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# Optical Materialography Part 2: Image Analysis

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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. Often, we would like to quantify the internal three-dimensional microstructure of a material using only the structural features evidenced by an exposed two-dimensional surface. While X-ray based tomographical methods can reveal buried microstructure (for example the CT scans we are familiar with in a medical context), access to these techniques is quite limited due to the cost of the associated instrumentation. Optical microscope based materialography provides a much more accessible and routine alternative to X-ray tomography.

In Part 1 of the Materialography series, we covered the basic principals behind sample preparation. In Part 2, we will go over the principals behind image analysis, including the statistical methods that allow us to quantitatively measure microstructural features and translate information from a two-dimensional cross section to the three-dimensional structure of a material sample.

## Principles

Morphological information from the internal three-dimensional structure of a material can be obtained by applying materialographical techniques, that is, techniques that do a statistical analysis of carefully chosen two-dimensional sections, to optical microscope images.

The porosity in a material, which is the fraction of the volume of a material that is open space (not occupied by atoms), can determine its mechanical, electrical, optical properties and directly affects mass transport through it (its permeability). Porosity as a volume fraction can be shown to be statistically equivalent to the area fraction or point fraction of voids in a representative two-dimensional slice:

$$f = \langle A_A \rangle \quad [1]$$

$$f = \langle P_p \rangle \quad [2]$$

where  $A_A$  is the area of void area, normalized by the total imaged area, and  $P_p$  is, likewise, the number of points lying in void divided by the total probe points. The brackets indicate an average over multiple samples.

The average grain size, the average lateral dimension of a crystal grain in a polycrystalline material, can be quantified by measuring the mean intercept grain size,  $G$ , which can be determined by overlaying test lines on the microstructural image:

$$G = \frac{1}{\langle I_L \rangle} \quad [3]$$

where  $I_L$  is the number of intersections between the test lines (see **Figure 2**) and the grain boundaries per unit test line length. For high porosity materials,  $G$  can be found by:

$$G = \frac{1 - \langle P \rangle}{\langle I_L \rangle} \quad [4]$$

Finally, the effective density of a material can be calculated by taking the porosity, measured by the materialographic techniques, into consideration. This Effective Density, takes into account the volume of the pores in a material, whereas the 'density' may refer only to the non-porous region (depending on the measurement method). This effective density of the material can be found using:

$$EffectiveDensity = (1 - Porosity) \times Density \quad [5]$$

where Porosity can be obtained by  $\langle P_p \rangle$  or  $\langle A_A \rangle$ .

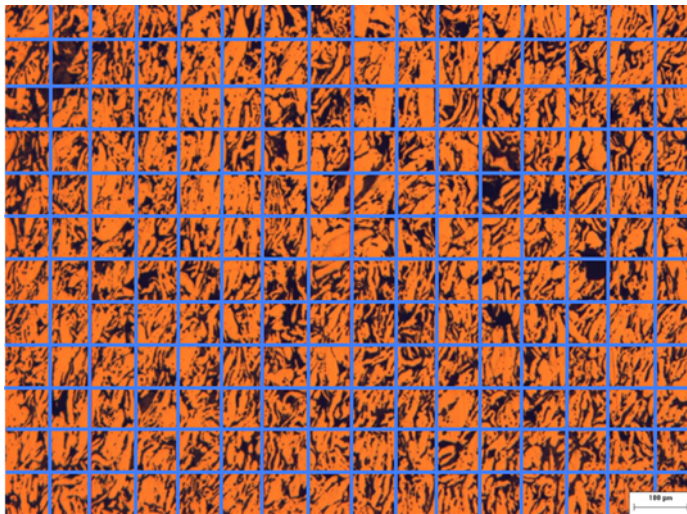
## Procedure

1. Complete all the procedures from Materialography Part 1. It should be reminded that the reproducibility of the following can only be assessed by analyzing multiple images from the same sample.
2. If digital analytical software is available, where the pixels can be categorized based on their brightness and counted accordingly, then it is possible to use Equation [1] to estimate pore volume the based on  $\langle A_A \rangle$ . Otherwise, this analysis can, of course, be done by hand.
3. Now estimate pore volume using  $\langle P_p \rangle$ .
4. Overlay a grid on the microstructural image. The intersection points of the lines on the grid are to be used as test points for the next step. There are 165 points shown in a representative result (**Figure 1**).
5. Count the total number test points and the number of test points contained in the porosity area (dark regions in **Figure 1**).
6. Calculate the fraction of test points falling on the porosity area for each image.

