

# Microscopy Simple or Advance Technique of Material Characterization

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## Abstract:

Optical microscopy with respect to advanced scientific research can be referred to as the basic techniques for the characterization of materials. Optical microscopy is expected to remain useful in the science world for a long time. Despite being presumed a basic tool, it is capable of giving very detailed information that reveals a lot about the materials preparations, heat treatment prior to observation, defects in the material as well as alloy composition. Relatively hard metals such as titanium, tungsten and steels are easy to prepare for microscopic examination unlike soft materials such as tin and aluminum that pose serious challenges during their preparation leading to defects such as deep scratch lines, embedded abrasive particles, dust particles, as well as water marks covering the polished surface. The material used in this study  $\text{Cu}_{50}\text{Ti}_{25}\text{Zr}_{15}\text{Ni}_{10}$  was produced by arc melting. The alloy ingots were re-melted at least 4 times for homogenization of the master alloy. The alloy ingot was then suction cast into a 3mm rod. The alloy metals used for the ingots had purities greater than 99.9% and were purchased from Zhongnuo Advanced Materials (Beijing) Technology Co. Ltd China. The objective of this work was to show the importance of proper and correct specimen preparation in revealing the basic and detailed information concerning the structure, as well as its morphology. A perfectly ground and polished surface is free from defect arising from preparation method hence the presumption that microscopy is a basic science does since even advanced structures can be revealed. It is imperative that a good quality optical microscope be purchased especially when resources are scarce and advanced materials characterization techniques are not available.

It can be concluded that with a good quality optical microscope, microscopy as a technique will continue to play a critical role during decision making and that a polished surface will only give accurate information about a given specimen if all preparation stages and steps are taken into consideration, adhered to and carefully and professionally done.

Keywords: Microscopy, arc melting, suction cast, polishing, amorphous

## Introduction

Optical microscopy is a materials characterization tool capable of revealing minute microstructural features and hidden details making it not only a basic technique but an advance technique for material characterization.

Microscopy has been used in many sectors of science and engineering in assessing thickness of coatings, effects of heat treatment on materials morphology, revealing effectiveness of a welding and brazing operation, extent of corrosion process, In analyzing failure analysis, forensic science [1] among many others. Even though initial stages of specimen preparation are more often taken as trivial, they are the most crucial stages if a representative, factual and conclusive observations are to be made. Microscopy may at time involve all or some of the following: sectioning, mounting, grinding, polishing, etching and finally microscopic observation. According to [2], before samples are mounted depending on the previous preprocessing undertaken, they should be ultrasonically cleaned, rinsed with ethyl alcohol, dried with compressed air and sprayed to remove any oil or debris introduced during sectioning [3]. The approximate time needed for each stage of grinding and polishing varies from person to person but generally 100-300rpm and 15-20seconds per grit used during grinding, 100-300rpm with 9, 3, 1, 0.3 microns and 5-10 minutes for each polishing time [4]. According to [4] fine polishing with 0.05 $\mu$ m diamond paste requires 1 min to several hours at 200-300rpm, 10-20 seconds at 100-150rpm with a lot of lubricant [5]. After each grinding and polishing step, sample and fixture are washed with micro-organic soap to remove debris and abrasive particulates, dried with compressed air spray to reduce the likelihood of scratches on the sample surface as a result of cross contamination [3] washed by swabbing with a liquid detergent solution, rinsed in warm running water, then with ethanol, and dried in a stream of warm air [6] or rinsed in mild soap and warm water or solvent and blow dried [5]. Fine polishing is followed by etching by either dropping etchant, immersing or swabbing the sample and immediately washing under running water, rinsing with alcohol, drying in an air blast and without touching, wiping or swabbing the specimen, after which microscopic examination done [3].

It has been observed that preparing soft materials like tin possess serious challenges more so with course grits. Course sized grits should be avoided as they cause deep cuts. When grinding soft materials, a thin layer of wax on SiC paper is applied as well as utilizing polish-etch-polish technique with 2% Nital or any other aggressive etchant between stages more so during grinding and polishing stage while applying light force with a more viscous lubricant [3], [4]

Water based lubricants should be avoided when dealing with soft materials since they favor deep deformation and pull-outs of hard inclusions from the material surface. It is therefore recommended that oil-based lubricant and small

volume of suspension be used [2]. Harder materials require greater force and a higher volume of diamond suspension to remove an adequate amount of material in the same time step [3].

