

SPECIMEN PREPARATION FOR OPTICAL MICROSCOPY

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INTRODUCTION

The most important single feature of metallographic technique is the preparation of specimens. This involves correct and representative sampling, cutting and grinding with a minimum of mechanical distortion, shaping the specimen or mounting the specimen, so that it can be conveniently handled during the subsequent operations removing the rough grinding marks and can be polished free from scratches and mechanical distortion, and finally etching the specimen to reveal and differentiate the various phases present in the metallic matrix. Even though it appears very simple, but in reality their technical art needs great care, patient and continued practice.

Though quality of any photomicrograph is a direct function of the quality of the micro specimen, unless the specimen is correctly prepared, the possession of the finest photomicrographic camera and the use of the most elaborate microscopic and photographic techniques will not produce good results. The specimen must first of all be correctly polished and then correctly etched. Incorrect and careless etching can completely ruin a good polishing technique. The whole complex of metallographic procedure is technical and human art-the results achieved at one stage of the operations depend entirely on the results obtained in the previous operations.

The technique chosen by any operator will depend partly on his preference, and considerably upon the type of alloy which has to be examined. For alloys of widely differing mechanical properties, for instance, the alloys of lead and the alloys of iron, the procedures will of necessity be widely different. In other cases, for instance, for steel and for cast irons, the procedures will be closely similar. The etching reagents used depend mainly upon the nature of the metal or alloy and what features of microstructure it is desired to reveal.

Sampling

The sampling for microscopic and macroscopic examination usually comes to the metallographist in the form of a casting, a forging or a test bar. It may have a complex shape or a simple shape and it may have a machined or cast surface. It may be scaled or it may have received some special surface treatment such as plating or enamelling. Finally it may have to be subjected to macroscopic or microscopic or to both. The nature of the sample and the reason for its examination will determine to some extent what type and what size of specimen shall be taken. If the sample has got some specific defect, the sample should be

cut in such a way that the defect is in the polished surface. In case of rolled and forged shapes, it may be necessary to take specimens both parallel and transversely to the flow lines. There are usually considerable differences in the structures of parts of a casting which have been subjected to different cooling rates. Sometimes, it may be necessary to have more than one specimen to get complete picture of the specimen. It is metallographer's intelligence and experience which help him to decide how and where to sample so that all the relevant features are recorded. When sufficiently big shapes are available, one can get specimen preferably not larger than 25 mm square and not smaller than about 10 mm in square; which can be easily handed for polishing without mounting. However, if the specimens are too small, then one should go for mounting.

For macro examination complete cross sections of forging and castings are frequently taken and one has to polish manually with great care and patience.

In cutting the metallographic specimen from the main body of the material, care must be taken to avoid affecting the structure of the metal. The specimens should be cut whenever possible by Power hack-saw, Metal cutting band-saw or Hand hack-saw with lubricant. In case of hard samples like cast iron, it has to be cut with abrasive wheel with cooling device. In no case it should be over heated, as it will alter the microstructure. In such cases, the specimen should be well ground after such cutting to remove altered microstructure. Flame cutting will completely alter the structure of the metal at the flame cut edge. This has to be remembered and small samples from the flame cut samples are obtained by recutting by some other means from places far away from flame cut edge.

Mounting Microspecimens

Frequently specimens are too small to be handled easily during polishing, or it is necessary to examine the extreme edge of the samples as in the cases of oxidation, carburisation, corrosion, plating etc. In such cases it is necessary to mount the specimen so that it can be conveniently handled or so that the edges are well protected. The mounting can be carried out in a variety of ways, but most popular methods are cold setting and thermoplastic material mounting under pressure. Both of these materials are available in all the standard laboratories, and can be obtained from Buehler, Struers or any other standard company.

Any specimen which is low melting or liable to undergo microstructural changes should be mounted in cold setting organic compounds. Even araldite is good for specimens which are fragile and porous like sponge iron or similar ones.

Hot mounting press usually utilises bakelite powder which melts and sets at a temp. of about 150-170°C under a pressure of 2000-5000 lbs/sq.in. The curing time is about 10 minutes and after this heating coil is switched off. The thermoplastics after setting is ejected out after sometime and we get good specimen surface mounted in the bakelite

mounting. This instrument is very common in all the metallographic laboratory. It is resistant to acid alcoholic etchant.

Grinding & Preliminary Mechanical Preparation

After the specimen has been cut to shape, or cut to shape and mounted, it is made plane by grinding on grinding wheel or preferably by motor driven emery belt of different ratings of 60 to 100 grits. Once this is done, it is taken to emery papers 1/0, 2/0, 3/0 and 4/0 or equivalent. The polishing should be cross each time at right angles so that previous scratches are removed. Rubbing down operation should be very gentle and methodical. Before changing of each grit, polishing paper, the sample is to be thoroughly washed to prevent carrying over of the previous grit particles and swart.

In case of soft materials like lead, zinc, etc. kerosene, paraffin lubricants may be used to get better results. Now-a-days a large number of specimens can be ground together in automatic machine. It all depends on individual requirements, and everybody has to develop one's own techniques.

The polishing Operation

After the intermediate grinding or rubbing down, the specimen is ready to be polished. This entails the complete removal of the scratches and the production of mirror finish surface. It should be remembered that even with the greatest care, the previous operations produce a certain amount of mechanically distorted metal on the surface of the specimen.

