

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

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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 ₂ O ₃ , Al ₂ O ₃ , B, Si, CoNi, Ni	XRD, NS	[14]
	Al ₂ O ₃	IXS	[15]
	Al ₂ O ₃	XRD	[16]
	Al ₂ O ₃ , ZrO	EXAFS	[17]
	Al ₂ O ₃	XRD	[18]
	MgAl ₂ O ₄ , CaAl ₂ O ₄ , Al ₂ O ₃	XRD, NS, IXS	[19]
	BaB ₂ O ₃	XRD	[20]
	Mg ₂ SiO ₄ , CuZr	XRD	[21]
	Y ₂ O ₃	XRD, NS	[22]
	Y ₂ O 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:

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

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 facilities [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 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 ($Q \sim 11.5 \text{ \AA}^{-1}$) can be obtained even for laboratory X-ray source (Mo K α). 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 CO₂ 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.

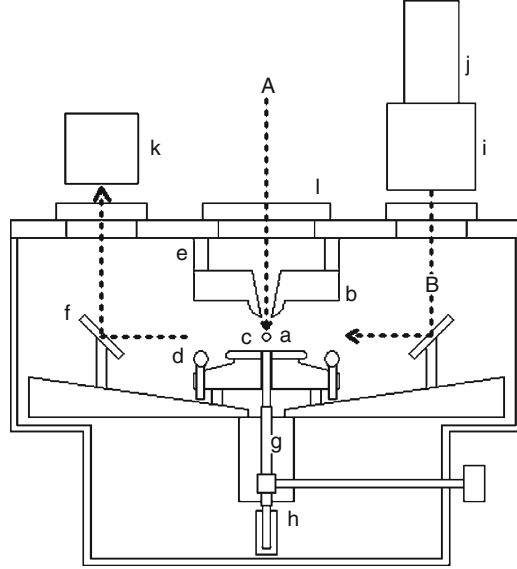


Fig. 1.1. Side view of the chamber for electrostatic levitation. *a*, levitated sample; *b*, upper electrode; *c*, lower electrode; *d*, side electrodes; *e*, ceramic support; *f*, mirrors; *g*, positioning rod; *h*, solenoid; *i*, beam expander; *j*, He-Ne laser; *k*, position detector; *l*, ZnSe window; *A*, beam path of heating CO₂ laser; *B*, beam path of positioning He-Ne laser

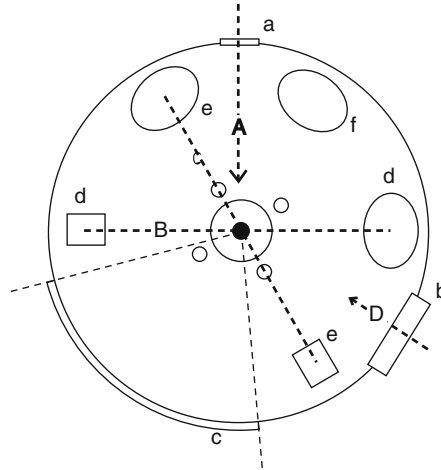


Fig. 1.2. Top view of the chamber for electrostatic levitation. *a*, sapphire window for the incident X-ray beam; *b*, glass window for the pyrometer; *c*, beryllium window for diffracted X-rays; *d*, mirrors for the He-Ne laser for positioning in *X-Z* directions; *e*, mirrors for the He-Ne laser for 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 CO₂ 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.

For the present experiments, the typical sample size was about 2 mm in diameter. Spherical zirconium samples were prepared in the following manner: 99.5 mass% pure zirconium wire was cut into 30–32 mg pieces; these pieces were melted with a diode laser (wavelength, 808 nm; max. power, 200 W) in a glove box filled with purified argon; the melted pieces took a spherical shape spontaneously because of the surface tension. Spherical samples of silicon and alumina were made similarly.

