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Aerodynamic levitation apparatus for structure study of high temperature materials coupled with Debye–Scherrer camera at BL19B2 of SPring-8

Ichiro Sakai [a,*], Kensuke Murai [b], Ling Jiang [b], Norimasa Umesaki [c], Tetsuo Honma [c], Akiko Kitano [c]

[a] *Hiroshima Synchrotron Radiation Center, Hiroshima University, 2-313 Kagamiyama, Higashi-Hiroshima 739-8526, Japan*
[b] *Photonics Research Institute, National Institute of Advanced Industrial Science and Technology (AIST), AIST Kansai, 1-8-31 Midorigaoka, Ikeda, Osaka 563-8577, Japan*
[c] *Japan Synchrotron Radiation Research Institute (JASRI), 1-1-1 Kouto, Mikazuki-cho, Sayo-gun, Hyogo 679-5198, Japan*

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Abstract

An aerodynamic levitation apparatus for determination of structural constants of high temperature materials has been assembled to a large Debye–Scherrer camera at the engineering science research beamline BL19B2 of SPring-8. Diffraction patterns were recorded on the imaging plate with diffraction angles, 2θ, between 0° and 75°. The ball-shaped sample 2 mm in diameter was floated in an inert gas flow system and heated to 1000 °C by controlling CO_2 laser power with a two-color thermometer. Rietveld analysis of our diffraction data for stainless steel (SUS304) as well as other metals was performed to determine lattice parameters and expansion coefficients.

Keywords: Synchrotron radiation; X-ray diffraction; Aerodynamic levitation; Laser heating

1. Introduction

There is increasing demand for characterization techniques for a variety of materials in the high temperature region. There have been a number of investigations of thermal processes in industry and attempts to standardize thermophysical properties and crystal structure. Synchrotron X-ray diffraction studies are among the most powerful tools for in situ investigation of temperature-induced structural changes.

A large Debye–Scherrer camera was developed to collect accurate powder X-ray diffraction data for temperatures up to 800 °C using a high-temperature gas flow system at beamline BL02B2 at SPring-8 [1]. A camera of the same design has been installed at beamline BL19B2 for industrial application [2]. A number of powder diffraction studies of crystalline material have been carried out at various temperatures. However, there are problems when the temperature rises above the melting point of the SiO_2 capillary tube used as the sample container. To overcome these problems, levitation methods have been developed to avoid the influence of the container in a conventional furnace, such as chemical contamination, heterogeneous nucleation, and disturbance of structural measurement. Examples of these methods are aerodynamic levitation [3], electrostatic levitation [4], electromagnetic levitation and aero-acoustic levitation [5]. They enable us to support a heated sample containerlessly. Taking into account the simplicity of the structure of the levitator, we adopted the aerodynamic levitation method and constructed an aerodynamic levitator suitable for the Debye–Scherrer camera at BL19B2 on SPring-8. Diffraction spectra of stainless steel (SUS304) and bearing steel (SUJ2) were measured at various temperatures. Here, we report the results along with a discussion of the effect of the levitation method.

2. Experimental procedures

Beamline BL19B2 is composed of a double crystal monochromator and deflecting focusing mirror. The available

* Corresponding author.
E-mail address: i-sakai@isc.osakac.ac.jp (I. Sakai).

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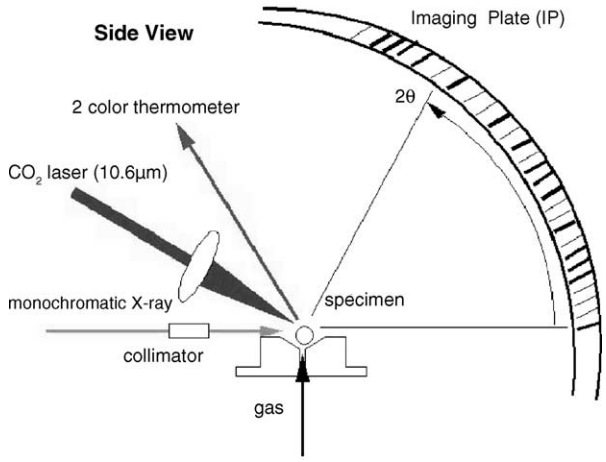

Fig. 1. Schematic illustration of the experimental setup.

photon beam energy range is from 5 to 117 keV with resolution of 10^{-4}; other performance parameters are described in Ref. [2].