It is worth noting that modern sample preparation methods have focused on reducing the number of SiC grinding steps and replacing SiC paper with other abrasives and surfaces with exceptions given where materials respond better to grinding with a series of finer and finer SiC papers [6]. During polishing stage new cloths should be used for each sample type to prevent cross contamination [3]. It has been observed that final condition of the polished surface is dependent upon many parameters grinding/polishing pressure, velocity or relative velocity distribution, direction of grinding/polishing, number of stations available, coolant and lubricants, polishing colloidal and the type of cloth used [3]. Experience has demonstrated that getting the required image quality to the microscope is by far the greatest challenge that many metallurgists face. Despite this, the specimen preparation process is often treated as a trivial exercise however; specimen preparation quality is the determining factor in the value of the examination [6].

Material preparation, should not introduce extraneous structures, disturbed metal, pitting, dragging out of inclusions and “comet tailing” [6]. The alkaline property of colloidal silica suspension makes it the preferred and ideal final polishing solution for most materials [3].

## Experimental procedure

The material used in this study  $\text{Cu}_{50}\text{Ti}_{25}\text{Zr}_{15}\text{Ni}_{10}$  was produced by arc melting. The alloy ingots were re-melted at least 4 times for homogenization of the master alloy. The alloy ingot was then suction cast into a 3mm rod. The alloy metals used for the ingots had purities greater than 99.9% and were purchased from Zhongnuo Advanced Materials (Beijing) Technology Co. Ltd China.

Bakelite was used as the molding resin at 130°C for 30 minutes and cooled for 10 minutes. Metallographic mounted samples were ground with a series of progressively finer SiC papers (220-2000). Polishing was done by ensuring that samples were turned 90° between two consecutive paper grits after all polishing lines faced the same direction SIC grit with fine surface was used. Final polishing stages was done with napped polishing cloth with a 3µm alumina colloidal produced by mixing aluminum powder and distilled water and chemo-mechanically polished in a 50 nm colloidal silica slurry. After polishing the specimens were swabbed with HCl: HNO<sub>3</sub> (1:3) etchant and finally the samples surfaces were then observed with an optical microscopy and scanning electron microscope.

## Results and discussion

### *a) Mounting and Polishing of the samples*

Figure 1(i) shows a sample mounted in bakelite by a 1-inch compression press. Immediately when the sample is withdrawn from the compression mounting machine very little can be revealed from the microscope. It is common for the surface to be given some initial grinding operation to remove bakelite engulfing the sample and to produce a flat surface. After grinding with course grits, the

sample, fixture and bakelite zones were very distinct. It was deduced that when a sample is properly mounted and correctly ground, the resulting surface is flat which reflect all the light and eliminates interference leading to accurate and correct results.

During manual polishing, it was observed that with well ground flat sample surfaces, about 15-20 seconds and speeds as low as 58rpm was enough to carry out polishing with each grit size. It is worth noting that specimen preparation for microscopy is majorly a factor dependent upon training and experience of the person carrying out polishing. When samples are well mounted, optical microscopes are capable of giving detailed primary information for decision making.

Figure 1(ii) (a-e) shows typical ground surfaces with grits ranging from 120 to 600 grits showing a reduction in the intensity and size of the resulting grit lines. The reductions of the size of the scratch lines are also accompanied with reduces in depth of penetration. It is common practice that as finer papers are used, pressure applied should also be reduced accordingly. Figure 1(ii) (f, g and h) shows the lines produced during the polishing stages with 6 $\mu$ m, 1 $\mu$ m, and colloidal silica. In these diagrams the line intensities are approaching defect free surfaces.

A well-polished surface should be devoid of extraneous structures, disturbed metal, pitting, dragging out of inclusions and “comet tailing” [6]. Water marks, deep cuts, lines arising from polishing stages. The alkaline property of colloidal silica suspension makes it the preferred and ideal final polishing solution for most materials [3].

During the actual grinding and polishing, it is advised that fewer grit sizes are used. But this will depend on the experience of the person doing grinding. For these samples not all the grit sizes were used but mostly 320, 600 1000 and 1500 were used before carrying out polishing operations

### *b) Microscopic observation of Polished surfaces*

Microscopes are useful during grinding, polishing and after etching treatment. With many microscopes existing Optical, microscopes (OM), scanning electron microscopes (SEM) , transmission electron microscope (TEM), the application of any type depends on the intricacies of details need. The more sensitive and the higher the magnification the more expensive the microscope is hence both SEM and TEM are only used if OM cannot be used.

During both grinding and polishing, an OM can be used to check the extent of grinding and if a change over from one grit size to another is necessary. It was the OM that showed the details in Figure 1(i) and (ii) and Figure 2 for etching stage to be carried out.

