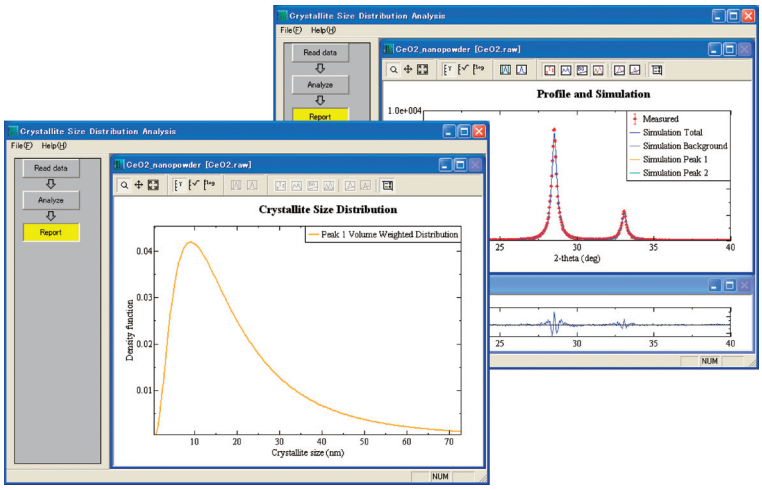
CSDA

Crystallite Size Distribution Analysis Software



1. Introduction

Rigaku is pleased to announce the release of CSDA, a powerful new tool for determining average crystallite size and size distribution.

It is well known that the physical and chemical properties of nanoparticles change remarkably with variations in particle size. For this reason, methods for determining particle size are important for investigating the correlation between the particle sizes of a nanomaterial and its properties. It is frequently difficult to synthesize particles of uniform size, so it becomes necessary to evaluate not only the average size, but also the size distribution.

A "crystallite" is a small region of a solid which can be considered a single crystal, and in general, a "particle" is composed of one or more crystallites. Therefore, "crystallite size" is normally smaller than "particle size". The simplest method for calculating crystallite size is the Scherrer method, in which crystallite size is calculated from the width of an X-ray diffraction peak. However, the average size obtained using the Scherrer equation can be misleading, particularly when the crystallite size distribution of a sample is broad and asymmetric. CSDA overcomes this limitation by simultaneously analyzing both the width and the overall shape of a peak.

2. Advantages

2.1. Analyze size distribution from one peak

When the crystallite sizes in a sample are small, the diffraction peak width is broadened. The shape of the diffraction peak changes depending on the crystallite size distribution.

When the crystallite size distribution is narrow, the peak shape becomes roughly Gaussian—the top of the peak becomes more rounded. Conversely, when the crystallite size distribution is wide, the peak shape becomes sharp, nearly Lorentzian. CSDA can be used to determine the crystallite size and distribution of a sample by analyzing the width and peak shape of one or more X-ray diffraction peaks.

2.2. No need to measure a reference material

The shape of an experimental diffraction peak cannot be used directly to determine the crystallite size distribution. First, it is necessary to make corrections for instrumental broadening, because the peak shape

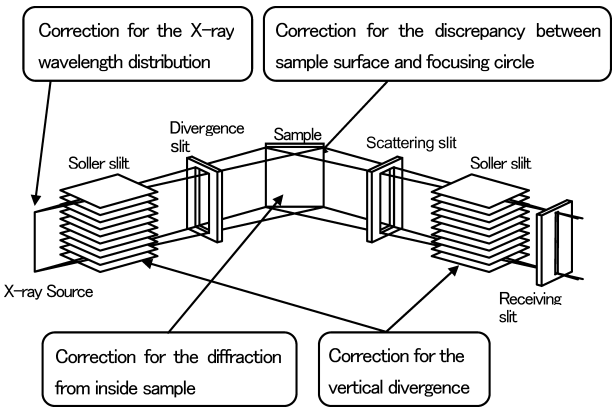


Fig. 1. Illustration of Bragg-Brentano geometry and the corresponding X-ray diffraction peak profile corrections made by CSDA.

changes depending on the experimental conditions (slit widths, specimen transparency, etc.).

CSDA automatically performs the necessary instrumental broadening corrections based on the slit width used in the measurement, the Bragg-Brentano geometry, the linear absorption coefficient of the sample etc. Because the changes in peak shape are taken into account, CSDA can calculate crystallite size distribution accurately. This also makes it unnecessary to use a standard sample to correct the diffraction peak width.

2.3. Use a conventional diffractometer

Because the profiles analyzed by CSDA are measured using the most common Bragg-Brentano geometry, neither a special diffractometer nor an attachment is necessary.

