Crystallography

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A new electric furnace for synchrotron powder diffraction measurements up to 1807 K

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Introduction

High-temperature synchrotron powder diffractometry is a powerful method to study the crystal structure and phase transformation at high temperatures. Here we report a new compact electric furnace to measure the high-resolution synchrotron powder diffraction profiles from materials at high temperatures up to 1807 K in air, using the multiple-detector system at the BL-4B$_2$ station (Yashima *et al.*, *J. Appl. Crystallogr.*, **38** (2005) 854-855, *J. Am. Ceram. Soc.*, **89** (2006) 1395-1399).

Design of the new furnace

We have designed and fabricated a new electric furnace to measure high-resolution synchrotron powder diffraction data from the specimen at high temperatures up to 1807 K in air. This furnace consists of ceramic refractory with $MoSi_2$ heaters, steel body cooled by flowing water and an automatic sample stage. The sample stage can be rotated about the normal to the sample surface and the position of the sample along the normal direction can be adjusted with a stepping motor. The specimen can quickly be exchanged without the need for further alignment of the furnace. Except for the beryllium or aluminum window for the thermal shield, no part of the furnace exists in the paths of the incident X-ray and diffraction signals from the sample to the Soller slits, leading to high-quality synchrotron powder diffraction data as described below. The temperature of the furnace was controlled with an R-type (Pt/Pt-13wt%Rh) thermocouple placed between the sample and heater. A heating test was performed from room temperature to 1807 K using the new furnace attached to the multiple-detector system installed at the BL-4B$_2$ experimental station of the Photon Factory, High Energy Accelerator Research Organization (KEK), Japan. The present furnace exhibited good temperature stability: the change of temperature with time was small. For example, the temperature at the thermocouple for control was able to be kept constant at 1773.1±0.1 K. Diffraction data from the NIST ceria powder at 1703 K in air were collected in asymmetric flat-specimen reflection geometry.

Performance of the new furnace

The whole powder pattern of NIST SRM ceria was scanned at a step interval of 0.004 deg. in 2θ and counting time of 1.5 s step^{-1}, in just 7 hours. It should be noted that no extra peaks appeared in the whole diffraction pattern. The background intensity was extremely low (8-30 counts) comparing with the strongest peak intensity (56047 counts) in the data measured at 1703 K. It means that the present furnace shows significant performance for precise evaluation of diffraction peak intensities considerably reduced by temperature factors at high 2θ angles, and it is also expected to be useful for study on diffuse scattering at high temperatures. It has been also confirmed that high-resolution diffraction data are obtained with the current system even at high temperatures. The full width at half maximum of the ceria at 1703 K was in the range from 0.0139 deg. at 2θ=21.83 deg. to 0.0575 deg. at 2θ=114.23 deg. Corresponding $\delta d/d$ resolution was ranged from 0.058 to 0.126 % at 1703 K where d and δd are the lattice spacing and peak width, respectively. These results indicate that the present new furnace yields high-quality, high-temperature and high-resolution synchrotron powder diffraction data. This high-temperature system would yield many applications in high-temperature crystallography and structural science.

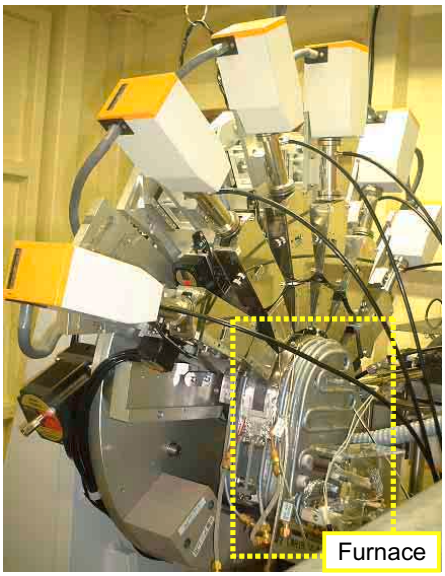

Fig.1. Photograph of the new furnace placed on the stage of the diffractometer at the beam line 4B$_2$ of the Photon Factory.

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