

## IMPROVING THE ACCURACY OF RIETVELD-DERIVED LATTICE PARAMETERS BY AN ORDER OF MAGNITUDE

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

This is the fourth in a series of papers on Rietveld materials characterisation methods presented at the Denver X-ray Conference [refs 1-3]. Reference 3 considered the importance of managing the zero-point and specimen displacement corrections in extracting lattice parameters from Bragg-Brentano x-ray diffraction data by Rietveld analysis. This paper emphasised the importance of pre-determining the zero-point of the  $2\theta$  scale, and then fixing the parameter in Rietveld calculations. At the same time, the specimen displacement correction must be refined in the Rietveld computations to optimise the reproducibility of the lattice parameters.

This paper evaluates the effectiveness of using the fixed zero-point and associated specimen displacement refinement approach to substantially improve the quality of lattice parameters obtained by Rietveld analysis. A careful zero-point determination will be described which uses the NIST Si 640 line-position standard to extrapolate the plot of  $2\theta_{\text{true}} - 2\theta_{\text{bias}}$  versus  $\cos \theta_{\text{obs}}$  to  $2\theta_{\text{obs}} = 180^\circ$  which then yields the zero-point with an esd of  $0.004^\circ$ . The effectiveness of this approach when used with specimen displacement refinement is demonstrated in the following exercises.

- (1) Determination of lattice parameters accuracy using the NIST SRM 660  $\text{LaB}_6$  standard as a 'test material'.
- (2) Analysis of the influence of specimen re-mounting on lattice parameter precision by the method.
- (3) An exercise on the extent to which use of a multi-position specimen changer influences the precision of the lattice parameter values.
- (4) Demonstration of trends in line-shift related microstrains for suites of  $\text{MgO}$  and  $\text{Al}_2\text{O}_3$  ceramics.

The accuracy of lattice parameters determined by this Rietveld approach is approximately 1 part in 50,000.

### REFERENCES

- [1] B.H. O'Connor and D.Y. Li (2000). *Influence of refinement strategies on Rietveld phase composition determinations*. Advances in X-ray Analysis, 42, 204-211.
- [2] B.H. O'Connor and D.Y. Li (2000). *Attaining 1% accuracy in absolute phase composition levels by Rietveld analysis*. Advances in X-ray Analysis, 43, 305-312.
- [3] B.H. O'Connor, D.Y. Li and B.A. Hunter (2001). *The importance of the specimen displacement correction in Rietveld pattern fitting with symmetric reflection-optics diffraction data*. Advances in X-ray Analysis, 44. In press.