Metallographic Sample Preparation

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Overview

High resolution pictures are paramount to determining void space in superconductive cable and angular dispersion in super alloy conduits. Precise methods in sample preparation are needed to produce the optimum surface for observation. Metallographic sample preparation requires a highly skilled hand to ensure that a sample is prepared in such a way as to gather useful information concerning the potential future use of the sample.

We had the opportunity to assist in preparing, and photographing, a sample of Niobium-Tin (Nb_3Sn) and super alloy conduits of Haynes 242. The process is long and involved, but crucial for obtaining usable images. Much information can be garnered from each sample, and goes a long way towards advancing the production of high powered magnets.

An Environmental Scanning Electron Microscope (ESEM), an instrument which uses a beam of highly energetic electrons to look at objects on a very fine scale, was used to observe samples once they had been prepared. The ESEM can yield information on topography, morphology, composition, and crystallographic information.

The inverted microscope, in which the light source and condenser are on the top above the stage pointing down and the objectives and turret are below the stage pointing up, was used to capture images of samples at 25X magnification.

Brooks and Tynica reassembling photographs of Nb_3Sn captured using a inverted microscope.

Process of Sample Preparation – Skills in Science

The process of preparing a metallographic sample is very intense and time consuming. The objectives are to reveal microstructual constituents and smooth it to a high luster. To achieve this, there are various forms of grinding, polishing and etching that takes place. The sample must be free of artifacts or damage that was introduced during prior preparation.

First, a portion must be cut from the original sample. This cutting process can take up to 45 minutes.

Next, we enclosed the sample with epoxy resin in a hot mounting press.

Then, we used Silicon Carbide paper (1000 grit) to grind the sample.

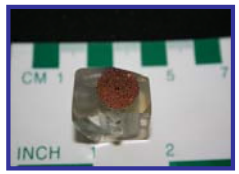
For polishing, we used two types of diamond solution - 9 microns and 3 microns, respectively. For the 3 microns, the sample must be turned parallel to the direction on the 9 microns.

We then look at the sample using an inverted microscope to detect any heavy scratches from grinding and polishing and to also take photographs.

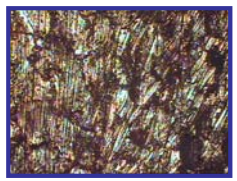
If the sample is free of scratches, it is then put into a vibratory polisher for its final stage in the sample preparation.

The effects of grinding and polishing

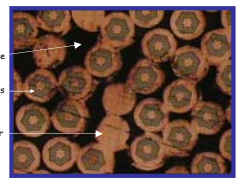
Not just anyone can prepare a sample. It takes great skill to achieve a functional sample. Below you see a sample of Nb_3Sn; the first image shows the sample as it looks after cutting, mounting, and polishing. The next two images are from pieces taken from the same sample. The image on the left was improperly prepared and photographed using the inverted microscope. The image on the right was carefully prepared and photographed using the inverted microscope. When you see these images side-by-side it is easy to see how important precision methods are to gaining useful data. The sample on the left is so full of scratches and flaws that it is impossible to even see the filaments within the cable. Once the sample is finished, scientist look to see how much void space is in between each wire. The void space is important because it detects how much liquid helium will flow through the wires to keep them cool. If it heats pass liquid helium temperature and enters a gaseous state, then the helium will not pass through the cable to cool it. Therefore, the void space must be adequate enough in size for liquid helium to pass through but no so large as to interfere with conductivity or potentially cause a quench.



This sample has been cut, ground, and polished.

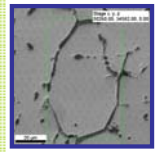


This is a sample that has been cut but has not been properly prepared.



This sample has been through proper preparation.

Angular Dispersion in a Polished Sample of Haynes 242



SEM image

This is an SEM image collected from a sample of Haynes 242, a super alloy that is used to create tubing for super conductive wiring, and shows an individual grain and the grain boundary. From this perspective it appears to be one solid grain.

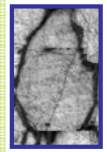
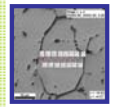


Image quality map

This image shows the same grain, but is reflective of the quality of the captured image. Those areas appearing brighter indicate a higher quality image. From this image we notice a long fracture running down through the grain, and we begin to see that there is more to this grain than originally thought.



Pointscan across suspected grain (left); Electron backscatter defraction pattern (right)

For the image on the right we identified individual coordinates through the center of the grain, which allowed us to obtain information, contained on the left hand image, about the angular dispersion within the grain itself. This made it possible for us to begin to view the grain as two individual grains.

Grain Map

The different colors shown on this image, of the same grain, now show us that there is, in fact, a grain boundary running through the grain.

Inverse Pole-figure map

Knowing that a grain boundary exists, it now becomes important to observe the angular divergence of the two grains. This image shows us, through the different colors, how the material has been stressed. We now know that each is a unique grain with a unique directional pattern.

Conclusion

What we were able to observe from our polished samples only scratches the surface of the potential data that can be collected from a properly prepared sample. The time and effort invested towards cutting, grinding, and polishing samples may seem exorbitant, but is an essential part of the research being done to improve the strength of high powered magnets. The materials used in high powered magnets will be subjected to extreme conditions and must be able to withstand the stress. The quality of the materials used will be dramatically improved through the careful efforts in metallographic sample preparation.

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