), particle distribution and the duration of mixing. Particle size and distribution are determined by carefully weighing out the correct proportions of powders prior to mixing. Also at this stage the necessary lubricants to ensure smooth operation of the die and punch are added to the powder.

One of the problems frequently encountered during mixing is the complete or partial segregation of the powders. To overcome this problem, the mixing time or method of mixing must be altered. This is only brought about by trial and error, or by experience on the part of the operator. The time required to completely mix the powder varies considerably—from a few minutes in some instances up to a period of 24 hours or more. It is important to realize that for most blends of powder there is an **optimum** mixing time; operation of the mixer beyond this time merely results in a steady unmixing of the powders. In general the greater the variation in size or density (especially) in the powder blend, the shorter will be the optimum time of mixing. Unfortunately, it is not possible to give precise data on suitable mixing times, since every mixing system is to some extent unique.

Powders may be mixed in the laboratory using a ball mill but dispensing with the balls. However, better results will be obtained

if mixing is carried out in a tumbling-barrel-type mixer, which rotates or tumbles about its longitudinal axis. Mixing by hand is not recommended owing to the fact that actual mixing of the powders is likely to be intermittent, owing to fatigue on the part of the operator. If powders are of different densities, problems may occur because the heavier powder will separate and fall to the bottom of the mixer. However, it has been suggested that if the less dense powder is finer than the heavy powder, this problem is lessened.

WET MIXING

The process of wet mixing will generally assist in producing a uniform mixture. This may be carried out by adding benzene or acetone in sufficient quantity to bring the powder to the consistency of thin paste. When the mixing process has been completed, the benzene or acetone may be removed by drying the mix in air. Forced drying may be employed **carefully,** by heating the powder. However, care should be taken to avoid the possibility of the powder catching fire due to the ignition of heavy vapours given off from the benzene or acetone. Where possible, the drying operation should be carried out in a well-ventilated area. The final alloy is produced when the compacted powder is sintered, due to diffusion of the individual particles.

Dies and Punches

In this section the dies and punches used to compact metal powders are dealt with. Whereas in industry these items are often large in cross-section, and designed to withstand the rigours of heavy, powerful industrial presses and the continuous usage associated with mass production, under laboratory conditions the dies and punches used are smaller and lighter in construction. This is because in the laboratory we are primarily concerned with the forming of compacts which are usually small in size and simple in shape.

210

The dies and punches used to compact the powdered metal are usually made from high-grade tool steel. They must be machined and ground to within very close limits of tolerance. If, for example, the clearance between the punch and the walls of the die is too close, then these components will become "jammed" together. This is caused by friction during the operation of the punch. Also when the punch is inserted into the die, air will be trapped within the die, leading to poor compaction owing to the creation of a pneumatic effect. The compact will also be extremely porous, and cracks may be formed because the air trapped within the die will be forced into the compact. Some form of lubrication is often employed—for example, stearic acid, vinyl chloride in acetone, graphite or a mixture of paraffin and wax.
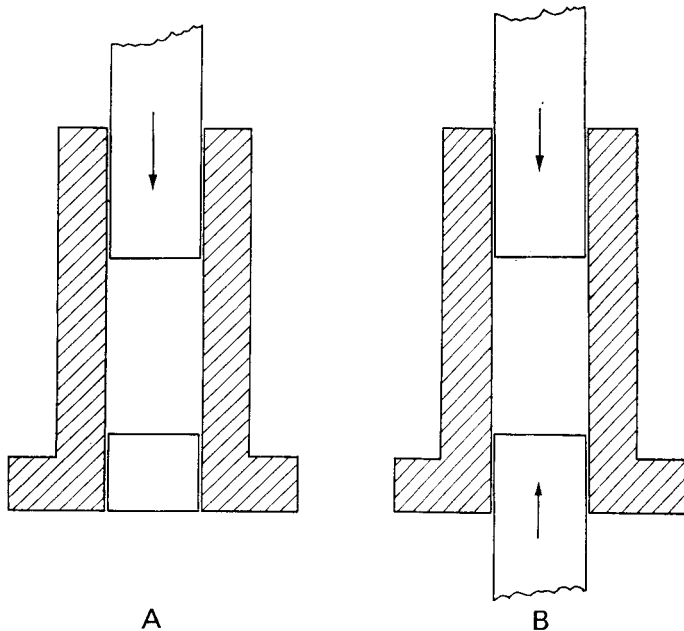

FIG. 7.4. Simple die and punch arrangement: (A) single action; (B) double action.

