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High-Temperature Measurements of Materials

With 125 Figures

Springer

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Preface of Series by the Editor-in-Chief

This book titled *High-Temperature Measurements of Materials* is the 11th volume in the series "Advances in Materials Research" edited by Professors Hiroyuki Fukuyama and Yoshio Waseda. The book is composed of nine chapters, the contents of which try to solve the currently important basic problems in high-temperature melts from scientific basics to real application fields related to industrial problems. The contents of this present volume are expected to contribute to solving the most important problem of sustainability of the global society by applying recent high-level scientific and engineering researches in materials science. Recent important contributions from large scale experimental facilities from over the world are collected and explained in detail in this book, where we can now measure clearly the important behaviors of high-temperature melts which have never been observed by traditional experimental techniques. This book also introduces recent advancements in computer simulations, which have become very effective not only in explaining experimentally observed phenomena but also in predicting materials properties and processes which are difficult to be measured for the materials under high-temperature environments.

As the series editor, I thank Dr. Claus Ascheron of Springer-Verlag, who always has interest in and kindly takes care of our research activity to encourage publication in this series of books.

Sendai, July 2008 *Yoshiyuki Kawazoe*

Preface

A variety of industries – information technology, aerospace, automobile, and basic and new materials manufacturing – need technological innovations, which bring high-value-added and high-quality products at low cost not only because of global competition, but also because of the perspective of environmental consciousness and regulation. Thermophysical properties of high-temperature melts are indispensable for numerical simulations of material processes such as semiconductor and optical crystal growth of the melt, and casting of super-high-temperature alloys for jet-engine turbine blades, in addition to welding in automobile manufacturing. Recent developments in process modeling provide 3D unsteady analysis of melt convection, temperature, and heat flux distribution, which enables us to predict product quality. In fact, 3D process visualization using computer modeling helps us to understand complicated phenomena occurring in the melt and to control the process.

Accurate data are necessary to improve the modeling, which cost-effectively engenders high-quality products. However, crucial obstacles render measurements of thermophysical properties difficult at elevated temperatures because of high chemical reactivity and fluidity of melts. Substantial and persistent challenges have been made to ascertain the precise thermophysical properties of high-temperature melts. This book describes the new techniques and latest developments in the measurements of atomic structure, density, surface tension, viscosity, heat capacity, thermal and mass diffusivity, thermal conductivity, emissivity, and electrical conductivity of high-temperature melts.

In addition to up-to-date improvements in conventional techniques, some new attempts are introduced to open a new scientific field, that is, physics of high-temperature melts. Space-related organizations such as Japan Aerospace Exploration Agency (JAXA) and German Aerospace Center (DLR) demonstrate the importance of noncontact measurements and microgravity environments. Recent progress in levitation techniques enables high-precision measurements of various properties of stable and deeply undercooled metallic melts. An electrostatic levitation apparatus is specially designed not only

to measure thermophysical properties but also to study the short-range order of metallic melts in combination with a synchrotron radiation facility: SPring-8 (Japan Synchrotron Radiation Research Institute, JASRI). A noncontact measurement of thermal conductivity was developed using an electromagnetic levitator incorporating a superconducting magnet (High Field Laboratory for Superconducting Materials, Institute for Materials Research, Tohoku University). An electromagnetically levitated droplet behaves as a hard sphere in a static magnetic field. The oscillation and convection of the droplet are suppressed because of the Lorentz force, which enables measurement of its true thermal conductivity using modulation laser calorimetry. Utilization of microgravity conditions provides an ideal environment without a fluid flow driven by buoyancy force. Diffusion coefficients of metallic melts have been determined using the shear cell technique under microgravity using a sounding rocket.

As described earlier, this book is a unique compilation of information related to recent advances in high-temperature measurements and thermophysical properties data. The editors earnestly hope that this book is a useful guide for the scientists and engineers who are working in the field of materials science and processing, and that it is attractive to students interested in the physics of high-temperature melts.

The editors thank Yoshimasa Ito and Miwa Sasaki at the Institute of Multidisciplinary Research for Advanced Materials (IMRAM), Tohoku University for preparing TeX manuscripts and figures. The editors gratefully acknowledge the encouragement and patience of Dr. Claus Ascheron of Springer-Verlag.

