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A compact furnace for synchrotron powder diffraction measurements up to 1807 K

Masatomo Yashima,[a]* Masahiko Tanaka,[b] Kenjiro Oh-uchi[a] and Takashi Ida[c]

[a] Department of Materials Science and Engineering, Interdisciplinary Graduate School of Science and Engineering, Tokyo Institute of Technology, 4259 Nagatsuta-cho, Midori-ku, Yokohama, Kanagawa, 226-8502, Japan, [b]WEBRAM, National Institute for Materials Science, SPring-8, 1-1-1 Kouto Mikazuki-cho Sayo-gun Hyogo 679-5198, Japan, and [c]Ceramics Research Laboratory, Nagoya Institute of Technology, Asahigaoka 10-6-29, Tajimi, Gifu 507-0071, Japan. Correspondence e-mail: yashima@materia.titech.ac.jp

A compact furnace has been designed and fabricated for measurements of high-resolution synchrotron radiation powder diffraction profiles from materials at high temperatures up to 1807 K in air, suitable for the multiple-detector system installed at the BL-4B_2 experimental station of the Photon Factory in Tsukuba, Japan. The whole powder pattern of the material at a high temperature can be scanned at a step interval of 0.004° in 2θ, with a counting time of 1.5 s step^{-1}, in just 7 h.

1. Introduction

High-temperature synchrotron powder diffractometry is a powerful method to study crystal structure and phase transformations at high temperatures (Yashima & Tanaka, 2004). Recently, we designed and fabricated a high-temperature furnace to measure high-resolution synchrotron powder diffraction data at the BL-3A experimental station of the Photon Factory (Yashima & Tanaka, 2004). High-resolution experiments with a single detector at the BL-3A station require much smaller step sizes to measure the diffraction profile, and the scan time for the measurement of the whole powder pattern is long. For example, the scan time for the whole powder pattern from 10.000 to 150.000° in 2θ with a step interval of 0.004° step^{-1} and 1.5 s counting time, was 44 h at BL-3A, including the time for moving the detector arm. Toraya *et al.* (1996) constructed a multiple-detector system with six detector arms for synchrotron powder diffraction measurements at the BL-4B_2 experimental station of the Photon Factory, High Energy Accelerator Research Organization (KEK), Japan. Using this diffractometer, the scan time could be reduced to 7 h. The space around the sample of the multiple-detector system is too small to install the previous furnace that was designed for the BL-3A station. Here we report a new compact electric furnace to measure 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.

2. The furnace

The new furnace, suitable for experiments at the BL-4B_2 experimental station of the Photon Factory (Fig. 1*a*), is a modified version of the previous one suitable for experiments at the BL-3A station (Yashima & Tanaka, 2004). The size of the new furnace (*ca* 20 cm in diameter) is smaller than that of the previous one (*ca* 30 cm in diameter). The distance between the Soller slit (Fig. 1*c*) and the sample is short; therefore a blower for air cooling (Fig. 1*b*) was installed to prevent heating of the Soller slits. This furnace consists of ceramic refractory with $MoSi_2$ heaters, a 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 temperature of the furnace was controlled with an R-type (Pt/Pt–13 wt% 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 (Toraya *et al.*, 1996) installed at the BL-4B_2 experimental station. The new furnace exhibited good temperature stability: the

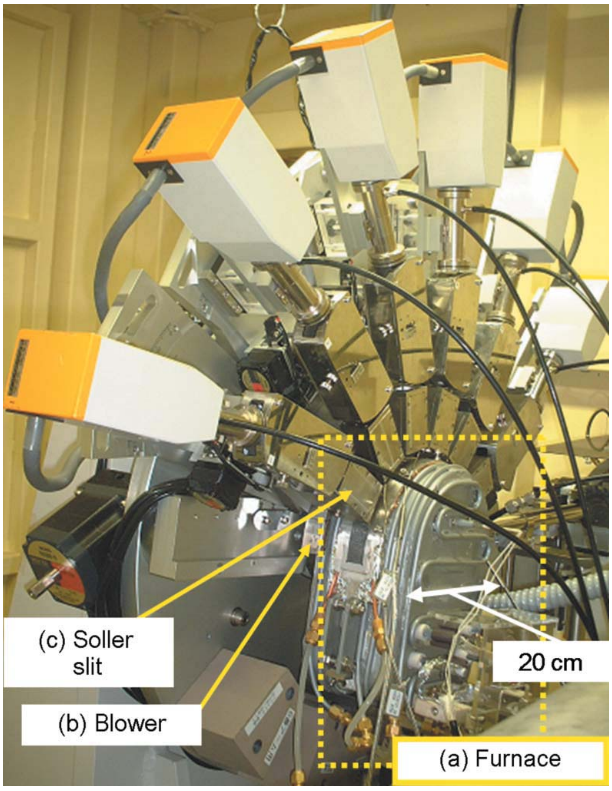

Figure 1
The new electric furnace installed at the multiple-detector system inside the hutch of the BL-4B_2 experimental station of the Photon Factory. The size of the furnace is about 20 cm in diameter.

change of temperature with time was small. For example, the temperature at the control thermocouple could be kept constant at 1773.1 $\pm$ 0.1 K. Using the new furnace, high-quality and high-resolution synchrotron powder diffraction data of NIST SRM ceria were obtained at 1703 K (Yashima *et al.*, 2005). This high-temperature system will be applicable to many applications in high-temperature crystallography and structural science.

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