Heating is the most delicate task on the electrostatic levitation of sample since the sample charge has a tendency to decrease due to the evaporation of absorbed gas or metallic oxides from its surface. In particular, heating of the sample from room temperature must be done carefully because the discharge of sample starts at a temperature of about 800 K. However, the charge can be increased through electronic emission if the sample reaches a temperature at which thermionic emission dominates ($\sim 1,500$ K in the case of metals). To overcome these difficulties, the “hot launch” method [48] was used. To initiate levitation, the sample was heated for the removal of surface oxides. When it reached $\sim 1,500$ K, at which thermionic emission dominates, high voltage was applied to the electrodes and the feedback control system was activated. Once levitated, the sample could then be brought to temperatures beyond the melting point or be maintained under undercooled conditions for hours. The aforementioned method was adopted for the levitation of liquid zirconium and alumina. However, low melting point materials (e.g., silicon) have a tendency to stick to the positioning rod and, therefore, the heated sample must be tossed while heating. In the present apparatus, a small solenoid, which created vibrations, was fixed at the lower part of the positioning rod. The solenoid was activated remotely during the monitoring of the sample temperature.

The temperature of the sample was measured with the use of a single-color pyrometer. The emissivity of the sample is necessary to obtain the exact value of the temperature. However, the emissivity strongly depends on the sample condition, the sample size, the focus of collimation lens of the pyrometer, and the transparency of the window of the chamber. In the present research, the emissivity calibrated referred to the melting points of zirconium and silicon. The undercooled liquid state was established simply by decreasing the laser power. After a certain depth of undercooling was reached, the sudden recalescence of the sample, due to the release of the latent heat of fusion, was observed on solidification. Therefore, the undercooled liquid state was confirmed by monitoring the signal of the pyrometer. The laser power was controlled to keep the temperature of the sample constant.

The high-accuracy measurement of the liquid structure is one of the major purposes of this research. The two-axis diffractometer is the most typical instrument used nowadays for the diffraction experiments. The X-ray source was selected by considering the absorption coefficient of the material. For zirconium, which has a high absorption coefficient, high-energy X-rays (113 keV) from the BL04B2 of SPring-8 were used. For silicon and alumina, the laboratory X-rays from a Mo rotation target were sufficient to carry out preliminary

diffraction experiments. The size of the incident beam was 0.7 mm in width and 2.0 mm in height for the synchrotron radiation X-rays, and $3 \times 3 \text{ mm}^2$ for the laboratory X-rays. The incident beam was collimated by the slit and delivered to the vacuum chamber through a sapphire window. The angular dependence of the intensity of the X-rays diffracted from the sample was measured in transmission geometry by a germanium detector or a proportional counter with a graphite monochromator. Slit collimation eliminated the scattering from the windows on the chamber. This configuration is very helpful in performing the precise measurements of the diffraction only from the sample. The intensity of diffracted X-rays was acquired in each diffraction angle by the step scan method. The diffraction data was collected over a 2θ range of 0.3° – 25° for measurements by synchrotron radiation and 0.5° – 80° for measurements by laboratory X-ray one. The obtained Q range of $S(Q)$ was 0.3 – $24.7 \text{ \AA}^{-1}$ in the former case and 0.08 – $11.4 \text{ \AA}^{-1}$ for the latter case. The duration of acquisition of data for each diffraction angle was greater than 5 s, which is sufficiently long with high statistics. To obtain the static structure factor of liquids, data correction of the absorption, background, polarization, and multiple scattering must be performed [1–3]. For the laboratory experiments, the cross-sectional area of the sample is smaller than that of the X-ray beam, and the correction of absorption and polarization can be performed by normal methods [2]. Though the width of incident X-rays (0.7 mm) is narrower than the sample diameter (2 mm) in the synchrotron radiation experiments, the influence of total angular dependence of the absorption and geometric factor was negligibly small, because diffraction experiments could be performed with rather small scattering angles for high-energy X-ray experiments. In addition, it is worth mentioning that the absorption coefficient itself is very small for high-energy X-rays (mass absorption coefficient is $0.673 \text{ cm}^2 \text{ g}^{-1}$ for zirconium [49]). This implies that the contribution of absorption correction is extremely small. The contribution of multiple scattering has been mentioned for the structural analysis of disordered matter not only for the neutron scattering experiments but also for X-ray diffraction measurements. In the present study, the sample was spherical and quite small. We evaluated the contribution of double scattering compared with that of the single scattering by the method reported by Warren [1]. The ratio of the double scattering to the single scattering in the present case was less than 1%, and therefore, the contribution was neglected.

