Examination of a high and low temperature gas flow equipments using the Large Debye Sherrer Camera

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Structure analysis would be the first step of an investigation of any newly found functional materials. Sometimes, measurement of lattice parameters at various temperatures, either at higher or lower temperatures is important.

Two types of nitrogen gas flow instruments of RIGAKU are individually equipped at the Large Debye Sherrer Camera, BL19B2: one is for lower temperatures, 100 - 450 K, named Low-temperature Spray Cooler (2364B311) and the other is for higher temperatures, 300 - 1000 K, named High Temperature Spray Cooler (2320A1). A performance test is carried out for each instrument and the result is presented here.

Diffraction data of CeO$_2$ powder of NIST, standard sample, are collected at various temperatures. The temperature of the specimen settled at the center of Large Debye Sherrer Camera is controlled by a thermocouple placed at the exit of gas flow tube.

The fine powder of CeO$_2$ is sealed in a silica glass capillary of 0.1 mm φ. The wavelength of incident X-ray is 0.05 nm and the energy of the photon is 24.8 keV. The lattice parameter is refined by the Rietveld method (1).

Figures 1 and 2 show the change in the lattice parameter a of the cubic CeO$_2$ for various temperatures produced by 2364B311 and 2320A1, respectively. Comparisons are made between calculated and measured lattice parameter in the figures. The calculated values are due to an equation, which is experimentally obtained by Taylor et al. (2).

Agreement is fairly good for both instruments.


Structure Analysis of Cellulose Fiber

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Recently, demands for material properties become more complex and severe. High mechanical properties such as high modulus, high strength, high thermal resistance are the most desired characteristics. In order to satisfy these demands, many high performance synthetic polymer fibers and engineering polymers have been developed.

Cellulose is the most popular natural fibers, which is well known to show high mechanical and thermal properties. So cellulose is widely used as clothes, papers and so on. Depending on its processing route, more than six polymorphs are reported for the crystal structure of cellulose. Among them, natural plant cellulose is indexed as so-called the cellulose I form. Further, cellulose I form can be classified into two polymorphs, that is, the algal-bacterial type, (Ia, rich type), and cotton-ramey type (Ib, rich type).

In this study, crystal structure of various cellulose (Ramie, Cladophora (Ia, Ia ≈ 7.3)), Halocynthia (Ib) was investigated using synchrotron radiation (beam size of 0.5mm × 0.5mm, wavelength of 1 Å) with high brilliance and high directionality in BL19B2 Debye-Scherrer camera.

Figure 1 shows the equatorial diffraction profiles of various celluloses. Halocynthia is clear to show the highest crystallinity. Focusing on doublet around 2θ of 10°, it has been reported that the lower angle peak is composed of 110 reflection of Ia and 110 reflection of Ib. On the other hand, the higher angle peak is composed of 010 reflection of Ia and 110 reflection of Ib. In spite of the same chemical structure, the peak intensity ratio was different depending on the origins of natural cellulose.

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Figure 1 Equatorial diffraction profiles of various celluloses

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Ramie

Cladophora (Ia, Ia ≈ 7.3)

Halocynthia (Ib)