211

This will assist in relieving any tendency of the punch to stick. However, if the punch is made with too much clearance, powder will be forced up the sides of the die during the pressing operation. A slight taper is often purposely machined at the exit end of the die to facilitate easy ejection of the compact. This ensures that the die is not damaged as the compact is ejected, owing to the lateral pressure caused by the elastic recovery of the compact after pressing. Also, because the compact is in a brittle state, unnecessary damage is avoided. Figure 7.4 illustrates two simple die and punch arrangements.

Pressing

The most important operation in powder metallurgy is without a doubt the pressing of the powder into its final shape. Metal powders suffer from two common faults. In the first instance they tend to be attacked by, and to absorb, moisture. This can be reduced to a minimum by proper storage of the powder and, more important, by manufacturing components under controlled conditions—for example by ensuring that the work area or room is kept at a relatively constant temperature, and dust and foreign matter are not allowed to come into contact with the powder. Also the dies and punches and the press should be kept clean. Apart from the possibility of contamination by moisture, it is possible for a compact to become contaminated by residues of powder from previous pressing operations.

In the second instance, powders when subjected to high pressures do not transmit the pressure uniformly throughout their bulk in the manner of a fluid. The pressure within the powder decreases considerably as the distance from the punch face increases, with the result that only a portion of the compact is subjected to the required pressure. It is this effect which exercises the greatest restriction on the size of compacts that can be produced from powders. As a result there is a strong tendency for compacts to vary considerably in density (in "green compacts", the low-density regions can be very brittle or friable), with

the result that sintering causes anomalous shrinkage behaviour leading to distortion. (See Fig. 7.5 for a diagrammatic illustration of this point.) Density variations due to poor flow of the powder (which to some degree depends on the shape of the
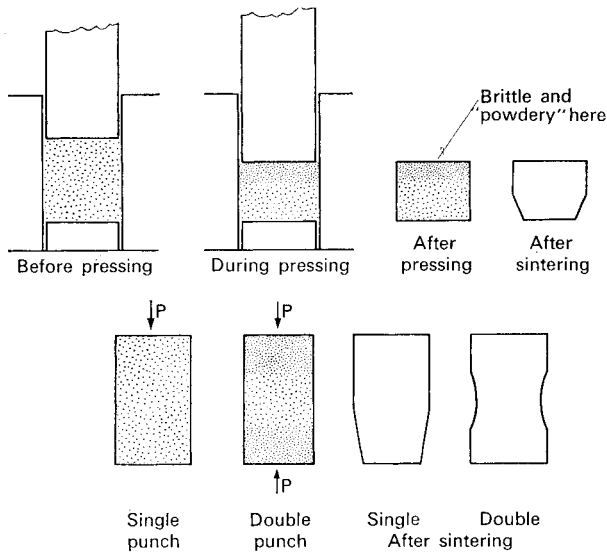

FIG. 7.5. Illustrating that sintering leads to shrinkage and distortion.

powder particles) and to friction between the powder and the die walls can be greatly reduced by the use of double action punches, and by the suitable addition of lubricant powders. It must be remembered that there is a limit to the amount of lubricant which can be added without detriment to the green strength of the compact. Lubricants can also be an additional complication during the sintering process, since at high temperatures they (or their decomposition products) may contaminate the powder.

213

PRESSING OPERATION

Pressing is usually carried out at room temperature, the powder being placed in the die and compacted under pressures varying from 10 to 50 t/in². Hot pressing which involves the sintering of the compact simultaneously with pressing, may be employed, but involves very much more complex apparatus. For the present text, we shall assume that pressing is carried out cold; readers requiring further information on aspects of hot pressing should consult the Bibliography. The press used for forming the compact is usually hydraulically operated and if necessary a machine of the type used for tensile and compression testing may be employed (see Chapter 4).