3. Application of ZnO nanocrystals

ZnO has attracted much attention because its photocatalytic properties give it potential to be used in applications for environmental sustainability. Bulk ZnO has a band gap of 3.37 eV, however, it responds to visible light and has attractive photocatalytic properties after a specific heat-treatment is applied. This is considered to be strongly related to its characteristic surface texture and crystallite sizes. Results of the determination of the average size and size distribution of heat-treated ZnO nanocrystals are described below.

Figure 2 shows the (110) X-ray diffraction peaks for ZnO heat-treated at 100 degree intervals from 400 to 800°C. It can be seen that the peaks become sharper as the treatment temperature increases, and therefore, that crystallite size increases with treatment temperature.

The measured peak profiles were analyzed by CSDA.

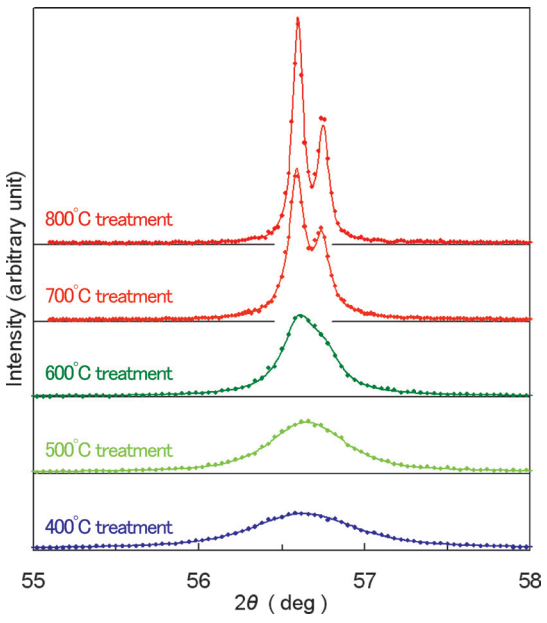
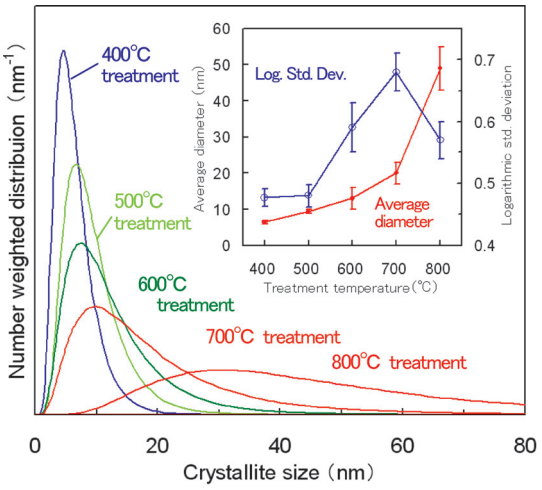


Fig. 2. ZnO (110) diffraction profiles after heat treatments; dots are measured profiles and solid lines are calculated profiles.

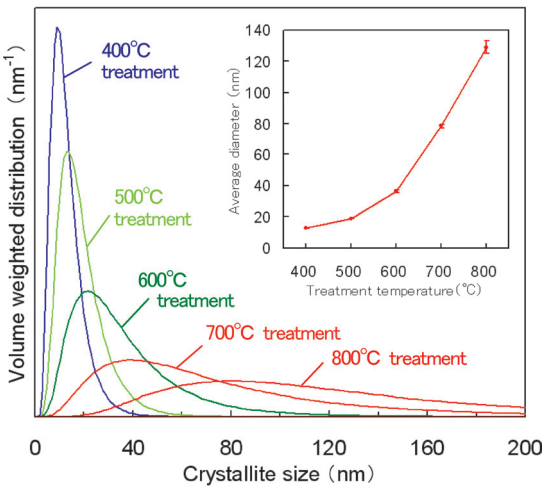
The number-weighted and the volume-weighted crystallite size distributions are plotted in Fig. 3, in Images A and B, respectively. Results on the average diameters of the ZnO crystallites are also shown at the top right hand corners of Images A and B.

Both the number-weighted and volume-weighted crystallite-size distributions curves become broader with increasing temperature. As expected, the average diameters of the crystallites also increase with temperature.

Samples offered by:
Prof. K. Haga, Sendai National College of Technology
Prof. T. Shishido, Institute for Materials Research, Tohoku University



Img. A



Img. B

Fig. 3. Change of crystallite size distribution in ZnO nanocrystal by treatment temperature.

Img. A: Change of number weighted distribution, number weighted average diameters and logarithmic standard deviation.

Img. B: Change of volume weighted distribution and volume weighted average diameter.