Despite well-polished surfaces being defect free, as polished surfaces due to reflection of light appears bright and reflective. No much information can be deduced from such a surface hence many materials after polishing and rinsing with de ionized water are given etching treatment to initiate contrasting colors between grains and/or grain boundaries. Mixture of chemicals solutions used for etching are referred to as etchants with each materials having its own suitable etchant(s) For the amorphous  $\text{Cu}_{50}\text{Ti}_{25}\text{Zr}_{15}\text{Ni}_{10}$  alloy, the etchant used was 1:3 HCl:HNO<sub>3</sub> ( Nital) solution. Figure 2 (b) grain growth start to take place but are not yet fully established, with further swabing a more pronounced dendritic grains are revealed Figure 2 (c). Both the primary dendrites and secondary dendrites are now evident. Swabing of most etchants are done for about 1-5

times, while for some the sample is dipped into the solution for 1-5 minutes. Time variation varies from material to material and experience of the person doing the etching process.

Figure 3 (a) shows optical micrograph of  $\text{Cu}_{50}\text{Ti}_{25}\text{Zr}_{15}\text{Ni}_{10}$  showing grains and their orientations after being polished and de-alloying in 8M  $\text{HNO}_3$  acid for 72 hours. De-alloying is done to dissolve some active components of the alloy. It can also be applied as an elimination method for amorphous materials having capacities of getting de-alloyed leaving behind vacant sites. It was deduced that de-alloying did contribute to the clarity of the grains with very clear grain orientations. It can be deduced that for these alloys capable of being deployed, the process can be used to help get from the alloy (b) SEM image of  $\text{Cu}_{50}\text{Ti}_{25}\text{Zr}_{15}\text{Ni}_{10}$  that was polished and deployed for 72 hours in 8M.  $\text{HNO}_3$ .

Scanning electron microscopes are used to reveal structures that cannot be resolved by an optical microscope. For  $\text{Cu}_{50}\text{Ti}_{25}\text{Zr}_{15}\text{Ni}_{10}$  alloy, the grain structure is seen as having mat like weave. Such like structural details can only be revealed by advanced material characterization method such as SEM. It is very difficult to reveal the structural details of a material using OM In conclusion, it can be stated that microscopes are a complex combination of machines capable of revealing macro, micro and nanostructure. They are in simple on the one side but complex on the other side hence microscopes are advance technique material characterization.

## Conclusion

The polished surface of the  $\text{Cu}_{50}\text{Ti}_{25}\text{Zr}_{15}\text{Ni}_{10}$  samples showed dendrite like structures resembling those common in crystalline materials. Optical microscope revealed microstructures that were dendritic in nature after etching with Nital. Microscopes are tools that reveal a lot about grains sizes, orientation, concentration and can be an indicator of the previous material treatment.

For micro and macrosized structures, Optical microscopes will do but if detailed features of about  $1\mu\text{m}$  and below then optical microscopes ceases to give any useful information hence need for more advance microscopes, SEM, high resolution Scanning electron microscope (HRSEM) and TEM will be needed.

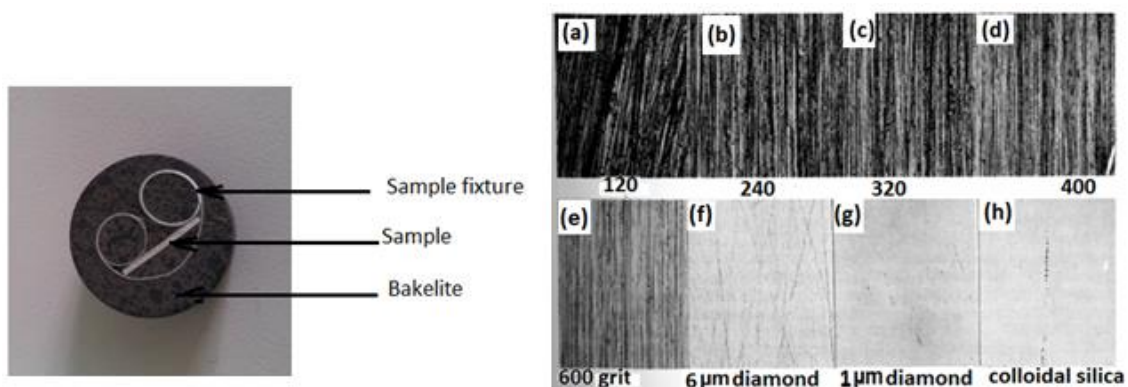


Figure 1: (i) Sample fixed in Bakelite

(ii) Diagrams showing surfaces ground with different sized emery papers (a,b,c and d) and then polished (f, g and h)

[Source: Buehler, 2004]