The method used for the production of a compact in the laboratory is given in the example and the shape of the compact will be assumed to be cylindrical. The operation is carried out as follows, using the die and punch illustrated in Fig. 7.4. The powdered metal already in the correctly mixed state is poured from a hopper into the die, tapped to settle the powder to the maximum "tap density" and made level. There should be sufficient room to allow for the insertion of the punch. (Alternatively where accurate size control is desired, the powder may be weighed into the die.) The punch should be inserted, and the pressure applied by way of the hydraulic press. The pressure is retained for a predetermined time, derived from trial pressings, after which it is released, and the compact carefully ejected from the die. If a single-action punch is used (see Fig. 7.5) the compact will be of greater density at the top than at the bottom because of the poor transmission of the pressure. However, the double-acting punch will produce a compact in which its density will be more uniform, because pressure applied is rather more uniformly conducted through the compact, although there is a tendency for a low-density region to remain in the centre unless the compact is a very squat cylinder. Ejection of the compact from the single-action punch is usually carried out by placing the die on a metal ring, of suitable height to allow the compact to be

pushed out of the bottom of the die by further travel of the punch. In the case of the double-action punch, the top punch is withdrawn and the lower punch is used to eject the compact from the top of the die. At the ejection stage, the compact is in a weak and brittle state known as a "green compact", and must be handled with great care. Therefore to render it suitable for use as a finished component, it has to pass through another process called **sintering**.

ISOSTATIC PRESSING

Isostatic pressing may be used to produce complex shapes from metal powders which are not readily obtainable by conventional methods. As mentioned earlier, powders do not behave as liquids in respect of the transmission of applied pressure. Hence the pressure applied in one direction is not transmitted along the axis at a right angle to the direction of pressing, and therefore the density of a green compact is normally non-uniform. To produce components of complex shape and with a closer approximation to uniform density, the pressure must be applied equally and at right angles to **all** parts of the surface of the compact; this may be achieved by isostatic pressing.

Isostatic pressing involves the use of a reversible gel, which is used in the form of a mould within the compression chamber or die (see Fig. 7.6). The most commonly used gel is a plasticized and gelled polyvinyl chloride with the trade-name Vinamold. Before compacting of the powder is carried out, the Vinamold is melted and poured around a pattern conforming to the final shape of the component and which allows for subsequent shrinkage of the compact; thus on solidification the gel forms a mould. Because the material has low thermal conductivity, it is cut into small segments and melted in a beaker, or a similar container, placed over a bunsen burner. Also because of its low thermal conductivity, the flame of the bunsen burner should be kept low and the Vinamold stirred frequently to avoid excessive burning.
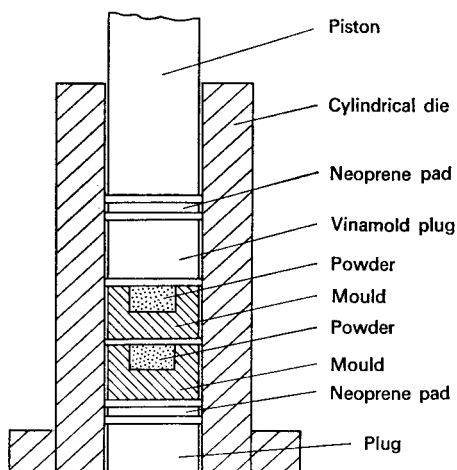
215

FIG. 7.6. Isostatic pressing involving the use of a reversible gel.

When it has solidified, the mould and pattern may be removed from the die and the pattern from the mould. A plug is then made in a similar way as a cover or lid for the powder. When the mould and plug have completely hardened (this may take up to 12 hours) a small shallow groove should be cut along the length to allow for the passage of air which may be trapped within the die during the pressing operation. The mould usually has a slightly greasy feel, therefore it should be lightly dusted with french chalk to facilitate easy insertion into the die and to prevent it from sticking to the walls of the die during compaction. The moulds made from this type of gel may be re-used for subsequent pressing operations and may also be remelted (i.e. reversed) and used again to produce moulds of different shapes, when so desired.

Using the punch and die arrangement illustrated in Fig. 7.4, the powder is poured into the mould and made level. The mould is then inserted into the die and the Vinamold plug is placed into position. At this stage a pad of neoprene or similar material may be placed over the plug as a precautionary measure, owing to the

216

fact that the Vinamold may be extruded past the punch when pressure is applied. A clearance of 0·001 in. is sufficient to allow extrusion past the punch; this stresses the need for close tolerances between the die and punch. Pressure is applied by way of a suitable press. After the compacting operation has been completed, the mould and compact may be removed from the die by the conventional method described earlier.

Sintering

The green compact is not ready for use as a finished component until its mechanical properties have been developed further by a process called sintering. Sintering really means heat treatment, and involves a form of pressure welding of the particles to each other. It is usually carried out at a much lower temperature than the melting point of the original metal (although where more than one metal is present, the melting point of the one with the lowest melting point may sometimes be exceeded). It removes residual stresses and absorbed liquids or solids (e.g. lubricants) and gases, thus leaving a compact of high density and high strength by promoting grain growth across the particle interfaces.

