The Importance of Sample Preparation in the Analysis of Powder Metallurgy (PM) Materials

Thomas F. Murphy

Hoeganaes Corporation, Cinnaminson, NJ 08077

Analyzing all materials is dependent on developing and following metallographic sample preparation procedures tailored to fit each individual combination of physical, chemical, etc., attributes. This is particularly true where variations are evident on a localized basis. Metal powders and the products made from them are excellent examples of these situations, where special attention must be paid to the materials and the unique features within the microstructure to ensure accurate results. Several cases illustrating these difficulties will be presented.

One characteristic of powders and consolidated components is the large amount of surface area both around and within the samples. When considering the examination of loose powder samples, the surface area and the size of the individual powder particles create a special set of difficulties to creating properly prepared planar surfaces. It is vitally important when examining their cross-sections to choose a mounting technique that will securely retain the particles and ensure a proper representation of the material is available for inspection. Figure 1, a and b, demonstrates a difference in mounting materials. In Fig. 1a, a diallyl phthalate was used to encapsulate the sample. The large dark gray regions in the planar surface are locations where individual powder particles were pulled-out of the mount during grinding and polishing. In contrast, another powder sample was mounted in a castable epoxy in Fig. 1b. The particles are retained and the edges are well defined.

Another area of concern, and possibly the most important, is revealing the proper appearance of the pore structure. Because pores are void space, the surrounding material must be cut and removed from the prepared plane. Difficulties arise when the metallic portion of the structure is forced into or over the pores and/or debris from grinding and polishing fills the voids. When viewed at a low magnification, the structure may appear correct as seen in Figure 2a. The appearance seems to be correct with a relatively uniform distribution in both the size and location of the pores, however, when viewed at a higher magnification, closed or partially filled pores are evident. An accurate analysis of this structure is not possible in either the etched or unetched conditions because the true microstructure has not been opened. In particular, performing any quantification on this structure would yield incorrect and grossly misleading results.

When prepared correctly, automated image analysis of properly selected cross-sections can be of great value in both the design and performance evaluations of PM components. In producing PM parts, the normal manufacturing sequence includes die fill and compaction of the powder mixture, then sintering of the ‘green’ compact. The distribution of the porosity within the sintered part is dependent on all of these steps along with several others, including the particle size and shape. Density distribution can have a large effect on the global and local properties of the part and accurately evaluating the local differences in porosity (density) can assist in improving the performance of the parts. Figure 3 is a porosity map of a component cross-section where each cell is colored to represent various porosity levels. Using this information, the designers of the part are able to alter the manufacturing details to improve part performance.
Figure 1. Images show the effects of grinding and polishing of loose powders in different mounting materials. 1a was mounted using diallyl phthalate and 1b with castable epoxy.

Figure 2. The left image (a) shows the as-prepared surface at a low magnification. The surface appears to be accurate, while Fig. 2b is a higher magnification image from the same surface showing unopened and filled pores.

Figure 3. The part cross-section is shown as a color-coded map of the porosity distribution. Overall dimensions are approximately 7.5 mm in height and >15 mm across. The complexity of the shape is seen as the colored cells, while the white cells in the center of the section are not part of the metallic cross-sectional area.