Polishing is done on a rotating wheel with sylvet cloth and alumina powder of 1 to 2 μ grades. These polishing machines can be found in any metallography laboratory. Speed of the wheel, amount of water and alumina powder are all determined by trial and experience. The specimen is polished till a mirror finish is obtained. Polishing produces a thin layer of amorphous metal on the surface of the specimen. This layer is variously referred to as the flowed layer which covers deep scratches, cavities, small nonmetallic particles and true structure of the specimen. This flowed layer can be completely removed by etching, when the true structure is revealed. If some scratches or distorted structures appear, repolishing and re-etching helps to give satisfactory microstructures. In short, this needs great patience and experience to obtain good microstructures. Whereas aluminium powder is used for ferrous and hard materials, silvo, brasso, liquid paraffin etc. are recommended for soft materials like, copper, brass, lead, zinc, samples. Polishing operation with diamond paste is also popular. Any standard hand-book can be consulted for particular specimen.

After polishing, the specimen should be washed with water, alcohol and dried so that staining does not take place. It should be observed on microscope about the suitability of its polishing surface, before it is processed further to etching to get good microstructures.

Electrolytic polishing and etching have also been used in some special cases. Since it does not come under day to day requirement, we are not discussing here.

For a metallographer, different types of structures are encountered. First and foremost will be cracks, porosity and inclusions. Then we come across cold worked and hot worked structures. Slowly cooled from liquid state of the metals and alloys will give different types of cast columnar grains whereas perfectly heat treated samples will give small and equiaxed grains. Stainless steel, brass will give twinning and so on. Therefore the next important step will be etching the polished specimen, which one can decide with experience. Macro and micro etching of the sample are needed according to requirement and few will be explained here.

Methods and Reagents for specific Metals & Alloys

Macro etching or micro etching is the process by which the flowed metal of the polished surfaces are dissolved by suitable reagents leaving behind the real structure, grain boundaries, inclusions etc. For different purpose, different reagents will be needed. Any good book on metallography or metals hand-book give the exact reagents. Some examples are given below.

1. Steels

- a) Macro etching is done by HCl diluted with 50% water. The sample should be immersed in boiling reagent for 10 to 15 minutes. It is good for flow lines in cold worked material, the structure of fusion welds, cracks, porosity and the depth of the hardened zone in tool steel. 25% HNO₃ in water can also be used cold for the same purpose on large surfaces. There are other reagents also for specific needs.
- b) Microetching : Nital i.e. 2 to 5% HNO₃ in ethyl alcohol is a general microetchant for steels which reveal pearlite, bainite, martensite, grain boundaries of the ferrite.

Picral (Picric acid 4g, alcohol 96 ml) is very good etchant for pearlite and spherodised structure for which it is superior to nital.

2. Reagents for Alloy Steel

- a) 2% Nital - good for austenitic Mn steel
- b) Acid ferric chloride

FeCl ₃	-	5 gm
HCl	-	50 ml
H ₂ O	-	100 ml (Distilled water)
- c) Chromic acid + HCl

HCl	-	25 ml
CrO ₃ (10% Soln. in water)	-	5 to 50 ml

suitable for heat treated 18-8 stainless steel

3. Aluminium & its Alloys

a) Macro reagent :

HCL	-	45 ml
HF	-	15 ml
HNO ₃	-	15 ml
H ₂ O	-	25 ml (Distilled water)

A general purpose reagent used at room temperature

b) Micro reagent :

- i) 0.5% HF in water is good reagent for aluminium alloys
- ii) 1 gm NaOH in 99 ml water is also good reagent for aluminium and its alloys.

iii) For duralumin type of alloys, the following may be used

HF	-	1.0 ml
HCl	-	1.5 ml
HNO ₃	-	2.5 ml
H ₂ O	-	95 ml (Distilled water)

4. Copper and its Alloys

a) Macro reagent

FeCl ₃	-	25 gm
HCl	-	25 ml
H ₂ O	-	100 ml (Distilled water)

For brass containing the β phase ammoniacal ammonium persulphate should be used.

NH ₄ OH	-	1 part
(NH ₄) ₂ S ₂ O ₈		
2% solution	-	2 parts
H ₂ O	-	1 part (Distilled water)

b) Micro reagent

- i) NH₄OH - 20 ml
(NH₄)₂S₂O₈ - 10 gm
H₂O - 80 ml (Distilled water)
- ii) (NH₄)₂S₂O₈ - 10 gm
H₂O - 90 ml (Distilled water)
Very good for brass, bronze, and nickel
- iii) FeCl₃ - 10 gm
HCl - 30 ml
H₂O - 120 ml (Distilled water)

For nickel rich alloys

5. Magnesium and its alloys

10% aqueous acetic acid, 2% oxalic acid in water on 4% aqueous picric, citric or formic acid are some of the useful reagents.

6. Tin & its alloys

FeCl ₃	-	10 gm
HCl	-	2 ml
H ₂ O	-	95 ml (Distilled water)

Suitable for tin rich babbitt metal.

It is beyond the scope of the lecture to enlist all the reagent. Only a few have been given. Metal hand book or Smithells hand book are recommended for consultation.

REFERENCES

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3. Metals Handbook, ASM.