Electric resistance furnaces are usually used to sinter compacts, the most common type being the muffle and tube furnace. When sintering is carried out as a continuous process, for example in industrial concerns, the furnace is usually equipped with a preheating chamber and a cooling chamber. However, in the laboratory, where the quantity of compacts being produced is usually small, the ancillary heating and cooling chambers are not required, and are dispensed with for the sake of simplicity in the design of the sintering apparatus.

The sintering operation may be divided into two stages on occasion, if the product needs machining but is inconveniently hard or brittle, for example with magnet alloys and alloys for cutting tool tips. The green compact then undergoes a "presinter", which is designed merely to give it adequate strength to

machine well without risk of it breaking up. After the machining process, the compact is carefully cleaned to remove oil or grease from the pores and then given the second and final sinter in order to develop its maximum hardness.

During the sintering operation it is important to control the atmosphere of the furnace, to prevent the formation of oxide on the surface of the compact. Refractory metals, copper and molybdenum (which, alone among the more familiar metals, forms a volatile oxide) are especially susceptible to oxidation at elevated temperatures. Whilst they may be sintered in an atmosphere of dry hydrogen, sintering under vacuum may also be employed. Table 19 lists some appropriate controlled atmospheres for the commoner metals.

TABLE 19. CONTROLLED ATMOSPHERES FOR COMMON METALS

Metal	Atmosphere
Tungsten	Hydrogen
Molybdenum	Hydrogen
Cobalt/tungsten carbide	Hydrogen
Iron	Hydrogen
Nickel	Hydrogen
Copper	Hydrogen or vacuum
Chromium	Vacuum
Aluminium	Vacuum
Tantalum	Vacuum
Vanadium	Vacuum
Platinum	Air
Gold	Air
Silver	Air or hydrogen

SINTERING UNDER VACUUM

An efficient furnace and vacuum system suitable for most applications where the sintering temperature does not exceed 1100°C may be easily constructed in the laboratory. For details regarding the construction of a suitable furnace, reference may

be made to Chapter 2. For temperatures above 1100 °C, a suitable furnace may be obtained commercially, whilst a simple vacuum system may be constructed by consulting the relevant sections of Chapter 6.

The procedure for sintering is as follows: the green compact is placed in the furnace and allowed to outgas at a temperature of approximately 200 °C. The slow heating is also beneficial in removing traces of lubricant. When the desired degree of vacuum has been obtained, at which time any significant amount of lubricant should have been removed, the temperature should be increased slowly to the optimum sintering temperature. When sintering is completed the compact should, of course, be cooled to room temperature before removal from the protective atmosphere.

It is unfortunately not possible to give the specific temperatures and times for sintering, as it is a highly complex process. It depends amongst other things upon the nature of the constituent powders, and the atmosphere during sintering, the density of the green compact, the required density of the final product and the time and temperature of sintering. Also these variables are partly interdependent; for example, the higher the temperature, the shorter the time to attain a given degree of sintering and vice versa. Thus the selected values given in Table 20 must be treated with caution, since they can only give an indication of the order of time and temperature required for adequate sintering; in the final analysis, the user must establish for himself the optimum conditions for his requirements and his experimental set-up.

TABLE 20. SUGGESTED SINTERING TEMPERATURES AND TIMES

Metal	Temperature (°C)	Time
Bronze	700–800	1–2 hr
Copper	850–950	1 hr
Iron	900–1100	45 min
Magnet alloys	1200–1300	2 hr
Refractory metals	2000–2500	30 min

Miscellaneous Questions

THE purpose of including this short selection of questions from past metallurgical examination papers is to provide a workable guide for students. The author expresses his thanks to the following examining bodies for permission to use these questions:

Northern Counties Technical Examination Council.
Union of Lancashire and Cheshire Institutes.
Bristol Technical College.
Union of Educational Institutes.
Welsh Joint Education Committee.
City and Guilds of London Institute.

1. Give an account of the principles of the magnetic crack detection technique for the non-destructive testing of metals.
Name three other methods of non-destructive testing and mention briefly a typical application of each of these three. (C.G.L.I.)

2. With the aid of a diagram describe a machine used for the notched-bar impact testing of metallic materials, and briefly explain the operation of the machine. (U.E.I.)

3. Sketch and describe either a radiation or optical pyrometer. State the conditions which affect the operation of the instrument you describe and mention for what applications you would use it. (U.E.I.)

