DEVELOPMENT OF MgO CERAMIC STANDARDS
FOR X-RAY AND NEUTRON LINE BROADENING ASSESSMENTS

Suminar Pratapa and Brian O’Connor; Materials Research Group, Department of Applied Physics, Curtin University of Technology, GPO Box U1987, Perth, WA 6845, Australia

The authors have developed a procedure for preparing new line profile standards for use in strain- and size-related broadening studies. The rationale for developing the standards has been the need to develop standards for use with integral breadth single-line procedures for parallel x-ray and neutron diffraction assessments of line broadening effects in MgO ceramics. Accordingly, it was necessary to develop MgO standards. The production of new standards was also of interest for use in general-purpose x-ray and neutron diffraction line profile work as the popular LaB$_6$ NIST SRM 660 cannot be used in neutron investigations due the severity of neutron attenuation.

A series of MgO sintering trials was conducted to establish the relationship between line profile shape and sintering conditions - see Pratapa et al. The basis of this approach is the strain relief achieved through sintering and annealing. An MgO ceramic sintered at 1450°C for 2 hours displayed slightly less Bragg-Brentano x-ray diffraction broadening than SRM 660. Peak profiling using the program SHADOW gave XRD full-widths at half-maximum (FWHM) values for the MgO ceramic and SRM 660 LaB$_6$ of 0.120°(4)* and 0.131°(5); 0.127°(3) and 0.133°(3); and 0.168°(4) and 0.188°(6) for comparable low, medium and high Bragg angles, respectively, for CuK$_{\alpha1}$. Rietveld analysis with the XRD data using the Voigt peak-shape function showed that the $U$ component of the Gaussian component for the MgO ceramic was marginally lower than that for the LaB$_6$ powder, ie. 0.0001(1) compared with 0.0007(3), indicating that less broadening due to microstrain is found with the MgO ceramic. The Lorentzian component gave similar values for both materials (0.0309(3) and 0.0320(6), respectively), indicating very similar and small crystallite size effects. The results show that the MgO ceramic is appropriate for line broadening corrections and therefore can be readily used for line profile analysis. These MgO standards have been used to good effect in both x-ray and neutron diffraction for line broadening studies.

* Number in parentheses is the estimated standard deviation for the least significant figure to the left.

REFERENCE