Sendai
July 2008

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1

Measurement of Structure of High Temperature and Undercooled Melts by using X-Ray Diffraction Methods Combined with Levitation Techniques

Tadahiko Masaki, Akitoshi Mizuno, and Masahito Watanabe

1.1 Introduction

The knowledge of the microscopic feature of matter is of paramount importance in materials science. In particular, the information about the atomic configuration is essential for the understanding of the characteristic properties of disordered matter. Therefore, a huge amount of efforts has been devoted to the development of experimental techniques coupled with X-ray or neutron diffraction techniques [1–3] to study the structure of liquids. In this decade, intense and high-energy X-ray beam sources, especially synchrotron radiation facilities, have emerged, and can be used for diffraction experiments of disordered matter. Compared to former experimental facilities, they enabled us to perform the highly precise investigation of the structure of liquids in a much shorter time.

Although the methods and facilities for diffraction experiments have been improved rapidly, the sample handling techniques of high-temperature liquids have not been advanced so much because of the difficulty in the selection of crucible materials. In the case of liquid metals, several ceramics (e.g., fused silica, sintered alumina, sapphire, graphite, boron nitride) have been used for crucibles. Despite the adoption of these various materials, the maximum temperature of the experiments has been limited due to the corrosion of the crucible.

Levitation techniques use a variety of external forces (e.g., aerodynamic [4], acoustic [5], electromagnetic [6], and electrostatic [7]) to hold a small amount of material in space without a crucible. When a levitated sample is in its liquid phase, it takes a spherical shape because the lack of crucible minimizes the surface free energy. In particular, a great deal of attention has been given to the measurement of the thermophysical properties of extremely high-temperature melts and the study of solidification phenomena from deeply undercooled liquids.

The levitation technique can also be a very elegant way to handle liquid samples on diffraction experiments. For these experiments, they offer many advantages. As the liquid sample is held containerless, there is no need to subtract the diffraction contribution of a crucible from the total scattering intensity. Hence, this reduces by half the measurement time because it is not necessary to measure the diffraction of the empty cell. In addition, the symmetrical shape (nearly spherical) of the sample enables us to evaluate easily the correction of absorption and multiple scattering. Moreover, the most important advantage is the fact that the liquid samples under containerless conditions can easily reach deep undercooled states because the heterogeneous nucleation sites, which are usually the inner wall of the crucible, are absent. The observation of the structure of undercooled liquids can be realized by these techniques.

Up-to-date, several levitation techniques have been applied to the diffraction experiments for the structural analysis of liquid matters, as shown in Table 1.1, in which the typical works of respective levitation techniques are summarized. The aerodynamic levitation is simple, yet useful for such experiments [8–25]. In this method, a sample is levitated in a conical nozzle at the location of the minimum potential well of the gas flow. Since the sample is small (1–3 mm diameter), this levitator is well suited for with the application to a high-energy synchrotron radiation X-ray beam. Moreover, this process can be applied to several types of materials under various atmospheres, though this cannot be used under vacuum conditions. So far, X-ray diffraction experiments of several high-temperature melts (e.g., liquid boron, alumina) have been performed by this technique and provided the static structure factors of these liquids [8–25]. Recently, using this method, liquid structures of glass-forming alloys and ceramics have been studied together with the discussion of total and partial structure factors with the aid of the computer simulations [11, 12]. The electromagnetic levitation is another technique applicable to diffraction experiments. In this method, a sample of conductive material is levitated in a RF coil. The high frequency current of the coil induces an eddy current in the metallic sample and the electromagnetic force is induced for the levitation. The levitated sample is positioned at a stabilized point, which depends on the shape of the coil and on the electromagnetic properties of the sample. Since the sample size is large (6–8 mm diameter), this method is especially well suited to neutron scattering experiments. Schenk et al. [29] applied this levitation method to neutron and X-ray scattering experiments of equilibrium and non-equilibrium liquid metals. Recently, Watanabe et al. [26] studied the structure of liquid silicon by using this technique.

