Lineal Intercept Technique for Measuring Grain Size in Two-Phase Polycrystalline Ceramics

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The characterization of polycrystalline ceramics with respect to their mechanical properties, sintering behavior, and grain growth kinetics invariably includes an evaluation of average grain size. The usual procedure for obtaining an average grain size is the lineal intercept technique, which involves measurements taken from polished sections. Ordinarily, a transparent overlay containing one or more test lines of known length is fixed in place over a photomicrograph of the polished section, and the number of intercepts between the test line(s) and grain boundaries are counted.

From this count, the average grain size is obtained using

$$\bar{D}=1.56\frac{C}{MN} \quad (1)$$

where $\bar{D}$ is the average grain size, C the total length of test line used, N the number of intercepts, and M the magnification of the photomicrograph. The proportionality constant, 1.56, is essentially a correction factor which was derived by Mendelson[1] for random slices through a model system consisting of space-filling tetrakaidecahedrally shaped grains with a log-normal size distribution.

In the strictest sense, the underlying assumptions in the derivation of the lineal intercept technique restrict its use to polycrystalline ceramics containing equiaxed grains which have grown normally to form a fully dense single-phase microstructure. In the nonideal microstructure, small amounts of a second phase or porosity have little effect on the accuracy of the measurement if phenomena such as directional solidification and discontinuous grain growth have not seriously affected the size distribution or shape of the grains. However, when the amount of second phase exceeds 5 to 10 vol%, corrections must be applied to achieve an accurate measurement. These corrections depend on the extent of the second phase and its placement in the microstructure.

In the present work, the relatively common situation in which a second phase is noncontinuous and occurs in isolated pockets on grain boundaries and at three-grain intersections is considered. Test lines projected on such a two-phase microstructure will cut across both phases. Thus, to determine the average lineal dimension of the primary phase, the effective length of the test line must be reduced by that amount which lies on the secondary phase. Moreover, it is necessary to recognize and account for two types of test-line intercepts, i.e. those with the boundaries between contiguous grains of the primary phase and those with the interfaces between primary and secondary phases.

Underwood *et al.*[2] have shown that, when lines are projected on random planes taken through a three-dimensional body containing a uniformly dispersed second phase, the proportion of test line lying on the second phase is numerically equal to the volume fraction of the second phase. Thus, the corrected test-line length, C_{eff}, for grain size measurements in the primary phase in a two-phase system can be obtained from either of two equivalent relations:

$$C_{eff}=C(1-v)=C(1-l) \quad (2)$$

where v is the volume fraction of the second phase and l its lineal-intercept fraction.

The lineal-intercept fraction can be measured directly from photomicrographs of polished sections as a function of the total length of test line lying on the second phase. Without the aid of quantitative image analyzing equipment or drum-micrometer microscope stages designed for lineal intercept analysis, direct measurements of the lineal intercept fraction can be quite tedious and subject to significant error. Conversely, the volume fraction of the second phase is often known or easily determined. For example, if the respective densities of the phases are known, the density of the two-phase composite can be measured, and the rule of mixtures can be applied to obtain the volume fraction of the second phase, v_b, from

$$v_b=\frac{\rho_\alpha-\rho_m}{\rho_\alpha-\rho_b} \quad (3)$$

where ρ is the density and the subscripts m, b, and α refer to the two-phase composite, the secondary phase, and the primary phase, respectively. When the second phase consists of grain-boundary porosity, Eq. (3) simplifies to

$$v_p=\frac{\rho_t-\rho_m}{\rho_t}=\frac{\Delta\rho}{\rho_t} \quad (4)$$

where v_p is the volume fraction of pores, ρ_m the bulk density of the porous ceramic, and ρ_t its theoretical density.

Equation (1) must also be modified to account for the two types of test-line intercepts. The effective number of line intercepts with grain boundaries of the primary phase becomes

$$N_{eff}=N_{aa}+\frac{1}{2}N_{ab} \quad (5)$$

where N_{eff} is the effective number of intercepts, N_{aa} the number of intercepts with the boundaries of contiguous grains of the primary phase, and N_{ab} the number of intercepts with the interfaces between the primary and secondary phases.

The modified lineal intercept equation to obtain the average grain size of the primary phase in two-phase systems containing minor amounts of an isolated, noncontinuous second phase is then given by

$$\bar{D}=1.56\frac{C_{eff}}{M\,N_{eff}} \quad (6)$$

where C_{eff} and N_{eff} are defined by Eqs. (2) and (5), respectively.

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