Furnace for Inelastic X-Ray Scattering from Liquids up to 1600 °C

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The design and implementation of a furnace for inelastic X-ray scattering from liquids with sample temperatures up to ∼1600 °C is described. Carbon composite heating elements operating in vacuum provide robust heating elements: one pair of heaters has been used for >18 days of operational time above 1500 °C, including eight cycles to room temperature. High-quality data are obtained to scattering angles as low as 7 mrad in 2θ (Q < 1 nm⁻¹ at 25.7 keV) from a sample at 1560 °C.

1. Introduction

The behavior of liquids on atomic length scales is an area of current scientific investigation. In particular, with the advent of practical inelastic X-ray scattering (IXS) setups at modern synchrotron radiation facilities which are not subject to the kinematic limitations of neutron scattering, experimental investigation of angstrom- and nm-scale dynamics of liquids has greatly expanded (see, e.g., discussion in the review [1]). However, many materials melt only at relatively high temperature (relevant references about liquid iron, which melts at 1538 °C, and will be shown as an example below, include refs. [2–6]) so, to extend the range of samples investigated, an IXS-compatible furnace is needed. Furthermore, this furnace must be stable on ≈day time scales (or longer) as, even at modern X-ray sources, count rates in IXS experiments are often low, so that spectra may be measured for ≈24 h. Finally, as it is highly desirable to extend IXS investigations to small scattering angles (long length scales), where signal rates can be very low, the background from the heating system (windows, cell materials) must be low, especially at small angles. Such a system, in principle, may also be interesting for X-ray small- and wide-angle scattering measurements to investigate, e.g., isothermal compressibility, or liquid–liquid phase changes, or other topics of current interest in disordered materials at high temperature.

Herein, we describe a furnace optimized for use at the high (meV)-resolution IXS spectrometer of BL43LXU [7,8] of the RIKEN SPring-8 Center. The furnace has been operating for about 2 years over >6 experimental runs. We find it to be reliable and straightforward to operate up to ∼1600 °C. This furnace was made after difficulties with another system [9] made it impossible to collect comprehensive IXS data sets: the heaters tended to fail on ≈hour time scales for higher temperatures (T > ∼1300 °C). The older system had performed better previously [10] but did not recover despite multiple weeks of work. The new system has three notable changes compared with that in ref. [9]: 1) the use of carbon composite heating elements which are robust and allow repeated operation at high (≥1600 °C) temperature for multiday runs, 2) a design that uses contact cooling initiated by thermal expansion, and 3) operation in vacuum. Additional details are discussed in the following paragraphs, including the overall concept, material and heating element choices, practical details for using thermocouples, and the performance.

2. Container versus Containerless Sample Support

An important distinction for high-temperature work is between a containerless sample environment and a container-based one. Containerless setups are possible using levitation via gas flow or electrostatic methods. They have the advantage that the sample is intrinsically thermally isolated from nearby solid materials, so that one does not have to worry about, e.g., exceeding the melting point of the surrounding material, thus allowing extreme (>3000 °C) temperatures with laser heating. Containerless setups also can facilitate supercooling of liquids. However, levitation usually requires mm-scale (e.g., ∼2 mm diameter) samples for the control/feedback on the sample position.
This means that while such methods work well for relatively low-Z/light materials such as alumina, where X-ray attenuation lengths are $\approx 0.01 \text{ mm}$ so the experiment is done in transmission, they have not been used for high-Z/heavy materials, where the attenuation length of the $\approx 20 \text{ keV}$ X-rays used for most IXS work is on the $0.01$–$0.1 \text{ mm}$ scale. There has been at least one case where levitation was used with a reflection geometry (liquid Ti, attenuation length $\approx 0.2 \text{ mm}$). If the position control is sufficiently good, such a geometry might also be used for heavier materials with shorter attenuation lengths, though, even then, measurements at small scattering angles may be tricky (the previous work was entirely above $5 \text{ nm}^{-1}$). However, in our experience, the position of levitated samples can fluctuate at the level of a few $0.01$, so a reflection geometry may be difficult. Further, as a levitated sample is not in a sealed environment, there can be issues with sample evaporation (e.g., the lifetime of a $2 \text{ mm}$ diameter sphere of liquid silicon electrostatically levitated in vacuum was only a few hours).

