University of Denver
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Experiment #3 - Metallography

Introduction

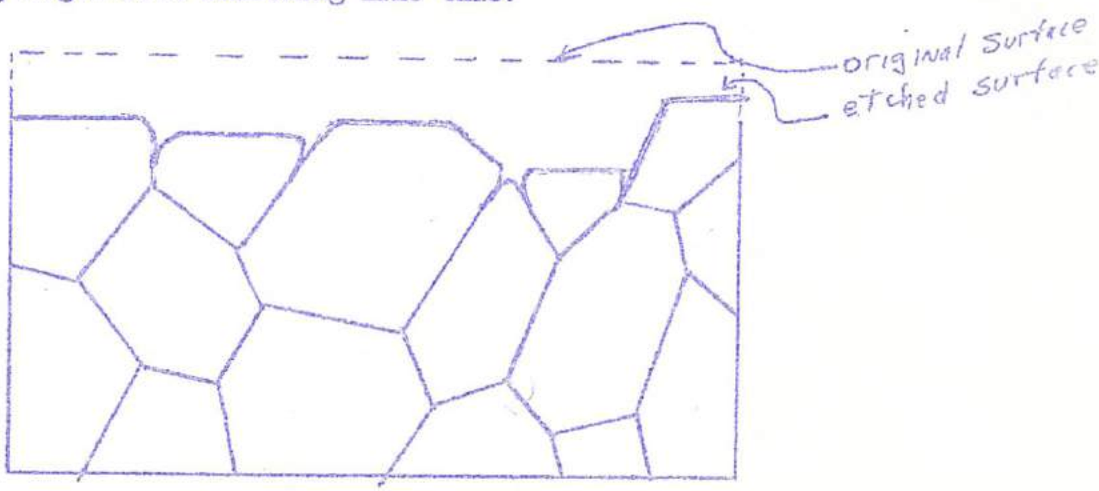
Metals have characteristic structures. If the structure is visible to the naked eye or under very low magnification, it is called a macrostructure. Structures observed under the metallurgical microscope are called microstructures.

As a metal cools from the liquid state, it begins to solidify (crystallize) at many points, forming minute crystals. These crystals then grow into the liquid, very often growing arms or projections into the liquid; these projections are called dendrites. Each of the minute crystals forms independent of the others, thus each has its own crystal orientation. The crystals then grow until their boundaries impinge on neighboring crystals. The tiny crystals are grains; their boundaries, grain boundaries.

Fig. I

Let us consider what happens when we etch the structure with a corroding solution. The grain boundaries tend to be preferentially attacked, as they are high energy areas. The grains are of random orientations; some of these orientations are more susceptible to chemical attack than others, thus some of the grains will be etched more than others. Our etched surface, in cross-section, might look something like this:

Fig. II



The etchant delineates the microstructure, which can be observed under suitable magnification in the metallurgical microscope. The opacity of metals dictates the use of incident reflected light. The light reflected and scattered from a grain boundary will differ from that reflected and scattered from the grains themselves; also the grains will scatter differently, depending on their orientation and etching properties. Thus the structure is easily and clearly observable.

In order to produce a good image, the surface must be perfectly flat and smooth--it should appear as a mirror surface before etching. The techniques of preparing a specimen for metallographic analysis are a bit tedious and require practice. Metallography is often more of an art than a science.

Techniques for the preparation of samples for both macro- and microscopic analysis are described in the Appendix.

The microstructures of metals are numerous and varied. The rate of cooling varies with the size and shape of the grains. In alloy systems often two different types of crystal structure form during solidification, thus two separate phases can be distinguished. They can appear in many ways, depending on the thermal treatments and compositions of the particular alloys. The two phases can be uniformly distributed as a eutectic structure; one phase may be surrounded by a uniform matrix of the second phase (hyper- or hypo-eutectic); the grains can be distorted or modified due to cold-working or thermal treatments (quenching); twins, slip lines, and other typical structures are all easily seen. Many of these structures will be observed in the lab. Specimens of future experiments may also be metallographically examined by you later in the quarter.

Procedure, Results, Questions:

1. Six metallurgical microscopes will be set up and 12 polished, etched, and labeled specimens will be provided. Examine all 12 specimens and sketch what you observe. Try your hand at polishing and etching some of the other samples provided.

2. Observe and sketch the macrostructures provided.

3. Identify the basic structure types and try to deduce the type of alloy and treatments involved. Identify and explain the different types of grains found in the macroetched samples--note any pipe, voids, etc. which you observed.

