Application Note B-XRD1110

View on rigaku.com

B-XRD1110 - Particle size distribution analysis of ferroelectric nano-powder by USAXS

Introduction

$BaTiO_3$ (BT) is a typical ferroelectric material used in capacitors. For miniaturization of devices, nanoscale BT research has been actively conducted, along with research on the enhanced dielectric properties of nano-capacitor materials. The ferroelectricity of BT at room temperature is strongly related to its tetragonal crystal structure. It has been reported that the dielectric constant increases as BT particle size decreases. However, below a certain critical size, ferroelectricity of BT disappears and it exhibits characteristics of a cubic crystal structure at room temperature. Various measurement methods have been deployed to study the relationship between particle size and structure and the compound's ferroelectricity. Among them, small angle / ultra-small angle X-ray scattering has been used to investigate the particle size of powder materials non-destructively, without dilution. Here, the average particle size and the size distribution of a BT powder are evaluated by ultra-small angle X-ray scattering.

Measurements and results

An Ultra-Small Angle X-ray Scattering (USAXS) measurement was performed on a commercial $BaTiO_3$ (BT) powder sample having particle diameters of 100 nm or less. The sample was sandwiched between Mylar films with a thickness of 2.5 µm and was measured for about 10 minutes. Figure 1(a) shows the USAXS profile and the result calculated by three spherical models. Figure 1(b) is the particle size distribution obtained from the calculation. As a result, it was found that the primary particle size had an average particle diameter of about 30 ~ 50 nm (Model 1 and 2), and an aggregated secondary particle diameter of about 700 nm (Model 3) (Table 1). In addition, BT particles with a diameter of 47 nm are distributed as mono-disperse particles (Model 2). USAXS measurements conducted in a short time allow particles from several tens to several hundred nanometers in diameter to be precisely evaluated at the same time. Furthermore, in addition to the particle size distribution, the dispersion state can be analyzed and discussed based on simulated results using the observed data.

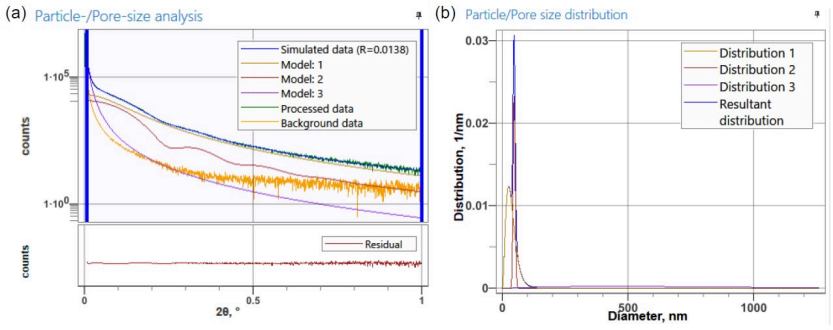

Figure 1: (a) Ultra-small angle X-ray scattering profile of $BaTiO_3$ powder (green line) and the result simulated by three spherical models (blue line), (b) their particle size distributions.

Table 1: Average particle sizes and their size distributions

#	Diameter (nm)	Size RSD (%)
Model 1	36.6	55.4
Model 2	47.2	8.9
Model 3	703.2	62.1

Related products

SmartLab

Advanced state-of-the-art high-resolution XRD system powered by Guidance expert system software

SmartLab Studio II

Windows-based software suite for Rigaku's X-ray diffractometers