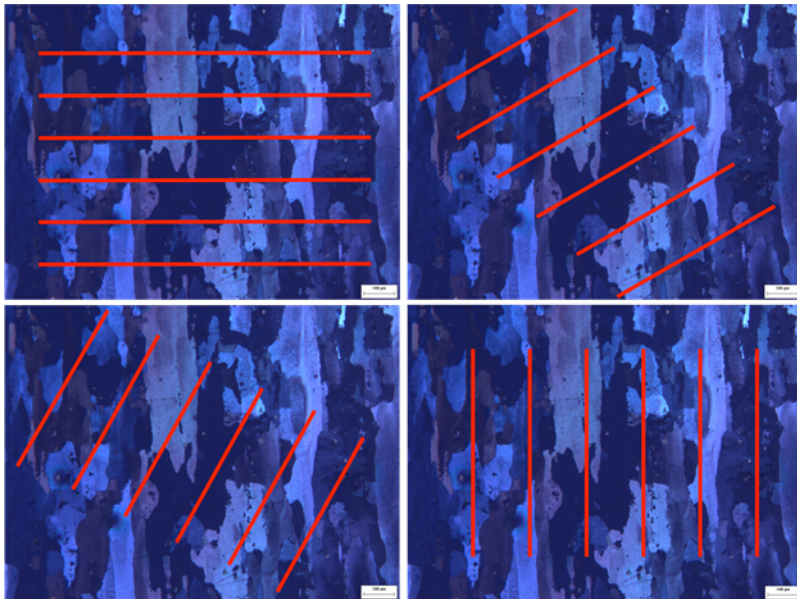
7. Determine the average value of this point fraction  $\langle P_p \rangle$ , which is the volume fraction of porosity in the sample.
8. Measure grain size by superimposing a set of test lines on the microstructural image and counting the number of intersections between the test lines and the grain boundaries (the boundaries between neighboring grains).
9. Straight lines at 0, 30, 60, and 90 degrees with respect to the horizontal direction straight lines are used (**Figure 2 a-d**) in order to get seek out any potential anisotropy (preferred orientation) to the shape of the grains.
10. Note the number of intersections between the test lines and the grain boundaries per unit test line length. Repeat the procedure with the test lines parallel to the vertical axis.
11. Calculate the mean intercept grain size  $G$  in both cases and compare the values.

## Results

In **Figure 1** we see a cross section of a porous material with a grid superimposed on it. The points of intersection can be used to determine  $\langle P_p \rangle$ . The number of intersection points that lie over dark regions (pores) is divided by the total number of intersection points to get  $P_p$  and by collecting and, by averaging  $P_p$  values from multiple images, we arrive at  $\langle P_p \rangle$ .



**Figure 1:** Materialographic image with a superimposed grid. The intersection points on the grid are used for analysis.



**Figure 2:** Grains size measurements using lines at a) 0, b) 30, c) 60 and d) 90 degrees orientation. The grains are obviously anisotropic in shape (longer in one direction than the other). This anisotropy results from the nonuniform forces acting on the samples during processing through which the grains become "squashed".

Image ID	Test points in porosity area	Total no. of test points	P <sub>p</sub>	<P <sub>p</sub> >	
				Avg.	Δ*
P1	32	100	0.32	29	1.77
P2	29	100	0.29		
P3	22	100	0.22		
P4	37	100	0.37		
P5	24	100	0.24		
P6	30	100	0.30		

Table 1. Porosity measurements.

ID	Probe L(mm)		Horizontal (Radial or Hoop)					Vertical (Axial)				
			I	I <sub>L</sub>	<I <sub>L</sub> >		G	I	I <sub>L</sub>	<I <sub>L</sub> >		G
					Avg.	Δ				Avg.	Δ*	
SL1	0.9		16	17.7	18.1	0.68	0.05mm	3	3.33	3.7	0.31	0.27 mm
SL2	0.9		14	15.5				2	2.22			
SL3	0.9		18	20				4	4.44			
SL4	0.9		16	17.7				3	3.33			
SL5	0.9		15	16.7				5	5.56			
SL6	0.9	19	21.1					3	3.33			

Table 2. Intercept measurements using straight line probes.

\*: Δ is the sampling error. Assuming a confidence level of 95%, the sampling error can be estimated with the equation below:

$$\Delta = 1.96 \frac{\hat{\sigma}}{N} = 1.96 \frac{1}{N} \sqrt{\frac{\sum (x_i - \mu)^2}{N-1}}$$

**N:** number of samples

**x<sub>i</sub>:** the *i*th sample

**μ:** sample average

The probability of population average lying in the range [μ- Δ, μ+ Δ] is 95%. The sampling error can be used as criteria in telling if the difference between two averages is significant (e.g. the difference between the average of I<sub>L</sub> estimated with vertical line probes and horizontal line probes).

## Applications and Summary

These are standard methods for analyzing two-dimensional cross sections in materials in order to extract three-dimensional information. We looked specifically at estimating the volume fraction of pores in one material and the average grain size in a second material.

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 permeability. 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.