METALLOGRAPHY

Objective

To study the structural characteristics or constitution of a metal or an alloy in relation to its physical and mechanical properties.

Introduction

There are two examination methods in metallography:
I) Macroscopy
2) Microscopy

In macroscopy the examination of the structural characteristics or chemical characteristics of a metal or an alloy is done by the unaided eye or with the aid of a low-power microscope or binocular, usually under l0x.

In microscopy similar examination is done with the prepared metal specimens, employing magnifications with the optical microscope of from 100x to as high as 2000x.

Specimen preparation

1. Grinding

A small piece of specimen is cut by a metal-cutting-saw. After cutting operation, burrs on the edges of the specimen should be carefully removed by a fine file or coarse grinding paper.

The silicon carbide grinding papers are held flat in a unit containing water facility for lubrication purpose. Each unit contains four grades of papers, starting with grade 400 (coarse) and finishing with grade 1200 (fine). Grinding of the work piece is done by starting with the coarse papers and then continuing with the fine papers. In each stage, grinding is done by rubbing the specimen backwards and forwards on the grinding paper in one direction only, until the surface is completely ground, that is, until only grinding marks due to this particular paper can be seen to cover the whole surface.

The specimen is washed thoroughly to remove coarse silicon carbide particles before proceeding to a finer paper.

The direction of grinding is changed from paper to paper, so that the removal of previous grinding marks is easily observed. The extra time spent on each paper should be increased as the finer papers are used. At the end of the grinding sequence, the specimen is washed thoroughly and dried. Now, the specimen is ready for polishing.

2. Polishing

The polishing is done on rotating wheels covered by a special cloth. Alumina is employed as polishing agent. The 1-micron size is commonly used, but the total polishing time shortened by starting on the 7 or 3 micron grade.
The pad should be kept well supplied with lubricant. The specimen should be held firmly in contact with the polishing wheel, but excessive pressure should be avoided. During polishing the specimen should be rotated or moved around the wheel so as to give an even polish. The specimen should be thoroughly cleaned and dried between each wheel.

3. Etching

Before etching, it is essential to ensure that the polished surface is grease and smear free. If the final polishing has involved the use of magnesia (in the form of an aqueous paste of fine magnesia) or alumina (in the form of an aqueous suspension of fine alumina), then thorough washing followed by drying off with acetone or alcohol will give a suitable surface, although it must not be fingered afterwards.

Etching is generally done by swabbing. Etching times will vary from specimen to specimen, however, a good general, procedure is to observe the surface during etching, and to remove the specimen when evidence of the grains first appears. Microscopical examination will then reveal whether the degree of etching is sufficient. Further etching can then follow to strengthen up the details as required.

After each etching, the specimen should be thoroughly washed in running water, followed by drying off with acetone or alcohol.

* As a guide the following etchants are commonly used:

Alcoholic Ferric Chloride	-copper alloys
Mixed Acids	-aluminum alloys
Nital (ethyl alcohol+ 2% HN03)	-iron and steel
Dilute HCI	-zinc alloys

A.MICROSCOPICAL EXAMINATION

The microstructural study of a material can provide information regarding the morphology and distribution of constituent phases as well as the nature and pattern of certain crystal imperfections. Optical metallography is a basic tool of material scientists, since the equipment is relatively inexpensive and the images can be obtained and interpreted easily. Distribution and morphology of the phases can be studied and, if their properties are known, a quantitative analysis of the micrographs provides some information about the bulk properties of the specimen. A limited study of line and surface informations is also possible with the optical microscope.

In order to obtain reproducible results, with good contrast in the image, the specimen surface is polished and subsequently etched with appropriate reagents before microscopic examination. In a polished specimen, the etching not only delineates grain boundaries, but also allows the different phases to be distinguished by differences in brightness, shape, and color of the grain. Differences in contrast may result from differences in light absorption characteristics of the phases. Etching results in preferential attack or preferential colouring of the surface. The preferential attack is electrochemical corrosion; it is well known that different materials corrode at different rates. Grain boundaries are often anodic to the bulk metal in the interior of the grain and so are etched away preferentially and delineated. Staining is produced by the deposition of solid etch product on the specimen surface. This is formed by chemical reaction between the etchant and the specimen. Under favorable conditions the use of a proper etchant enables the identification of constituents. Failure analysis depends a great deal on metallographic examination.

Microstructural examination can provide quantitative information about the following parameters:

1) The grain size of specimens
2) The amount of interfacial area per unit volume
3) The dimensions of constituent phases
4) The amount and distribution of phases.