4. What changes are brought about in the structure and properties of a $\frac{1}{2}$ in. steel bar of 0·4 per cent plain carbon steel when it is heated to 850°C and then quenched in water? How may the structure and properties be modified by subsequent tempering? (C.G.L.I.)

5. Give the meaning of each of the following terms as applied to the heat treatment of steel: (a) annealing, (b) normalizing, (c) overheating, as opposed to burning. (C.G.L.I.)

6. Explain the principles on which hardness tests are based. Describe two different hardness tests, in each case referring to a simple sketch of the machine used. (U.E.I.)

7. Distinguish between the following terms: (a) ductility and malleability, (b) brazing and welding, (c) martensite and pearlite, (d) wrought iron and cast iron. (W.J.E.C.)

8. State the approximate composition of each of the following: (a) a soft solder for the joining of lead pipes, (b) a soft solder for the joining of electrical connections, (c) a spelter for the joining of mild steel components, (d) a hard solder for the joining of copper and brass. (W.J.E.C.)

9. Describe one method for the measurement of the hardness of metals. What general precautions are necessary to obtain accurate results in hardness testing? (C.G.L.I.)

10. Outline the principles on which a pyrometer is designed in order to investigate, by means of cooling curves, the solidification of metals in the temperature range of 500 °C to 1000 °C. Describe such an installation. What is the significance of "cold junction temperature"? (U.L.C.I.)

11. Using a typical stress–strain diagram, show the significance of (a) proof stress and (b) yield point. Explain how each is determined in a simple tensile-test. (U.L.C.I.)

12. Compare the metallurgical features of joints made by soft soldering and by welding. What are the common defects associated with each type of joint? (C.G.L.I.)

13. Mention three methods of welding and discuss the metallurgical problems of making a satisfactory joint (B.T.C.)

14. Discuss the important properties of a refractory material. Mention three types and examples of the uses of each. (B.T.C.)

15. A thermocouple gave the following readings:

Correct temp.,°C	200	300	400	500	600	700	800	900
e.m.f., mV	1·3	2·1	3·0	4·0	5·2	6·7	8·7	10·0

The millivoltmeter was replaced by a similar instrument with a linear scale marked in °C adjusted to read correctly at 400°C and 800°C. Plot an error curve between 300°C and 900°C for this new arrangement. (C.G.L.I.)

16. State the principles on which measurement of temperature by a thermocouple depends. Discuss the main sources of error in thermoelectric pyrometry. (U.E.I.)

17. Draw labelled sketches of any two metallurgical furnaces of different types, indicating clearly the main features of such furnaces. State one use for each type of furnace, mention the refractory material which would be used for the important part or parts of the lining, and explain why such a refractory is used. (U.E.I.)

18. What is meant by (a) toughness; (b) elasticity; (c) malleability; (d) ductility? Select two of these, and describe, in each case, a test used for determining the property quantitatively. Include simple sketches which show the principles of the apparatus used. (N.C.T.E.C.)

19. Describe in detail how a longitudinal section of a $\frac{1}{2}$ in. long 6 B.A. brass bolt should be prepared for examination of the structure under the microscope. (C.G.L.I.)

20. Describe two methods of making a brazed joint. What factors would you consider in deciding which of these methods should be used? (C.G.L.I.)

21. Draw a simple sketch which shows the principles on which the metallurgical microscope is based. Describe the preparation of a metallurgical specimen for micro-examination, stating the precautions which are necessary in order to obtain satisfactory results. (N.C.T.E.C.)

22. A tensile test was made on a test piece of 0·25 in². cross-sectional area. At a load of 1 ton an extensometer having a 2 in. gauge length was fixed to the test piece and the bezel set to zero. The load was then increased and the following readings taken:

Load, tons	Extension, inches
1·0	0·0000
2·0	0·0006
3·0	0·0012
4·0	0·0018
5·0	0·0024
6·0	0·0030
6·5	0·0034
7·0	0·0040
7·5	0·0048
8·0	0·0062
8·5	0·0078

Plot a graph of load against extension, and from this determine the 0·1 per cent proof stress.

The extensometer was removed and the load increased until the test piece broke at 10·3 tons load. The broken halves when fitted together showed an extension of 0·45 in. on the gauge length. Calculate the ultimate tensile stress and the percentage elongation. (C.G.L.I.)

23. Why is the tensile test considered to be a most useful test? Give details of the information which may be derived from the test. What effect has the gauge length on the results? Compare the stress/strain curves for mild steel, grey cast iron and copper. (B.T.C.)