After the correction of the absorption [49], background, and multiple scattering, the contribution of Compton scattering [50] was subtracted and then the X-ray static structure factor [51], $S(Q)$, was derived from the corrected coherent intensity, $I(Q)$, based on the equation

$$I(Q) = \left| \langle f(Q) \rangle \right|^2 [S(Q) - 1] + \langle |f(Q)|^2 \rangle, \quad (1.2)$$

where the angular brackets represent averages over all atoms and $f(Q)$ is the atomic form factor [52].

1.4 Results and Discussion

The zirconium, silicon, and alumina samples were representative materials for metals, semiconductors, and ceramics, respectively. All these samples could be levitated successfully in their molten states and could be maintained at a fixed temperature for more than 1 h, which was sufficient for the measurement of X-ray diffraction. The fluctuation of the sample position was less than 0.1 mm for all materials during measurements. The diffracted X-rays were counted for 5 s for each diffraction angle. In addition, in the case of the laboratory X-ray facility, the area of the incident X-ray beam ($3 \times 3 \text{ mm}^2$) was much larger than the cross-sectional area of the sample. Therefore, the sample fluctuation did not affect the diffraction data. The diffraction patterns for all three materials are shown in Figs. 1.3–1.5. As can be seen in Figs. 1.3 and 1.4, diffraction from the empty chamber (background) was almost negligible at $Q > 1 \text{ \AA}^{-1}$. This was attributed to the use of vacuum and proper shields to the detector.

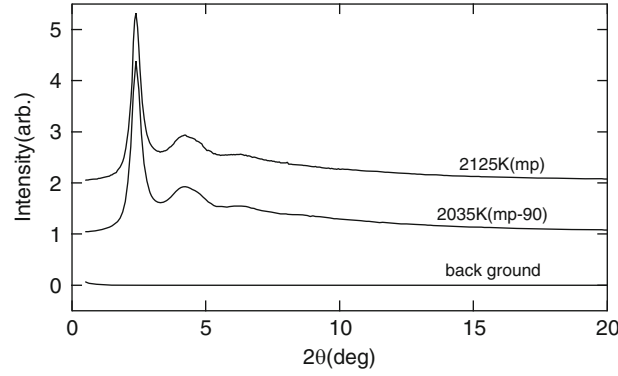


Fig. 1.3. High-energy synchrotron X-ray diffraction pattern of liquid and under-cooled zirconium and background

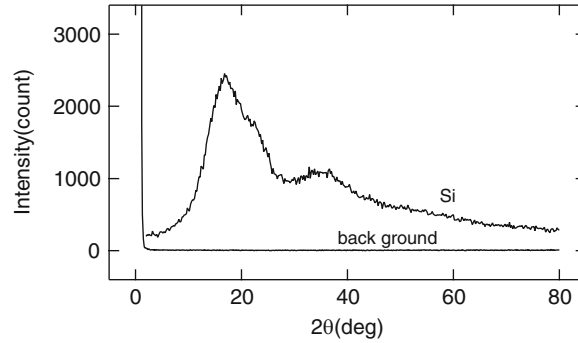


Fig. 1.4. X-ray diffraction pattern of liquid silicon at the melting point and background