4. Which of the samples most correspond to the "expected" structures of last week's phase diagram samples?

5. What determines the resolution limit of the optical microscope? Can you optimize the resolution in any way?

6. What is polarized light? What are some of its advantages for metallurgical microscopy?

7. Explain dark-field illumination. Does it have any advantages over bright-field? Can it be used with polarized light?

8. Be sure you know how a metallurgical microscope functions, and how and why metal structures etch.

9. What is wrong with Fig. I on page 1? Why?

References

1. W. D. Biggs, "Elements of Metallography," Chapter 11 in Physical Metallurgy ed. R. W. Cahn, Wiley, 1965, pp. 541-64.

2. W. G. Moffatt, et al., Structure, Vol. I. of Structure and Properties of Materials, Wiley, 1964, Chapter 6: "The Shapes and Distributions of Phases in Solids."

3. R. C. Hendrickson and B. N. Iannone, Applications of Metallurgical Microscopy, a short booklet published by the Bausch and Lomb Co. containing many excellent micrographs.

4. Photomicrography of Metals, Kodak Industrial Data Book, 1959.

5. Kehl, Principles of Metallographic Laboratory Practice, 1949.

Appendix: Metallographic Sample Preparation, by R. W. Krenzer

Macroscopic Examination

Macroscopic examination refers to the investigation of large areas of metals and alloys by means of direct observation or with the aid of a low-powered microscope (up to about 10X). This type of examination is widely used for quality control purposes to detect defective material. Macroscopic examination reveals such data as location and extent of segregation, detection of fabricating defects, residual ingot defects, and flow lines in forged materials.

The nature of inhomogeneities in metals and the extent to which they exist are best determined by macro-etching the surface and examining the specimen with the unaided eye or a low-powered microscope. The macro-etched sections may reveal conditions in the metal that are related to one or more of the following conditions:

1. Crystalline heterogeneity - which depends on the manner of solidification and crystalline growth of the metal.

2. Chemical heterogeneity - which depends on segregation of constituents of the alloy.

3. Mechanical heterogeneity - which arises from any process which introduces permanent stresses into the metal or alloy.

When a suitable specimen has been obtained by such operations as shearing, cutting, grinding, or machining, the external surface of the metal may have flowed, thus concealing the internal structure. If the surface flow is extensive, it is generally necessary to remove the outer layer of the metal by first grinding through a series of coarse papers and finally using a 600 grit paper.

Prior to etching the sample, any grease or oil on the surface should be removed. The specimen is then placed in a container of water heated to the desired etching temperature, and maintained in the water bath until the entire specimen has reached the same temperature throughout. The preheated specimen is then transferred to a vessel containing the etching reagent, and completely submerged for the required length of time.

The specimen is then removed and thoroughly rinsed with water, alcohol and dried.

Some of the structures revealed by macroscopic examination include the following:

1. Dendritic Structures:

The grain structure of most cast metals consists of relatively large, elongated crystals called dendrites, which result from crystallization characteristics of the metal. The presence of a dendritic structure usually indicates the existence of chemical inhomogeneities, and represents a relatively weak and brittle pattern when compared with fine, equiaxed grains of most metals that have been subjected to mechanical working and heat treatment.

2. Columnar Structure:

In many cast metals, a columnar structure can be identified at the edges of the section, resulting from a slow rate of solidification. During solidification, the columnar grains grow normal to the walls of the mold at approximately the same rate until further growth is obstructed by contact of other similar grains growing from opposite walls. The planes between the grains are potential sources of weakness and the columnar structure is thus undesirable.

3. Piping:

As a metal solidifies, considerable contraction takes place, resulting in a shrinkage cavity, or pipe. The presence of pipe will lower considerably the mechanical properties of a metal, and upon mechanical working of the casting, the pipe will become a flaw in the metal.

4. Blowholes:

Blowholes are voids left in the structure as a result of entrapment of gases by the metal during solidification.

5. Internal Cracks:

Internal cracks, also called flakes, hairline cracks, or cooling cracks, result from unfavorable cooling conditions after hot-working. These cracks are discontinuities in the metal and are sources of failure when the piece is subjected to more mechanical working or heat treatment.

6. Porosity:

Porosity refers to the presence of small voids in a metal and can result from insufficient supply of molten metal during solidification or to the evolution of gases. If the voids result from shrinkage, they are termed as pipes, or if they result from gas evolution, they are termed as blowholes.

7. Inclusions:

Large nonmetallic inclusions, such as slag or molding sand are clearly visible by macroscopic examination due to differences in color and luster between the inclusion and the metallic matrix.

