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Optical Materialography Part 1: Sample Preparation

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Overview

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The imaging of microscopic structures of solid materials, and the analysis of the structural components imaged, is known as materialography. Qualitative information such as, for example, whether or not there is porosity in the material, what the size and shape distribution of the grains look like, or whether there is anisotropy to the microstructure can be directly observed. We will see in Part 2 of the Materialography series, however, that statistical methods allow us to quantitatively measure these microstructural features and translate the analysis from a two-dimensional cross section to the three dimensional structure of a material sample.

This presentation will provide an overview of the techniques and procedures involved in preparing solid material samples for optical microscopy. While materialography can be conducted with optical as well as electron-based microscopy, this presentation will focus on the sample preparation specifically for optical microscopy. It should be noted, however, that a sample prepared for optical materialography can be used for scanning electron microscopy as well with minimum, if any, additional steps.

Principles

The most reliable way to obtain a sample section that is representative of its true structure is to follow a systematic sample preparation method. The preparation of a sample should be somewhat customized to its mechanical properties but the procedures outlined here should work well in general and particularly well for materials with similar hardness to that of steel.

Sample preparation for materialography via optical microscopy involves first mounting the three dimensional solid sample. Materialographic samples are usually mounted using thermosetting materials (here we will use bakelite, a phenolic thermosetting resin). Alternatively, epoxies can also be used.

Next, the sample is cut to expose a representative cross section surface of the sample. The cut is positioned on the sample so that it will define the cross section to be observed on the microscope. In samples where the microstructure is expected to be isotropic, the orientation of the cut can be arbitrary. However, in non isotropic samples, the orientation of the cutting vector, that will define the plane normal of the exposed cross section, should be set according to specific directions within or planes of the sample itself.

Coarse polishing, sometimes referred to as grinding, and subsequent finer polishing of the sample will reveal a representative cross section of the sample from which microstructural features can be observed and from which statistical analysis of this microstructure can be conducted. One of the principles followed during the polishing steps is that polishing success at each step can be measured by the complete removal of the scratches left on the sample from the previous step. Steps can be continued until the polishing agent is smaller than the resolution of the microscope. An etching step is usually necessary in order to expose the microstructure better. This step typically consists of dipping the polished face of the sample in a dilute acid for tens of seconds, before rinsing the etched surface clean. Since the grain boundaries have more atomic defects than the grains, they are etched by an acid solution at a faster rate than the grains are. As a result, slight recesses are created at the boundaries which enhance the contrast between the grains in a optical microscope.

The sample examined in this video is a Toroidal inductor core. Toroidal inductor cores are commonly used in electronic applications to regulate electromagnetic interference. These cores are economically manufactured by compacting and sintering iron powder. Compaction occurs along the axial direction and may deform the powder particles and grains, while increasing the overall density of the core. Porosity and grain size of the core material both impact the electromagnetic properties of the inductor, and also indicate the processing path used for an inductor core.

Quantitative information on the porosity and grain size in the three-dimensional material can be obtained by applying stereological techniques, which involves statistical analyses of the observed features in the optical microscope images, of carefully chosen two-dimensional sections.

Procedure

1. First cut the inductor core sample normal to the hoop plane using a linear precision saw in order to isolate undamaged microstructural features for later viewing.
2. Mount the specimen with the side to be imaged facing down on a mounting press. You must, of course, make sure that the sample is small enough to fit in the die cavity of the press, both laterally and vertically.
3. Fill the remaining volume of the mounting press cavity with Bakelite, a thermosetting resin.
4. Press the sample and using the prescribed heat, pressure and duration prescribed for Bakelite or other thermosetting mounting materials you may be using.
5. Over the next steps, polish using sequentially finer polishing media, starting with a coarse grit paper (e.g. 600 and 1200 grit paper, where the abrasive particles are 25.8 and 15.3 μm, respectively) followed by finer polishing suspensions (e.g. 1 and 0.01 μm alumina particles). These coarse-to-fine polishing steps should be done on rotating polishing wheels.
6. Between each step, the sample should be rotated by 90^0 in order to obtain a directionally uniform polishing and such that the scratches from the prior step can be distinguished from the current one.

7. The sample should be checked on an optical microscope that the scratches on the sample surface left by each step in the schedule will be removed by each subsequent step (**Figure 1a-d**), with the goal of leaving no observable scratches at the highest magnification of the optical microscope after the final polishing step **Figure 1d**. Table 1 indicates an example of a polishing schedule that can be used.
8. In the final sample preparation step, prepare a 2% nital solution (2% by volume concentrated nitric acid in ethanol), and dip the polished face in the solution for about 20 seconds. Rinse the sample with ethanol before observing the effects of the etch on a microscope. The etching step is repeated until it is judged that there is sufficient contrast in the granular structure observed. The etching time (and the number of iterations), therefore, is somewhat flexible and depends on the subjective judgment of the materialographist.
   NOTE: Care should be taken during abrasion to keep constant pressure and direction of wheel motion constant. When grit sized is changed, the sample is also rotated in order to distinguish the newly introduced scratches from those that were introduced in the previous step.

Step	Media	Grit	Time (min)	Speed (rpm)	Comments
2	SiC	600	2 min*	120	Rotate 90° before step 3
3	SiC	1200	2 min*	120	Rotate 90° before step 4
5	Al_2O_3	0.3 #m	2 min*	120	Rotate 90° before step 6
6	Al_2O_3	0.01 #m	2 min*	120	* or until scratches from previous step are removed

Results

From the series of images in **Figure 1**, particularly from the etched sample (**Figure 1e**), one can observe that the powder pressing process by which this sample was made rendered the grains to have non-circular, elongated shapes, with non-isotropic grain orientation. There is a significant amount of porosity retained in the material through this processing. Part 2 of the Materialography series will explore the statistics of the grain anisotropy as well the porosity.

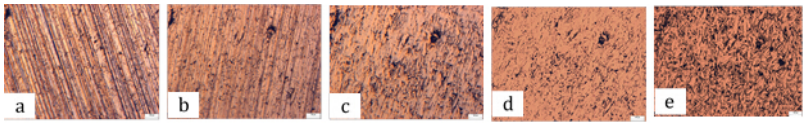

Figure 1: Polishing the sample with a) 600 grit, b) 1200 grit, polishing papers followed by c) 1 µm, d) 0.01 µm alumina suspensions on polishing cloths. Finally, e) etching for 20 seconds a nital solution revealed the porosity.

Applications and Summary

These are the standard methods for preparing cross sections of samples for microscopy. While the procedures detailed here are optimized to provide the best results in optical microscopy, some of the steps are unnecessary for scanning electron microscopy, and are inadequate for transmission electron microscopy. For the latter two, separate sample preparation procedures should be followed.

Materialographic sample preparation described here are the necessary first step towards the analysis of internal microstructure of three-dimensional materials using two dimensional information. For example, one might be interested in knowing how porous a membrane material is since that will affect its gas pearmenability. An analysis of the void structure of the 2D cross section will provide a strong indication of what the porosity is in the actual 3D structure (provided the sampling statistics are high). Another application would be in analyzing, for example the orientation of the polycrystalline grains in oil pipeline alloys. The orientational distribution function (ODF) can be directly related to the axial and transverse mechanical strength of the pipes, and so our sample preparation procedure is an important component of such an analysis.