Development of an Electrostatic Levitator for Neutron Diffraction Structure Analysis

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Abstract

An electrostatic levitation (ESL) furnace was designed for the neutron diffraction study of condensed matter. This apparatus is composed of an electrostatic levitator, a neutron inlet path with Cd slit, a 160 degrees window to detect scattered neutrons, a CO_2 laser for sample heating, and an optical pyrometer for temperature measurements. A preliminary neutron diffraction experiment with this ESL was performed with polycrystalline alumina samples. The sample position could be controlled with an accuracy of ±0.1 mm. The observed Bragg peaks were in complete agreement with those derived from the lattice data of alumina obtained from the literature. This indicates that this facility is effective for the structural study of condensed matter without containers.

Introduction

In recent years, a lot of attention has focused on the utilization of the containeless technique for structure analysis of materials[1-5]. The containerless technique enables structure analysis of corrosive materials or liquids at high temperatures. For these materials, it is difficult or sometimes impossible to find suitable containers. In addition, levitation methods offer samples with nearly spherical shape which simplifies diffraction data analysis. Moreover, because containerless processing removes the risk of heterogeneous nucleation from the wall of a container, the structural study of deeply undercooled liquids can be performed. In particular, electrostatic levitation (ESL) can be applied to a wide variety of materials, from metals to insulating materials. In addition, ESL experiments can be performed over wide temperature ranges because the mechanism of levitation is independent of that of sample heating. Until now, several studies have been performed to implement electromagnetic levitation and aerodynamic levitation techniques to synchrotron X-ray or neutron structure analysis of liquids. The motivation of developing an ESL lies in the fact that it can be applied to various kinds of materials. Therefore, it is highly desirable to develop such a facility for the structure analysis of condensed matter coupled with either a synchrotron X-ray source or a neutron source. This short report describes the development of an ESL for liquid structure analysis and the result of a preliminary application to neutron scattering structural analysis. In NASDA, the meta-stable material research team had established an electrostatic levitation furnace for processing and thermophysical measurement of high temperature materials[6-7]. The development of an ESL furnace for neutron diffraction is based on their technique which was described in detail elsewhere[8].

Experimental

Fig.1 depicts the ESL developed. The dimension of the chamber is 0.25 m in diameter and 0.35 m in height. Sample loading was achieved by dropping a solid sample from a 18 specimen capacity carousel from the top part of the chamber. The levitator consists of a pair of parallel disk electrodes (vertical control) and two pairs of spherical electrodes (horizontal control). The sample position was monitored by two sets of He-Ne laser and was controlled independently in X,Y, and Z directions via feedback loop. During levitation, ultraviolet light beam irradiated the sample to keep its electric charges through photoelectric emission. For high temperature experiment, the sample was heated by the radiation of a CO_2 laser coming from the top part of the chamber. Computer controlled laser power allowed to maintain a constant temperature sample. The temperature of the sample was recorded with a pyrometer. The incident neutron beam is introduced from the entrance port onto the sample. A cadmium slit was located at 50 mm ahead of the sample position to adjust the incident neutron beam size to eliminate sharp background intensity from the copper electrodes. The slit width was set to 3 mm, corresponding to sample size. The scattered neutrons exited the chamber through an aluminum port. As a preliminary test, a neutron diffraction experiment was performed with an alumina polycrystalline sphere. A 3

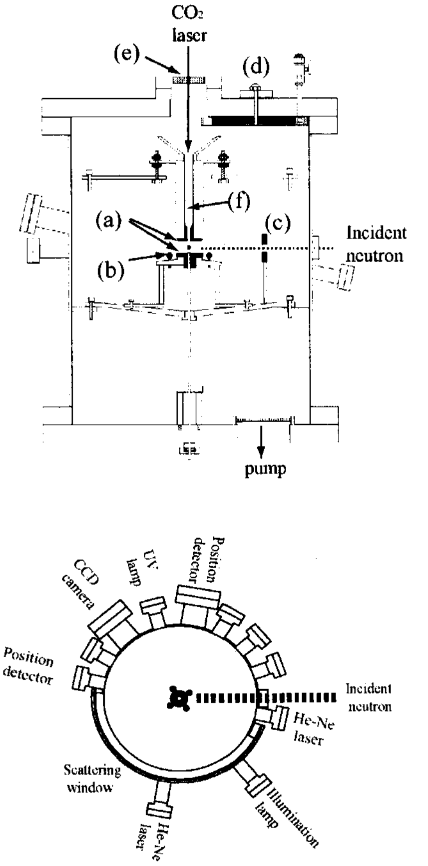

Fig. 1. The developed electrostatic levitator for neutron diffraction:
(a) main electrodes
(b) side electrodes
(c) cadmium slit
(d) carousel
(e) ZnSe window
(f) guide tube for sample loading and CO_2 laser heating.