Magnifications up to 1000x can be obtained with a resolution of 2 μ. For grain size measurements, the grains along a line, circle, or within a known area are counted. It would be useful to obtain an average value of grain diameter from a microstructural section. In linear intercept method, the grains intercepted by a theoretical line on the specimen surface are counted (Fig. 1). The number of grain boundaries intersected per unit length of a test line (n_L) can be noted. The average grain size is indicated by the inverse of n_L, corrected for the magnification, M, of the micrograph. In general, then, the average grain diameter, d, is given by

$$d = C/n_L.M$$

where C is some constant greater than 1 (Typically, a value of C=1.5 is adequate).

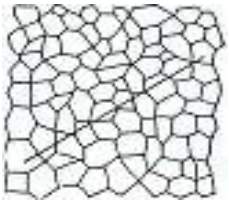

Fig.1 Linear intercept method for grain size determination

Experimental

Specimens are going to be polished and etched as explained above. With an unmounted specimen, even if it is almost level, it is best to fix it on a glass or metal slide by means of a piece of plasticine. The prepared surface is made level by means of a suitable levelling device.

The correct procedure is to start with low power examination in order to obtain an overall picture, and then successively higher magnifications are used to resolve the fine details. In focusing, the stage is gradually moved towards the objective and when the image appears, focusing is completed with the fine adjustment.

It is almost good practice to examine specimens first in the polished condition, as certain features, such as the presence of inclusions, cracks, porosity, and sometimes even the different phases, are revealed. This is followed by an examination in the etched condition.

Do not examine one or two fields only. It should always be remembered that the structure of a given specimen exists in three dimensions, whereas the prepared surface represents a two-dimensional picture in the plane of the micro-section. This fact should be taken into account in the interpretation of the geometry of any particular configuration.

Results

The investigation result of the optical microscopy is going to be recorded as follows:

1. Examine each specimen and sketch typical microstructure
2. Estimate the carbon content of the steels.
3. Calculate the average grain diameter of the micrograph, using linear intercept method.

B. MACROSCOPIC EXAMINATION OF METALS

Objective

To examine the nature of inhomogenities and flow lines in a metal by unaided eye or with the aid of a low-powered microscope or magnifying glass.

Introduction

Metallurgical data obtained by a chemical and metallographic analysis of a metal or an alloy are usually not representative of the entire piece. These data represent the characteristics of the metal only at the particular section of the piece. The general distribution and variation in size of nonmetallic inclusions; the uniformity of structure; the location and extent of segregation; the presence of fabricating defects, such as seams or hammer bursts; and residual ingot defects, such as pipe can not be examined by microscopy.

The nature of inhomogenities in a metal, and the extent to which they exist therein, are best determined by macroetching a representative piece and subsequently examining the conditions thereby revealed with the unaided eye or with the aid of a low-powered microscope or magnifying glass. Such an examination is referred to as a macroscopic, or macro, examination. The magnification employed is usually not over 10x.

Macroetching sections may reveal conditions in the metal that are related to one or more of the following heterogeneous circumstances:

1. Crystalline heterogeneity, the presence and extent of which depend upon the manner of solidification and the crystalline growth of the metal or alloy.
2. Chemical heterogeneity, owing to impurities in the metal or alloy and to localized segregation of certain chemical constituents. Such segregation may be intentional (the introduction of carbon into the surface of steel during the process of case carburizing), or may be unintentional and undesirable, as for example, the segregation of sulphur or phosphorus that is so often found in cast steels.
3. Mechanical heterogeneity, arising from cold-working or process that introduces permanent stresses into the metal. Such heterogeneity seldom occurs in cast metals, but its presence is of importance in cold-rolled metals, forging, etc.

Experiment

Three experiments will be performed:

1. Sulphur Printing:

Impurities may exist in steel products. It is desirable that their amounts should be minimal and homogeneously distributed within the product. These impurities degrade the mechanical properties of the steel, especially sulphur content in steel makes it brittle.

Sulphur may exist chemically in steel in one of two forms, either as manganese sulphide or as iron sulphide. Sulphur printing detects and permanently records the distribution of sulphur in steel.

The surface of interest to be tested for distribution of sulphur should be reasonably smooth and free from foreign matter such as dirt and grease. Grinding the surface on papers, numbers 400 to 1200 and subsequent thorough washing will generally produce a surface satisfactory for the purpose.