It is also possible to levitate matter by applying electrostatic forces, under an active feedback system, to charged samples by electronic emission [39–43]. Electrostatic levitation is extremely attractive for X-ray diffraction experiments for several reasons. The size of the levitated sample (1–2 mm diameter) is suitable for the diffraction of high-energy X-rays from synchrotron radiation source, taking into account the X-ray absorption coefficient and the atomic

Table 1.1. Liquid structural analysis by using diffraction methods coupled with levitation techniques

Levitation	Material	Structural analysis	Ref
CNL	Si	XRD	[8]
	Si	XRD	[9]
	Si	IXS	[10]
	CuZr, NiZr	XRD	[11]
	CuZr	XRD	[12]
	$Co_{80}Pd_{20}$	XRD	[13]
	Y_2O_3, Al_2O_3, B, Si, CoNi, Ni	XRD, NS	[14]
	Al_2O_3	IXS	[15]
	Al_2O_3	XRD	[16]
	Al_2O_3, ZrO	EXAFS	[17]
	Al_2O_3	XRD	[18]
	$MgAl_2O_4$, $CaAl_2O_4$, Al_2O_3	XRD, NS, IXS	[19]
	BaB_2O_3	XRD	[20]
	Mg_2SiO_4, CuZr	XRD	[21]
	Y_2O_3	XRD, NS	[22]
	Y_2O_3	AXRD	[23]
	YAG	EXAFS	[24]
	SiO_2	XRD	[25]
EML	Si	XRD	[26]
	Si	XRD	[27]
	Si	ED-XRD	[28]
	Ni, Fe, Zr	NS	[29]
	Ti	NS	[30]
	Fe, Zr, Ni, $Al_{65}Cu_{25}Co_{10}$	NS	[31]
	Al–Cu, Al–Ni	XRD	[32]
	AuCu, PdCuSi, Si, CoPd	EXAFS	[33]
	$Co_{80}Pd_{20}$	EXAFS	[34]
	Co–Pd, Au–Cu–Co	EXAFS	[35]
	Ni–V	XRD	[36]
	Nd–Fe–B	XRD	[37]
	Ti–Fe–Si–O	ED-XRD	[38]
ESL	Si	XRD	[39]
	Ni, Ti	XRD	[40]
	$Ti_{39.5}Zr_{39.5}Ti_{21}$	XRD	[41]
	TiZrNi	XRD	[42]
	Ba–Ge	XRD	[43]

CNL Conical nozzle levitation, *EML* Electromagnetic levitation, *ESL* Electrostatic levitation, *XRD* X-ray diffraction, *NS* Neutron scattering, *ED-XRD* Energy dispersive X-ray diffraction, *IXS* Inelastic X-ray diffraction, *AXRD* Anomalous X-ray diffraction, *EXAFS* Extended X-ray absorption fine structure

scattering factor of typical high-temperature metallic melts. In addition, as the charged liquid sample is levitated between pairs of electrodes, the sample is free from any obstacle, such as the nozzle or the coil in other levitators. Moreover, to avoid electrical breakdown on the application of a high voltage between electrodes, electrostatic levitators have to be operated either under pressurized atmospheres (∼0.4 MPa) or under high vacuum. The high vacuum conditions are particularly advantages for X-ray diffraction because there is no need to consider the scattering from the ambient gas. Recently, Gangopadhyay et al. [44] used such levitators for X-ray diffraction in a synchrotron radiation facility and observed the static structure factors and solidification behavior of several metallic melts. Electrostatic levitation has also been applied by Aoki et al. [45] to neutron diffraction experiments. They successfully measured the diffraction pattern of sintered alumina at room temperature. Although the validity of electrostatic levitation for diffraction experiments has been recognized, the previous electrostatic levitators exhibited limitations for precise measurements. In particular, the limitation was present for the observable range of the diffraction angle, which affects the resolution of the data obtained through a Fourier transform. The atomic configuration of liquids in real space can be investigated from the radial distribution function, $g(r)$, which is obtained by a Fourier transformation of $S(Q)$ as follows:

$$g(r) = 1 + \frac{1}{2\pi^2 \rho r} \int_0^\infty \Big[S(Q) - 1\Big] Q \, \sin Qr \, \mathrm{d}r, \tag{1.1}$$

where ρ is the number density, $S(Q)$ is the static structure factor, and Q is the momentum transfer. The $g(r)$ obtained from diffraction experiments involves experimental errors, essentially because of the limited Q range of $S(Q)$ due to the wavelength of X-rays and the available range of 2θ, and the diverted tail of the direct beam. In addition, the previously developed electrostatic levitators could be used only in large beam source facilities (e.g., synchrotron radiation facilites [44] or nuclear reactors [45]). Therefore, this situation restricts the opportunities for experiments because of the limited machine time.