24. You are provided with a standard tensile specimen in each of the following materials: (a) brass, (b) dural, (c) mild steel.

Use the Hounsfield Tensometer to plot the stress/strain curve for each material. Determine the U.T.S., ductility and Vickers hardness of each material. Comment on the nature of the curves. What may be deduced from the curve for the mild steel? (B.T.C.)

25. Give a brief description of the principles of two methods of non-destructive testing applicable to the detection of surface cracks. State the advantages and the limitations of each method. (U.E.I.)

26. Sketch a metallurgical microscope, indicating the chief features of its construction. How does the illuminating system differ from that of an ordinary microscope? (U.E.I.)

Appendix 1

Frequently Used Letters of the Greek Alphabet

α	alpha	η	eta	π	pi
β	beta	θ	theta	ϱ	rho
γ	gamma	ι	iota	σ	sigma
$\left.\begin{array}{l}\Delta\\\delta\end{array}\right\}$	delta	$\varkappa$	kappa	τ	tau
		λ	lambda	φ	phi
ε	epsilon	μ	mu	ω	omega
ζ	zeta				

Appendix 2

Metric Weights and Measures

10 millimetres	= 1 centimetre
10 centimetres	= 1 decimetre
10 decimetres	= 1 metre
1000 metres	= 1 kilometre

100 sq. millimetres	= 1 sq. centimetre
100 sq. centimetres	= 1 sq. decimetre
100 sq. decimetres	= 1 sq. metre

1000 cu. millimetres	= 1 cu. centimetre
1000 cu. centimetres	= 1 cu. decimetre
1000 cu. decimetres	= 1 cu. metre

10 millilitres	= 1 centilitre
10 centilitres	= 1 decilitre
10 decilitres	= 1 litre
100 litres	= 1 hectolitre

N.B. 1 litre = 1 cubic decimetre = the volume of 1 kilogram of distilled water at 4 °C and 760 mm of mercury pressure.

10 milligrams	= 1 centigram
10 centigrams	= 1 decigram
10 decigrams	= 1 gram
10 grams	= 1 decagram
10 decagrams	= 1 hectogram
10 hectograms	= 1 kilogram
1000 kilograms	= 1 tonne

Appendix 3

Conversion Tables

1 inch	= 25·4 millimetres
1 foot	= 304·8 millimetres
1 yard	= 0·9144 metre
1 mile	= 1·6093 kilometres
1 millimetre	= 0·03937 inch
1 metre	= 39·37 inches = 3·2808 feet
1 kilometre	= 0·6214 mile

1 sq. inch	= 645·16 millimetres
1 sq. foot	= 929·03 centimetres
1 sq. yard	= 0·83613 sq. metre
1 sq. millimetre	= 0·00155 sq. inch
1 sq. centimetre	= 0·155 sq. inch
1 sq. metre	= 10·764 sq. feet

1 cu. inch	= 16·387 cu. centimetres
1 cu. foot	= 0·028317 cu. metre
1 cu. yard	= 0·76455 cu. metre
1 cu. centimetre	= 0·061024 cu. inch
1 cu. metre	= 35·315 cu. feet
	= 1·308 cu. yards

1 ounce	= 28·3495 grams
1 pound	= 0·45359 kilogram
1 hundredweight	= 50·802 kilograms
1 ton	= 1016·05 kilograms
1 gram	= 0·035274 ounce
1 kilogram	= 2·20462 pounds
1 tonne	= 0·9842 pounds
(1000 kilograms)	

1 pint	= 0·568 litre
1 quart	= 1·136 litres
1 gallon	= 4·54596 litres
1 centilitre	= 0·070 gill
1 decilitre	= 0·176 pint
1 litre	= 1·75980 pints
	= 0·21998 gallon

1 ton per in².	= 1·57488 kilograms per mm²
1 pound per in².	= 0·070307 kilogram per cm²
1 kilogram per mm²	= 0·63497 ton per in².
1 kilogram per cm²	= 14·2233 pounds per in².

