Development of a NIST SRM 1979 Nano-Crystallite Size Standard for Broadening of X-ray Line Profiles

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The development of traceable material standards is critical in the advancement of nano-technology. A NIST nano-crystallite size Standard Reference Material (SRM) 1979 has been developed for this very purpose, assuring improvement in the measurement accuracy of the size distribution and shape of nano-crystallites from X-ray line profile analyses and electron microscopy techniques.

SRM 1979 will consist of two materials prepared with the use of flow reactor technology to permit large, 1 kg batches. Production methods were chosen to minimize the presence of structural defects that may contribute to line broadening. The first material sample is ceria (cerium (IV) oxide, CeO₂) with an (approximate) average spherical crystallite size of 20 nm over a size range of 5–35 nm. The second is zinc oxide (ZnO) with approximately cylindrical crystallite morphology of 80 nm and a size range of 60–100 nm.

The certification of the SRM has also seen the development of a Bayesian/maximum entropy method. This analysis takes full account of the form of the instrumental, background and statistical noise contributions embedded in the diffraction data. As well as providing the most probable solution, the method produces a full error analysis of the size distribution— a critical element in certifying SRM 1979.

The X-ray analysis presented here will be compared with the results of direct observations of SRM 1979 using TEM imaging, and a discussion based on this comparison will be presented.

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