Recently, we developed an electrostatic levitator for X-ray diffraction measurements with a high applicability to various beam sources [46]. Since the system is very compact, it can be utilized coupled not only with the diffractometer at the high-energy X-ray diffraction beamline, BL04B2 of the synchrotron radiation facility, SPring-8 [47], but also with a laboratory X-ray system (RIGAKU RINT). The present electrostatic levitator was designed for the X-ray diffraction measurements by a two-axis diffractometer with slits collimation coupled with a germanium detector or a proportional counter. The scattering intensity for each scattering angle, 2θ, was acquired by the counter with the step-scan method. The high-energy X-ray beam from the synchrotron source of over 100 keV is a very attractive probe for the liquid structure analysis compared with the laboratory X-ray sources. The structure of liquid 3d or 4d metals can be measured by using the high-energy X-rays due to the high penetration of incident X-ray to samples. In addition, since

the momentum transfer, $Q = 4\pi \sin\theta/\lambda$ (2θ, scattering angle; λ, wavelength of incident X-rays), is proportional to the X-ray energy, the static structure factor, $S(Q)$, in sufficiently wide Q range can be obtained from the measurement of diffraction pattern with small scattering angles. On the other hand, the laboratory X-ray source can be used for diffraction experiments of lighter materials, such as silicon. Since the laboratory X-ray source is free from the restriction of user time of the facility, preliminary or challenging experiments can be performed with trial and error. Therefore, the laboratory X-ray experiments complement synchrotron X-ray ones.

In this report, we describe the electrostatic levitation system for the observation of high temperature liquid structures and present the results of a preliminary application to the atomic structure analysis by X-ray diffraction measurements.

1.2 Electrostatic Levitator for the Structural Analysis by X-Ray Diffraction Technique

The design of the present apparatus was based on an electrostatic levitator developed by Rhim et al. [7]. However, the present levitator was optimized for the liquid structure analysis of high-temperature melts by X-ray diffraction technique. The apparatus consists of a vacuum chamber, a sample position control system, and a sample heating source. The sample, charged by electronic emission, is levitated by applying an electrostatic field (typically 20–30 $\mathrm{kV\,cm^{-1}}$ for metallic materials) between two electrodes. To prevent the electrical breakdown, the electrodes are contained in a chamber that is evacuated to a level of vacuum lower than 1×10^{-4} Pa by a turbo molecular pump attached directly to the side of the chamber. Figures 1.1 and 1.2 illustrate the side and top views of the chamber, respectively. The chamber has a cylindrical shape (height, 200 mm; diameter, 200 mm) and comprises several view ports. A thin sapphire window (thickness, 0.5 mm; diameter, 17 mm) enables the incident X-ray beam to reach the sample. With the use of a rectangular and curved beryllium window, the intensity of the X-rays diffracted by the levitated sample can be detected over a wide angle. The available range of 2θ is $-5°$ to $80°$, which is wider than that previously reported [44]. Sufficiently wide Q range ($Q \sim 11.5\,\mathrm{\AA^{-1}}$) can be obtained even for laboratory X-ray source (Mo Kα). Five silica glass windows, located on the top of the chamber, are used, along with mirrors inside the chamber, for the position control system and the sample observation by a camera. A ZnSe window (or lens) in the middle of the top plate was used for the sample heating by a CO_2 laser (wavelength, 10.6 μm; max. power, 240 W). A glass window on the side of the chamber was employed for the temperature measurement by a single-color pyrometer. Two valves located on the top plate acted as an air lock that is equipped to insert samples without breaking the vacuum.