1 lb per inch	= 17·8580 kg per metre
1 lb per foot	= 1·48816 kg per metre
1 lb per yard	= 0·49606 kg per metre
1 lb per in².	= 0·0703 kg per cm²
1 lb per ft²	= 4·88243 kg per m²
1 ton per in².	= 1·575 kg per mm²
1 lb per in³.	= 0·02768 kg per cm³
1 lb per ft³	= 16·0185 kg per m³
1 gallon per min	= 272·758 litres per hr
1 gallon per hr	= 0·07577 litre per min
1 ft³ per min	= 1·69901 m³ per hr
1 foot pound	= 0·13826 kg
	= 1·35582 joules
1 inch-ton	= 25·8000 kg
1 horse-power	= 76·0402 kg per sec
	= 0·74570 kilowatt
	= 1·0139 force de cheval
1 B.t.u.	= 0·25200 kg-calorie
1 kg per metre	= 0·0560 lb per inch
	= 0·6720 lb per foot
	= 2·0160 lb per yard
1 kg per mm²	= 0·6350 ton per in².
1 kg per cm²	= 14·223 lb per in².
1 kg per m²	= 0·2048 lb per ft²

1 kg per cm³	= 36·1273 lb per in³.
	= 62428·0 lb per ft³
1 litre per min	= 13·1985 gallons per hr
1 litre per hr	= 0·00037 gallon per min
1 m³ per hr	= 0·58879 ft³ per min
1 kg	= 7·23301 foot-pounds
1 joule	= 0·73756 foot-pound
1 kg	= 0·03870 inch-ton
1 kg per second	= 0·01315 horse-power
1 kilowatt	= 1·34102 horse-power
1 force de cheval	= 0·9863 horse-power
1 kg-calorie	= 3·96800 B.t.u.

Appendix 4

Comparison Between Different Hardness Scales

Brinell			Vickers	Rockwell "c" 120° diamond cone
Dia. (mm)	Hardness number	Calculated tonnage		
2·20	782	171	1170	70
2·25	744	162	1050	68
2·30	713	155	935	66
2·35	683	149	865	64
2·40	652	142	802	62
2·45	627	136	756	60
2·50	600	131	708	58
2·55	578	126	670	57
2·60	555	121	634	55
2·65	532	116	598	53
2·70	512	112	570	52
2·75	495	108	545	50
2·80	477	104	521	49
2·85	460	100	500	47
2·90	444	97	480	46
2·95	430	94	462	45
3·00	418	91	447	44
3·05	402	88	426	42
3·10	387	84	409	41
3·15	375	82	394	40
3·20	364	79	382	38
3·25	351	76	366	37
3·30	340	74	352	36
3·35	332	72	343	35
3·40	321	70	331	34
3·45	311	68	321	33
3·50	302	66	311	32

Appendix 5

Functions of π

$$\pi = 3 \cdot 1416 \qquad \pi^2 = 9 \cdot 8696 \qquad \pi^3 = 31 \cdot 006$$

$$\frac{1}{\pi} = 0 \cdot 3183 \qquad \sqrt{\pi} = 1 \cdot 7725 \qquad \sqrt[3]{\pi} = 1 \cdot 4646$$

$$\frac{\pi}{2} = 1 \cdot 5708 \qquad \frac{\pi}{4} = 0 \cdot 7854 \qquad \frac{\pi}{6} = 0 \cdot 5236$$

Appendix 6

Imperial Standard Wire Gauge

Number	Diameter		Number	Diameter	
	in.	mm		in.	mm
0,000,000	0·500	12·70	23	0·024	0·610
000,000	0·464	11·79	24	0·022	0·559
00,000	0·432	10·97	25	0·020	0·508
0,000	0·400	10·16	26	0·018	0·457
000	0·372	9·45	27	0·0164	0·417
00	0·348	8·84	28	0·0148	0·376
0	0·324	8·23	29	0·0136	0·345
1	0·300	7·62	30	0·0124	0·315
2	0·276	7·01	31	0·0116	0·295
3	0·252	6·40	32	0·0108	0·274
4	0·232	5·89	33	0·0100	0·254
5	0·212	5·38	34	0·0092	0·234
6	0·192	4·88	35	0·0084	0·213
7	0·176	4·47	36	0·0076	0·193
8	0·160	4·06	37	0·0068	0·173
9	0·144	3·66	38	0·0060	0·152
10	0·128	3·25	39	0·0052	0·132
11	0·116	2·95	40	0·0048	0·122
12	0·104	2·64	41	0·0044	0·112
13	0·092	2·34	42	0·0040	0·102
14	0·080	2·03	43	0·0036	0·0914
15	0·072	1·83	44	0·0032	0·0813
16	0·064	1·63	45	0·0028	0·0711
17	0·056	1·42	46	0·0024	0·0610
18	0·048	1·22	47	0·0020	0·0508
19	0·040	1·016	48	0·0016	0·0406
20	0·036	0·914	49	0·0012	0·0305
21	0·032	0·813	50	0·0010	0·0254
22	0·028	0·711			