8. Flow lines:

Flow lines are a normal effect of rolled or forged materials, and in wrought metals, inclusions and segregated areas are stretched out in the direction of flow of the metal. After macroetching, flow lines appear as thin lines which indicate the direction in which the metal has flowed during the forming operation.

Etching Procedures for Macroetching

1. Steel

Use a 50% HCl, 50% H_2O by volume at 160°F. Etch until structure is revealed, usually in 20 to 45 minutes.

2. Aluminum

Use 10 gms. NaOH in 90 ml. H_2O. Etch by immersion for 5 to 15 minutes at 140 to 160F. Wash in water, dip in concentrated HNO_3 to remove surface products, and rinse again in water and dry.

3. Copper and Copper Base Alloys

Use HNO_3 diluted with varying amounts of water. Etch by immersion at room temperature until structure is revealed.

Microscopic Examination of Metals

A sample for microscopic examination must have a plane surface free of scratches and the structure must be revealed and not altered during preparation. To accomplish this, the specimen must be carefully ground and polished and chemically etched to reveal the structure.

Grinding

After a specimen has been cut to the proper size and mounted in a specimen holder, the next operation is to prepare a smooth surface to be examined by grinding the specimen down through a series of different grit papers (normally 240, 400, 600).

The edges of the specimen mount should be beveled to prevent seizing during subsequent grinding and polishing operations. When the surface has been uniformly flattened on one paper, the specimen is then ground on the next finer grit paper. The sample is rotated 90° on changing papers so that one can easily tell when the scratches from the coarser preceding paper have been replaced by a finer set of scratches. This procedure is repeated through successively finer grinding papers until the 600 grit paper is reached. The specimen should be carefully rinsed between each paper to prevent contamination of the finer papers with grit from the coarser papers.

Polishing

Polishing is carried out on polishing laps. The wheel is covered with a suitable cloth such as broadcloth, billiard cloth, etc., and is impregnated with a polishing media such as alumina, magnesia, rouge, diamond paste, etc. The most common media is alumina because of its relatively low cost and good abrasive characteristics.

The specimen is polished on the wheel by firmly holding the specimen and revolving it around the wheel in the opposite direction of the wheel's rotation. Keep the wheel well lubricated with a suspension of alumina to avoid seizure, which will tear the cloth.

The first polishing operation uses a 5 micron cloth with a fairly coarse alumina (No. 1). After removing all of the grinding scratches, rinse the specimen with water and dry in a warm blast of air. Proceed to the next wheel with a 1 micron cloth using a finer alumina (No. 3). The specimen can be observed under the microscope to determine if all of the polishing scratches are removed. During any stage of grinding or polishing it is important to remove all scratches from the previous operation before moving on to the next finer paper or cloth.

Etching

The polished surface should be plane and free from scratches. To the naked eye the surface should have a featureless mirror-like surface, and in the "as polished" condition, the surface will only show such characteristics as inclusions, voids, and major differences in phases. To prepare the surface for microscopic examination the specimen must be selectively etched to bring out the contrast between the phases present.

Iron and steel are generally etched in an oxidizing acid such as nitric (25 ml. conc. HNO_3, 75 ml. H_2O). There are also several other etching reagents used depending upon the desired results. (See ASM Handbook).

Hydrofluoric acid (0.5 ml. conc. HF, 99.5 ml. H_2O) is generally used for etching aluminum and its alloys. The acid is swabbed on the surface with soft cotton for 15 seconds and then rinsed clean. A complete list of aluminum etches is given in the "ASM Metals Handbook" and in "Principles of Metallographic Laboratory Practice" by Kehl.

The Metallographic Microscope

Since metal surfaces cannot be illuminated by transmitted light it is necessary to use vertical illumination to make possible direct examination of the fine grain structure and structural characteristics of a suitably prepared surface. The magnification can be varied in two ways by changing the eye-piece or the objective. A nominal magnification is obtained by multiplying together the magnification of the eye-piece and the objective.

The light is reflected onto the surface of the specimen by a series of mirrors and condensers. The metal surface reflects the light through the objective lens and eye-piece to the observer's eye. The amount of light reflected by different structural features of a metal will vary according to their orientation with the light source and composition.

The Metallograph

The metallograph is used to record what is observed through the microscope on film. This equipment consists of three main parts, (1) the light source, (2) an inverted microscope, and (3) the camera. The use of the metallograph will be demonstrated at the beginning of the laboratory period.