The large Debye–Scherrer camera is installed at the second experimental hutch. Diffracted X-ray intensity was recorded using an imaging plate (IP) on the 2θ arm with a radius of 286.5 mm, so that it is possible to obtain diffraction patterns between 0° and 75° with high resolution within a reasonable time (5 min).

Fig. 1 shows a schematic illustration of the experimental setup showing the levitated sample using an inert argon gas flow system with a conical nozzle. The ball-shaped sample 2 mm in diameter was floated using the inert gas flow system.

A CO_2 laser (SYNRAD 100; 100 W at 10.6 μm) was used to heat the sample. The laser beam was focused on the sample using a ZnSe lens. The temperature of the heated sample was measured with a two-color thermometer (Hamamatsu Photonics Co., Ltd., Hamamatsu, Japan) while controlling the laser power. Sample temperatures were stable to about ±20 °C during the diffraction measurements. Taking into account detection efficiency, an X-ray energy of 24.8 keV (0.5 Å) was selected by the Si(1 1 1) reflection system. The levitation system was assembled with the Debye–Scherrer camera as shown in Fig. 2. The flow rate of argon gas was 100 cm^3/min. Fig. 3 shows a photograph of the levitated sample taken with the CCD camera. The sample was rotated automatically when it was levitated. We measured the diffraction patterns of SUS304 and SUJ2 with changes in power of the CO_2 laser.

3. Results

Fig. 4 shows the measured diffraction patterns of SUS304 for various temperatures up to 900 °C. Combustion of the sample was caused by oxidation over this temperature. The position of the peaks decreased appreciably with increases in temperature. We applied Rietveld analysis to the present

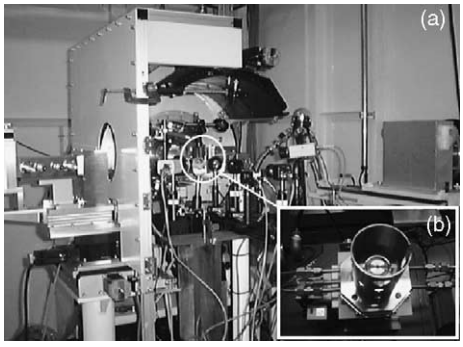

Fig. 2. Photograph of the large Debye–Scherrer camera coupled with levitation systems showing (a) the optical observation systems and large cylindrical IP and (b) a part of the conical nozzle on the *xyz*-stage.

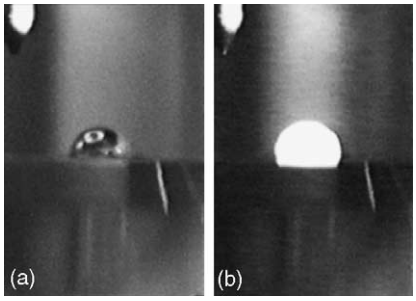

Fig. 3. CCD images of the levitated sample with the laser (a) off and (b) on.

experimental results for determination of the lattice constants. Fig. 5 shows a typical example of the Rietveld fit [6] for the SUJ2. Dots are data points and full lines indicate the results of the Rietveld fit. The obtained cubic lattice parameters *a*, *b* and *c* were 2.824 ± 0.009 Å.

Thermal expansion coefficients can be obtained from measured lattice spacings for various temperatures. The estimated thermal expansion coefficients are plotted against the temperature in Fig. 6. The error bars represent the errors in the measured values of lattice constants and temperature. The thermal expansion coefficient varied gradually with increasing temperature.