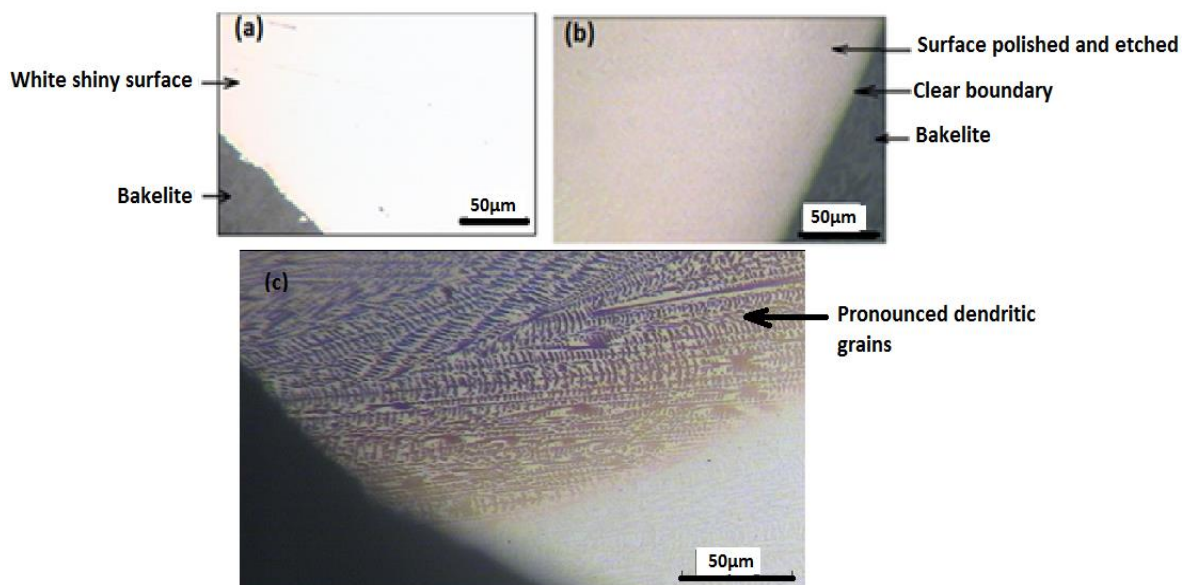


Figure 2; (a) Optical Micrograph of polished sample (b) Polished and etched in Nital ( 1:3 HCl:HNO<sub>3</sub>) (c ) Polished and etched in Nital with increased number of swabs( 1:3 HCl:HNO<sub>3</sub>)

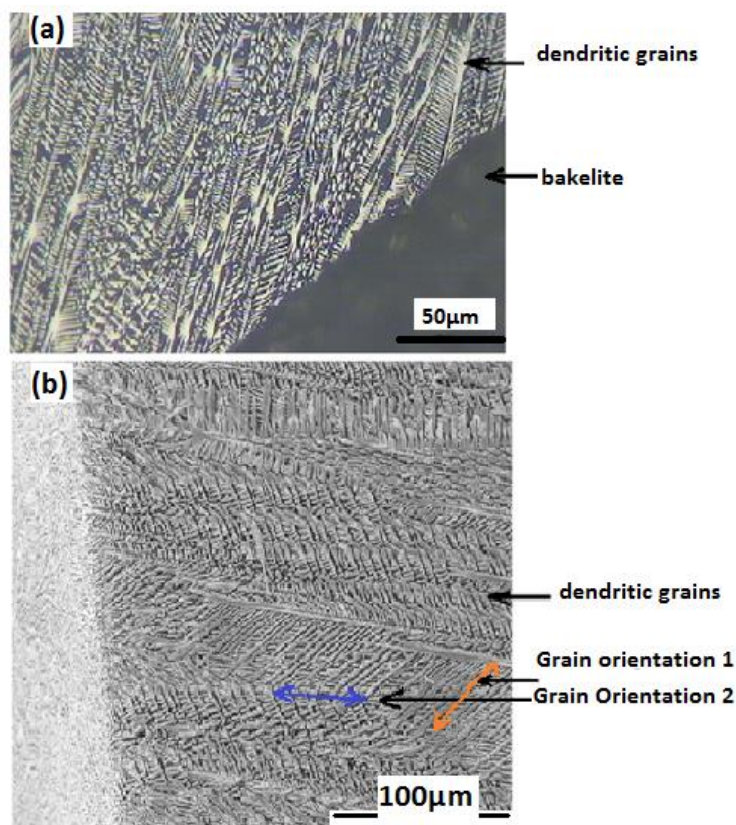


Figure 2: (a) Optical micrograph of  $\text{Cu}_{50}\text{Ti}_{25}\text{Zr}_{15}\text{Ni}_{10}$  showing grains and their orientations after polished and de-alloying in 8M  $\text{HNO}_3$  acid for 72 hours. (b) SEM of  $\text{Cu}_{50}\text{Ti}_{25}\text{Zr}_{15}\text{Ni}_{10}$  polished and de-alloying in 8M  $\text{HNO}_3$  acid for 72 hours.

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