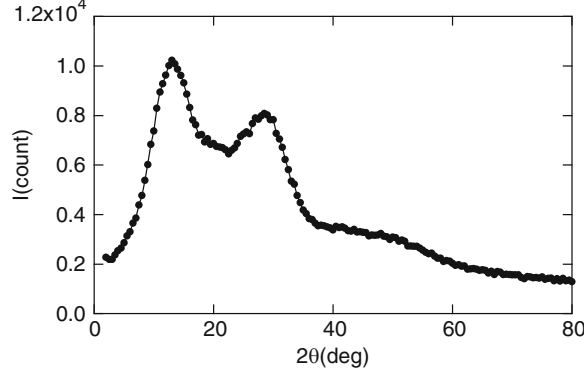


Fig. 1.5. X-ray diffraction pattern of liquid alumina at the melting point

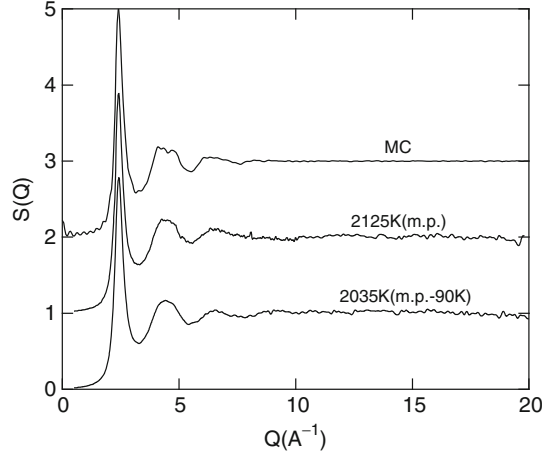


Fig. 1.6. Static structure factor of normal and undercooled liquid zirconium obtained from high-energy X-ray diffraction experiment and Monte Carlo simulation (2,125 K); normal, melting point; undercooled, the temperature of 90K below than the melting temperature

This small error from the background correction in the present experiments is remarkably different from the case of conical nozzle levitation. Thus the combination of containerless conditions and high vacuum enabled us to obtain the very reliable liquid structure.

The static structure factor $S(Q)$ of liquid samples can be obtained from the diffraction intensity. The static structure factors shown in Figs. 1.6–1.8 demonstrate that we have succeeded in performing precise observations of the liquid structure with the present electrostatic levitation apparatus, not only for synchrotron radiation X-rays but also for laboratory X-ray source. The liquid structures of the materials investigated in this study have been measured

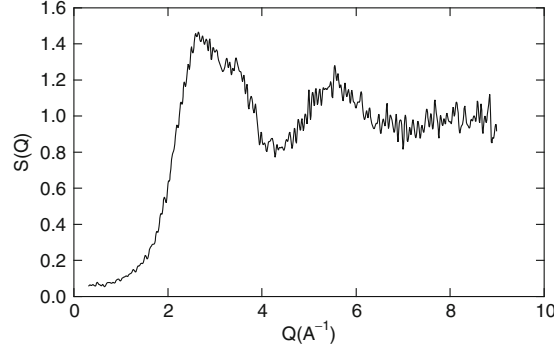


Fig. 1.7. Static structure factor of liquid silicon at the melting point

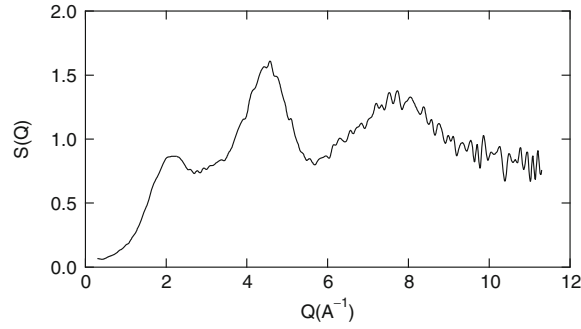


Fig. 1.8. Static structure factor of liquid alumina at the melting point

by means of other types of levitators coupled with neutron or X-ray scattering. Our experimental results are in good agreement with those previously published (Zr, [29, 31]; Si, [26–28, 39]; Al_2O_3 , [16, 18]). However, the quality of data, such as the Q range of $S(Q)$ and low background, was significantly improved. For example, the $S(Q)$ of liquid zirconium was observed in the range of $Q = 0.5\text{--}20.0\text{ \AA}^{-1}$, which was wider than that of a previous research [44]. To demonstrate the quality of the obtained $S(Q)$, the effective pair potential, $u_{\text{eff}}(r)$, was deduced for liquid zirconium based on the Modified Hypernetted Chain approximation [53] as follows:

$$u_{\text{eff}}(r)/k_{\text{B}}T = g(r) - 1 - c(r) - \ln g(r) + B_{\text{HS}}(r, \eta), \quad (1.3)$$

where $c(r)$ is the direct correlation function, k_{B} is the Boltzmann constant, and T is the temperature. The $c(r)$ was calculated from $S(Q)$ as follows:

$$c(r) = \frac{1}{2\pi\rho r} \int \left(1 - \frac{1}{S(Q)}\right) Q \exp(-iQ \cdot r) dQ \quad (1.4)$$

The reliability of $S(Q)$ in the small Q region is quite important for this calculation because the Fourier transform of the term, $1/S(Q)$, must be calculated.

