EVALUATION OF PARTICLE STATISTICS BY A SPINNER-SCAN METHOD

Takashi Ida, Taishi Goto and Hisashi Hibino
Ceramics Research Laboratory, Nagoya Institute of Technology,
Asahigaoka 10-6-29, Tajimi, Gifu 507-0071, Japan

It has been proposed [1,2] that the relative error caused by particle statistics in powder diffractometry is given by

$$(\Delta I_{\text{particle}}) / < I > = n_{\text{eff}}^{-1/2} = [4\pi \mu_0 v_{\text{eff}} / A m_{\text{eff}} \Delta\omega \Delta\chi]^{1/2}$$

where n_{eff} is the effective number of crystallites that contribute to the diffraction intensity; μ_0 is the bulk absorption coefficient; A is the cross-section of the beam; $\Delta\omega$ and $\Delta\chi$ are the tolerance angles for the normal direction of the diffraction plane to deviate from the normal of the specimen face along equatorial and axial directions, respectively; v_{eff} is the effective particle volume defined by $< v^2 > / < v >$; m_{eff} is the effective multiplicity of reflection, given by $m_{\text{eff}} = (\Sigma_j m_j I_j)^2 / \Sigma_j m_j I_j^2$, when the observed diffraction peak is composed of multiple reflections with the multiplicity m_j and intensity I_j. The above formula has been originally proposed to describe the integrated intensity of a diffraction peak, but it can also be applied to the peak intensity, only by modifying the interpretation of the equatorial tolerance angle $\Delta\omega$. When we restrict our attention to the peak intensity, $\Delta\omega$ and $\Delta\chi$ are estimated at about 0.03º and 5º/sin θ for a usual powder diffractometer, which suggests that slight rotation of the specimen can alternate most of the diffracting crystallites at a fixed goniometer angle. It is then expected that the particle statistics can be experimentally evaluated by the analysis of intensities measured simply by rotating the specimen stepwise.

Spinner scan measurements with the step interval of 0.9º over 360º were conducted for 11 diffraction peaks of standard Si powder (NIST SRM640c) and three fractions (nominally 3-7, 8-12 and 18-22 μm in Stokes' diameter) of quartz powder samples separated by a sedimentation method. As a result, it has been found that the values of $n_{\text{eff}} \sin\theta$ evaluated from the observed data for the Si sample are almost proportional to the known effective multiplicity m_{eff} ranging from 6 for 400-reflection to 48 for 531-reflection. Since any kind of possible origins for statistical errors other than particle statistics cannot explain such behavior as is proportional to the multiplicity of reflection, it is concluded that particle statistics can certainly be evaluated by the spinner-scan method. When the data of Si powder, the effective diameter of which was estimated at 6 μm by SEM image analysis, are used to determine the instrumental parameters for particle statistics, the effective diameters of the three quartz powder samples are estimated at 7, 12 and 23 μm by the analysis of spinner scan data, while the effective particle diameters evaluated by SEM image analysis were 7, 12 and 25 μm.

References

[1] L. Alexander, H. P. Klug & E. Kummer, *J. Appl. Phys.* **19**, 742 (1948).
[2] P M. de Wolff, *Appl. Sci. Res.* **7**, 102 (1958).