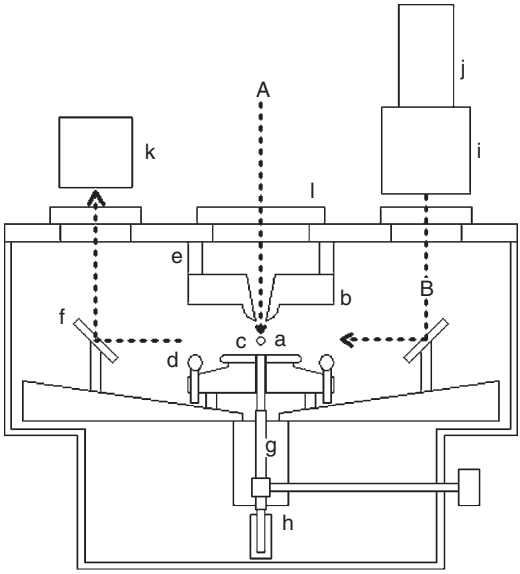

Fig. 1.1. Side view of the chamber for electrostatic levitation. *a*, levitated sample; *b*, upper electrode; *c*, lower electrode; *d*, side electrodes; *e*, ceramic support; *f*, mirrors; *g*, positioning rod; *h*, solenoid; *i*, beam expander; *j*, He–Ne laser; *k*, position detector; *l*, ZnSe window; *A*, beam path of heating CO_2 laser; *B*, beam path of positioning He–Ne laser

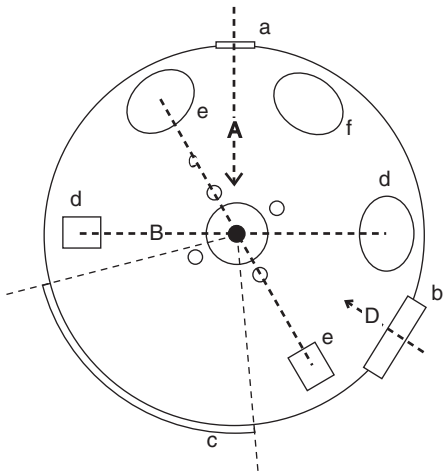

Fig. 1.2. Top view of the chamber for electrostatic levitation. *a*, sapphire window for the incident X-ray beam; *b*, glass window for the pyrometer; *c*, beryllium window for diffracted X-rays; *d*, mirrors for the He–Ne laser for positioning in X-Z directions; *e*, mirrors for the He–Ne laser for positioning in Y direction; *f*, mirror for CCD camera; *A*, path of incident X-rays; *B* and *C* path of He–Ne lasers; *D*, path of pyrometer

The design of the electrodes is of utmost importance for electrostatic levitators. In our levitator, there are two main electrodes for vertical and horizontal control and four side electrodes for additional horizontal control. The main electrodes consist of two parallel disks. The upper electrode (40 mm diameter), which is connected to a high voltage amplifier, is suspended from the top plate by using insulating ceramic rods. The upper electrode has a spherical end, which generates a concave electrostatic field that helps the position of sample to stabilize laterally. In addition, a hole (3 mm diameter) in its center is available for the sample heating by the CO_2 laser. The lower electrode (20 mm diameter) is electrically grounded and possesses a hole, which is available for sample handling by a positioning rod. This rod can be moved up and down from the outside of the chamber to set the initial position of sample. Four small spherical electrodes are distributed around the lower electrode for additional control of the sample position along the horizontal direction.

To maintain stable sample levitation, the voltage of the electrodes is controlled actively coupled with a position sensing system, a computer, and high-voltage DC amplifiers. The position of the levitated sample is detected by two sets of position sensors and associated He–Ne lasers. In each set, the expanded He–Ne laser beam (10 mm diameter) illuminates the sample and its shadow is projected on the position detector, which is located on the opposite side of the He–Ne laser. The computer receives from the position detectors an electric signal that corresponds to the sample position. It then calculates the control signal by using a PID control scheme and sends the proper information to the voltage amplifier that changes the voltage of the electrodes. By continuing this sequence at a feedback rate of 1,000 Hz, the sample can maintain a fixed position. The position control system used in this study is similar to that reported in this literature [7], though the optical paths for position sensing are modified because of the constraint of X-ray scattering facility. The He–Ne lasers and the position detectors are located on the top plate, and therefore, the optical paths of the lasers are bent twice by mirrors. This optical configuration offers a wide observation view of the sample as well as the miniaturization of the chamber. These enable us to set up the experimental configuration easier at the synchrotron radiation facility.

1.3 Experimental

Electrostatic levitation is, in principle, applicable to a wide variety of materials because all charged materials can be levitated by the action of electrostatic forces. For the first experiment, zirconium was selected and the structural analysis of its liquid phase was carried out by high-energy X-ray diffraction measurement at SPring-8, which is the third generation synchrotron radiation facility in Japan. Similar experiments were performed for molten silicon and alumina samples by using a laboratory X-ray source.