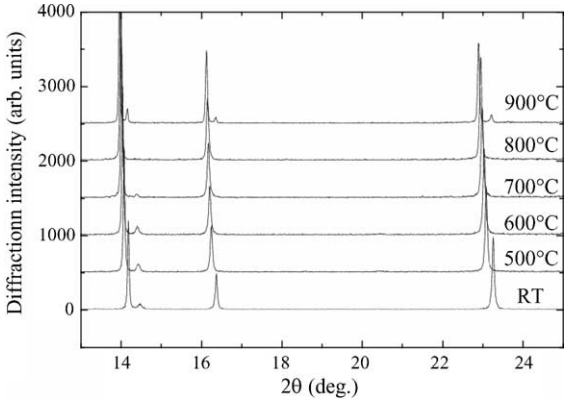

Fig. 4. Shifts of the diffraction peaks with SUS304 sample temperature.

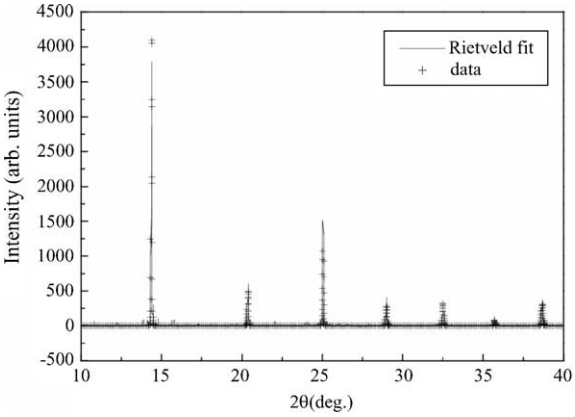

Fig. 5. Example of Rietveld fit of SUJ2 at 700 °C. R factors were $R_{wp} = 24.26$, $R_p = 18.81$, $R_R = 44.14$, $R_e = 14.50$ with a goodness-of-fit indicator of $S = R_{wp}/R_e = 1.6739$.

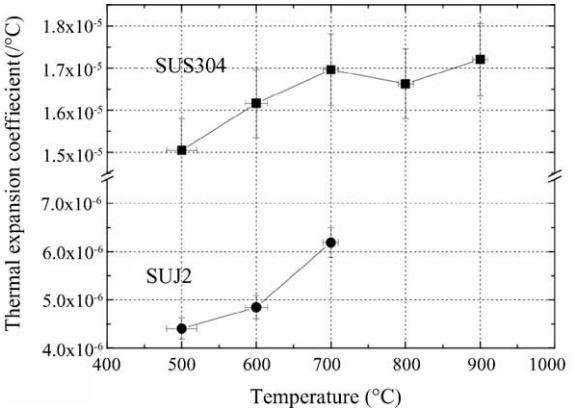

Fig. 6. Thermal expansion coefficients of (■) SUS304 and (●) SUJ2 as a function of temperature.

4. Discussion

The results of the present study indicated that the levitation method enables to increase the temperature up to 1000 °C for metals and this method was introduced successfully into diffraction experiments using the Debye–Scherrer camera. Temperatures over 2000 °C can be achieved for oxide specimens. A combination of aerodynamic levitation and laser heating may be useful for glass and ceramics as well as metals. For further measurement, the levitation apparatus under vacuum should be employed to avoid combustion of easy oxidizable materials, such as metals.

If measurement conditions above the melting point are required, oxidation of samples must be avoided. We have begun to construct an aerodynamic levitation system under vacuum, which will be especially useful for easily oxidisable metal samples.

Possible sources of error in measurement of the lattice parameter are the error in the origin of the 2θ axis derived from measurement of the direct beam profile and in the primary X-ray beam energy. The lattice parameters were estimated to be accurate to about ±0.3%. On the other hand, there may also be possible error induced by temperature fluctuation of the sample. Taking all these points into consideration, the thermal expansion coefficients obtained in the present measurement were estimated to be less than ±5% error.

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