mm diameter sphere was coated with a platinum layer of a few ten angstroms thickness. The coating was performed to maintain the positive charge on the sphere. The sample was kept levitated for more than 12 hours. The sample position oscillation was estimated to be less than ±0.1 mm along the three axes from the video images. The vacuum level of the furnace was about $5x10^{-4}$ Pa. The intensity of scattered neutrons was analyzed with the HRPD (High Resolution Power Diffractometer) spectrometer of the JRR-3M reactor of JAERI (Japan Atomic Energy Research Institute). The HRPD has 64 detectors which covers the range of scattered angle, 2θ, from 5 to 165 degrees. The neutron wavelength used was 1.823 Å. The diffraction data of levitated sample were obtained with and without the cadmium slit. The background intensity from the furnace was also obtained for both cases. The intensity data were obtained by increments of 0.05 or 0.10 degree as a function of the scattering angle, 2θ and were normalized to the incident neutron number, which were counted by the monitor counter. The whole measurement was performed at room temperature.

Results and Discussions

The intensity data without the slit are shown in Fig. 2 for the alumina sample and the background respectively. The strong diffraction intensity was observed from the copper electrodes. The intensity of the sample was smaller than that of copper. In addition, some peaks of the sample overlapped with those of copper. By contrast, as shown in Fig. 3, there were no sharp peaks in the background data when the cadmium slit was employed. This indicates that the ESL technique can remove easily the sharp diffraction peaks of the furnace materials. This is an advantage compared with the electromagnetic levitation and the aerodynamic levitation methods for which the rf coil and the gas nozzle located near the sample generated sharp peaks. Peaks of platinum were not observed because the diffraction intensity of the platinum coating of a few ten angstroms was quite small in comparison with that of alumina itself. The diffraction intensities of the alumina specimen after the subtraction of the background are shown in Fig. 4. The ten peaks were clearly observed. These peaks were identified as those derived from the mirror indices of

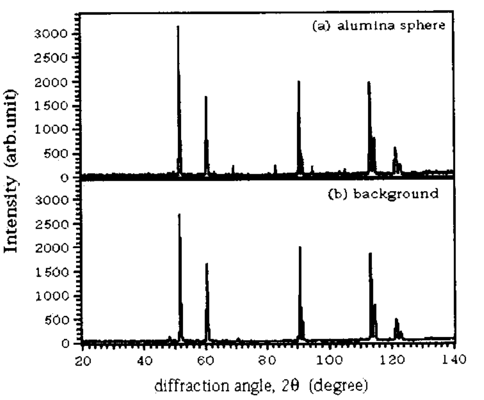

Fig.2 Intensity of alumina and background without the Cd slit.

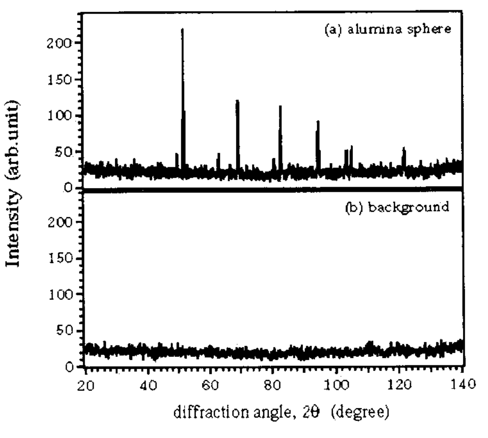

Fig.3 Intensity of alumina and background with the Cd slit.

hexagonal structure of alumina. Observed peaks were in complete agreement with the literature[9]. The lattice parameters were determined with the use of these ten peaks by the standard procedure. The determined lattice constants were a=4.760 Å and c = 13.00 Å. These values are in close agreement with the previous data[9].

Previously, a neutron diffraction experiment of solid alumina was performed by aerodynamic levitation. The background intensity of the present data was weaker than that of this previous experiment. In addition, rather broad peaks were observed for alumina sample in the previous study. The present study shows sharp peaks, as depicted in Fig. 4.

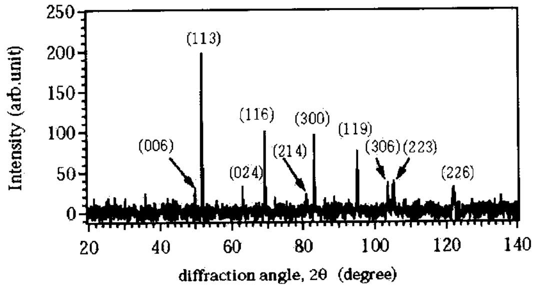

Fig.4 Intensity of alumina with the corresponding mirror indices.

Conclusion

The present study indicates that the ESL coupled with neutron diffraction is a promising method for structural study of condensed matter. With some modifications, a similar useful facility could also be available for X-ray structure analysis. Preliminary experiments to investigate the structure of liquid zirconium with neutron scattering are currently underway.

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