Photographic bromide paper is soaked in a 2% aqueous solution of sulphuric acid for approximately 3 or 4 min. The paper is removed from the acid solution and allowed to drain free from excess solution. The emulsion side of the paper is then placed in direct contact with the prepared specimen surface and allowed to remain in contact under moderately applied pressure for 1 or 2 min. Care must be taken that all entrapped air bubbles between the paper and the specimen surface are eliminated.
The reaction of the sulphuric acid with the sulphide regions of the steel produces hydrogen sulphide gas, which reacts with the silver bromide in the paper emulsion, forming a characteristic brown to gray-black deposit of silver sulphide. These reactions may be expressed as follows:

$$FeS + H2SO4 \sim FeS04 + H2S$$
or
$$MnS + H2SO4 \sim MnS04 + H2S$$
$$H2S + 2\ AgBr \sim Ag2S + 2\ HBr$$

When the reaction has proceeded for approximately the recommended length of time, the photographic paper is removed from the surface of the specimen, rinsed in clear running water, and then fixed permanently by placing it in a photographic fixing solution for about 15 min. When fixation is completed, the print is again washed in running water for approximately 30 min. and subsequently dried in the usual manner.
The examination of properly prepared sulphur print will disclose quite clearly, because of the presence of darkly colored areas of silver sulphide, the precise location of sulphur inclusions on the prepared surface of the metal. A grouping or gathering of such silver sulphide areas indicates the presence of sulphur segregation, whereas a random dispersion of the spots denotes a more uniform, distribution of the sulphur inclusions.

2. Flow lines:

It is sometimes desirable to determine whether or not a finished piece has been forged, cut, or cast into shape and to note, in the event that it was forged, the direction of metal flow. Flow lines as revealed by macroetching in forgings are a natural consequence of applied mechanical working. If the flow pattern shows highly selective etching characteristics, it is likely that the material may be defective and may contain an excessive amount of inclusions and segregated areas.
A macroetched forged-material shows a directional flow pattern consisting of streaks and striations. The orientation of this pattern with respect to the plane of the prepared surface indicates the direction of metal flow during deformation. The flow lines are made visible because the elongated inclusions of impurities, such as oxides, sulphides and other elongated heterogeneous areas are selectively attacked by the etching reagent.

3. Welded sections:

It is often desirable, and frequently necessary, to determine the soundness of a welded joint and to observe macroscopically over a cross section of the weldment the various zones wherein structural changes have occurred. This is accomplished by macroetching the prepared surface with the usual hydrocWoric acid solution, or in some cases with a milder reagent such as 2 or 3 % nital (ethyl alcohol + 2% HN03).
In ferritic welds, the specimen of interest is prepared in a manner described for metallographic specimens and finally alternately polished and etched in saturated picral to remove disturbed

metal. The prepared surface is then etched for 10 to 20 sec. in 5% nital, after which the surface is thoroughly washed and lightly rubbed on a metallographic polishing cloth until the columnar grains in the weld metal show distinctly. This procedure is repeated several times to lessen the light reflectivity characteristics of the surface and to produce some relief of the macrostructure. The specimen of interest is then etched by immersion for about 2 min. in saturated picral, followed by thorough washing in cold running water and swabbing with a tuft of cotton to remove the loosely adhering reaction products formed on the surface. The specimen is finally rinsed in alcohol and dried in a stream of warm air.

Specimens

1. Sulphur printing: a piece of rail material
2. Flow lines: done without a specimen, shown on blackboard
3.Welded sections: finding the location of weld section of a welded-material

Procedure

1. Sulphur printing: follow the instructions in the introduction section.
2. Flow lines:
3. Welded section: follow the instructions in the introduction section (for grinding the specimen use number 240 to 400 grinding papers then rub the specimen surface using a cotton tuft by 9g $FeCl_3$ + 6 cm3 HCl + 100 cm3 H2O solution until the weld section appears).

Results

1. Draw schematically sulphur inclusions in the rail material (Specimen 1).
2. Are the sulphur inclusions distributed homegeneously or as segregated points in the material? Discuss their effects on the mechanical properties of the material.

3 What causes the flow lines to appear. Do flow lines exist in casted materials? Why?
4. What do the flow lines indicate in terms of mechanical properties of the material?
5. Draw schematically welded-section of specimen 3.
6. What do you find out by examining the welded-sections? Can you talk about the weld quality, appearance and the welded materials, etc.? By inspecting the weld section, relate this result to the mechanical properties of the material.

References

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3. Imperial College of Science and Technology, Department of Metallurgy and Materials Science, 2nd Year Materials Laboratory Notes for Mechanical and Aeronautical Engineers.
4. ASTM Standards and ASM Standards (they exist in our library, please consult with the librarian).