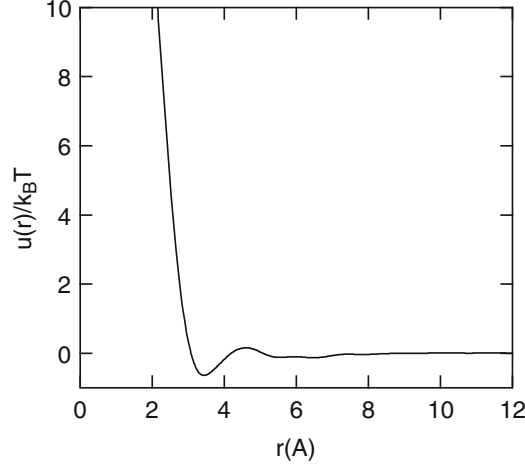


Fig. 1.9. Effective pair potential of liquid zirconium (2,125 K) based on modified hypernetted chain approximation

In addition, high- Q data are necessary to reduce the truncation error in the Fourier transform of $S(Q)$ on the evaluation of $g(r)$ and $c(r)$, which is necessary for the precise determination of the repulsive part of $u_{\text{eff}}(r)$, as shown in (1.3). The $B_{\text{HS}}(r, \eta)$ is the bridge function of the hard sphere fluid and η is the packing fraction. For the conventional estimation of B_{HS} , the η of liquid zirconium was taken as 0.46, which is generally adopted for the packing fraction at the melting point of liquid metals [54]. The $u_{\text{eff}}(r)$ obtained is shown in Fig. 1.9. The validity of obtained $u_{\text{eff}}(r)$ was investigated by a Monte Carlo (MC) simulation [55, 56], in which the obtained $u_{\text{eff}}(r)$ was employed. The temperature of MC was 2,125 K. The $S(Q)$ derived from MC is in good agreement with the experimental results, as can be seen in Fig. 1.6. The obtained $u_{\text{eff}}(r)$ is widely applicable to the evaluation of not only the static properties but also the dynamic properties. For example, transport properties, such as self-diffusion and viscosity coefficients, can be estimated from the combination of $u_{\text{eff}}(r)$ and molecular dynamics (MD) simulation. The viscosity coefficient of liquid zirconium has been already measured by the oscillation drop method coupled with the electrostatic levitator [57]. The detail analysis for the viscosity coefficients by the MD simulation by using this $u_{\text{eff}}(r)$ is in progress.

The evaluation $S(Q)$ of liquid silicon was performed for the laboratory X-ray source. The Q range of $S(Q)$ was rather small for the calculation of $g(r)$ by the Fourier transform shown in (1.1). Nevertheless, the present $S(Q)$ for liquid silicon was sufficiently good as the structure data. For example, the $g(r)$ of liquid silicon was calculated by introducing the present $S(Q)$ into the Reverse Monte Carlo (RMC) simulation [58]. The obtained coordination number of nearest neighbors was 5.9, which agrees well with previous research [35].

The present $S(Q)$ of liquid alumina shows a better agreement with the results of molecular dynamics simulation [59] compared with the experimental $S(Q)$ reported by Krishnan et al. [59]. This can be explained by the fact that the reliability of our $S(Q)$ in the low Q region is much better than the experiment reported in [59]. It was previously reported that the structure of liquid alumina depends on the ambient atmospheric oxygen concentration [59]. No such behavior was found in our data. Furthermore, our preliminary high-energy X-ray diffraction experiments using a conical nozzle did not show such a behavior. This may suggest that the structural difference of liquid alumina in oxidizing and reducing condition is still open to question. Diffraction experiments on liquid silicon and alumina by synchrotron radiation X-ray source are being planned in a near future.

It is desired that different types of materials can be successfully studied by using a single apparatus. For the diffraction experiments, this feature is very advantageous because a common optical set-up and background calibrations can be used. We developed such an electrostatic levitator which is applicable to a wide variety of materials and X-ray sources. Furthermore, we confirmed that the quality of structure data for high-temperature liquids by the present apparatus is much better than that in previously published literatures. We believe that we present a reliable apparatus for the structure study of high-temperature liquids and undercooled ones. The present apparatus will be able to perform the experimental analysis of high-temperature melts with high precision and will contribute to the fundamental understanding of the essential feature of liquids in normal and undercooled states.

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