Appendix 7

Colour Guide to Furnace Temperature

Colour	Approximate temperature (°C)
Pale red	525
Dull red	700
Cherry red	900
Bright cherry red	1000
Deep orange	1100
Bright orange	1200
White	1300
Bright white	1400
Dazzling white	1500

Appendix 8

Calibration Tables for Thermocouples

Chromel/Alumel

Temperature (°C)	mV	Temperature (°C)	mV
0	0	600	24·91
50	2·02	650	27·03
100	4·10	700	29·14
150	6·13	750	31·23
200	8·13	800	33·30
250	10·16	850	35·34
300	12·21	900	37·36
350	14·29	950	39·35
400	16·40	1000	41·31
450	18·51	1050	43·25
500	20·65	1100	45·16
550	22·78		

Iron/Constantan

Temperature (°C)	mV
0	0
100	5·27
200	10·78
300	16·33
400	21·85
500	27·39
600	33·11
700	39·15
800	45·53

Copper/Constantan

Temperature (°C)	mV
0	0
50	2·02
100	4·24
150	6·63
200	9·18
250	11·86
300	14·67
350	17·58
400	20·59

Appendix 9

Physical Properties of Metals

Metal	Symbol	Melting point (°C)	Crystal structure
Aluminium	Al	660	Face-centred cubic
Antimony	Sb	630	Rhombohedral
Cadmium	Cd	320	Close-packed hexagonal
Chromium	Cr	1890	Body-centred cubic
Cobalt	Co	1495	Close-packed hexagonal
Copper	Cu	1083	Face-centred cubic
Gold	Au	1063	Face-centred cubic
Iron	Fe	1539	Body-centred cubic
Lead	Pb	327	Face-centred cubic
Magnesium	Mg	650	Close-packed hexagonal
Manganese	Mn	1245	Cubic (rather complex)
Molybdenum	Mo	2625	Body-centred cubic
Nickel	Ni	1455	Face-centred cubic
Potassium	K	63	Body-centred cubic
Silver	Ag	960	Face-centred cubic
Sodium	Na	97	Body-centred cubic
Tin	Sn	231	Body-centred tetragonal
Titanium	Ti	1820	Close-packed hexagonal
Tungsten	W	3410	Body-centred cubic
Vanadium	V	1735	Body-centred cubic
Zinc	Zn	419	Close-packed hexagonal

Bibliography

Practical Microscopical Metallography, 3rd edition, GREAVES and WRIGHTON (Chapman & Hall Ltd.).

Polarized Light in Metallography, CONN and BRADSHAW (Butterworths Ltd., London, 1952).

Metallurgy for Engineers, 2nd edition, ROLLASON, E.C. (Edward Arnold).

Engineering Metallurgy, HIGGINS, R.A. (The Universities Press Ltd.).

Text Book of Metallurgy, BAILEY, A.R. (Macmillan & Co. Ltd.).

Metals Handbook, 1948 edition (The American Society for Metals).

Instruction Handbook for a Disappearing Filament Pyrometer (Cambridge Instrument Co. Ltd.).

Procedures in Experimental Metallurgy, SEYBOLT, A.U. and BURKE, J.E. (John Wiley, New York).

Steel and its Heat Treatment, Vol.2, 5th edition, BULLENS, D.K. (John Wiley, New York, 1948).

Mechanical Testing of Materials, FENNER, A.J. (George Newnes Ltd., London).

Direct Reading Hardness Testing (Rockwell), (British Standard 891:1940).

The Non-Destructive Testing of Metals (Institution of Metallurgists, 1954).

Magnetic and Electrical Methods of Non-Destructive Testing, LEWIS, D.M. (George Allen & Unwin, 1951).

Industrial Radiography (Kodak Ltd., London).

Select Methods of Metallurgical Analysis, NAISH, W.A. (Chapman & Hall Ltd.).

Low Temperature Silver Brazing, Cat. 1100 (Johnson, Matthey Co. Ltd.).

Vacuum Techniques in Metallurgy, BELK, J.A. (Pergamon Press).

Fundamental Principles of Powder Metallurgy, JONES, W.D. (Edward Arnold Ltd.).

Powder Metallurgy, its Physics and Production, SCHWARZKOPF (Macmillan & Co., New York, 1947).

Practical Course in Powder Metallurgy, YARNTON, D. and ARGYLE, M. (Cassell & Co. Ltd., London).

Index

237

Index

238