Container-based methods allow flexibility to tailor the sample thickness down to the level of $0.1$ or even, with care, to $\approx 0.01 \text{ mm}$. Further, the containers may be sealed so that sample evaporation is not an issue. In addition, given an appropriate chamber, they can be operated at relatively high pressures ($\approx \text{kbar}$) as is interesting to explore the liquid-phase diagram, though high pressure is not the goal in the present work. However, the melting point of the container material will limit the maximum achievable temperature. Also, scattering from the container can create background (as is also an issue for gas flow levitation), so that background subtraction is often needed, and the container material must be chosen with care to avoid the background dominating the signal. The background can be particularly problematic when data at small scattering angles are desired. Single crystal sample containers (or at least single crystal windows) are often preferable as they have less small angle scattering than other (polycrystalline or glassy) materials, and will have well-defined phonon modes that can often be subtracted from data relatively easily.

The work presented here relies on the single crystal sapphire cell technology developed by Tamura, Inui and Hosokawa at Hiroshima University. The single crystal material means that the small angle scattering background from the container can be reduced, whereas the relative hardness of the sapphire means the inelastic (phonon) background from the cell will tend, at least for small momentum transfers, to be at high energy transfers so is often out of the sample window of interest for liquids. The long shape of these sapphire cells with the sample position about $25 \text{ mm}$ from where the cell is sealed allows the sample temperature to be different than the temperature of the seal. This means that 1) it is possible to seal the cell (melting a glass at $\approx 1200 \text{ °C}$ without heating the sample too much and 2) when the cell is used the sample can be heated to high temperature in the interior of the furnace, whereas the sealing, far from the center of the furnace, remains relatively cool and does not melt, preserving the integrity of the cell. While the use of the sapphire does limit the maximum temperature (the melting point of sapphire is $\approx 2040 \text{ °C}$), there are still many different materials that melt in the nominally accessible temperature range (say, $<1900 \text{ °C}$). The present furnace is designed for use with Hiroshima style sapphire cells.

### 3. Materials

Material choices are critical for proper operation of the system. Table 1 shows some of the materials used, their purpose, and approximate limits on upper temperature.

![Image](https://www.advancedsciencenews.com)

| Material       | Melting point ($T_m$) or maximum use temperature ($\text{°C}$) | Comment                                                                 |
|----------------|---------------------------------------------------------------|-------------------------------------------------------------------------|
| Copper         | $T_m = 1084$                                                 | Soft—used primarily for electrical connectors. (beryllium copper preferred for structural parts) |
| Beryllium      | $T_m \approx 850–905$ (depending on alloy)                    | Structural alternative to copper                                        |
| Copper         | $T_m \approx 500–600$ (depending on alloy)                    | Easy to machine, light weight, good thermal conductor                   |
| Molybdenum     | $T_m = 2623$                                                 | Machinable with effort, sheets (0.1 or 0.05 mm thick) readily available.|
| Tungsten       | $T_m = 3422$                                                 | Difficult to machine                                                   |
| Stainless Steel| $T_m \approx 1400–1500$ (depending on alloy)                  | Relatively poor thermal conductivity.                                  |
| Alumina        | $T_m \approx 2070$                                           | Hard, thermal insulator                                                |
| Sapphire       | $T_m = 2040$                                                 | Hard, sample cell                                                      |
| Macor          | $\approx 1000$                                               | Insulator (machinable ceramic used as electrical insulator)            |
| Hexagonal BN   | $\approx 3000$ (sublimation)                                 | Soft (can be cut with a knife but slow delivery and expensive.)        |
| Brass          | $\approx 450$ (max use)                                      | Outgasses Zn above $\approx 450 \text{ °C}$                            |
| Kaowool        | $>1000 \text{ °C}$                                          | Flexible alumina/silica packing material                               |
Swagelok connector. This unit performed well (after modification of the thermocouple feedthroughs discussed in Section 11).

5. X-Ray Windows

The outer X-ray windows for the chamber are polished single crystal sapphire, either 0.08 (incident) or 0.2 (outgoing) mm thick from Sartonworks (http://www.sartonworks.com/). These are epoxied onto aluminum frames that are attached to the chamber using o-ring seals (see Figure 2, and also Figure 1). The (1/e) attenuation length in sapphire is \( \approx 1.5 \text{ mm} \) at 21.7 keV and \( \approx 2.5 \text{ mm} \) at 25.7 keV, so 0.28 mm of sapphire will transmit approximately 84% or 89% (at 21.7 and 25.7 keV, respectively) of the radiation. The use of the epoxy was regarded as speculative for high temperatures—but seems to work well. The windows are clear so they do not absorb much radiated heat from the interior of the furnace: even when the furnace is operated near 1600 °C they remain relatively cool so one can easily touch them with bare fingers (in contrast, the heat radiated through the window is large and will eventually burn a finger placed near the window). In operation, a layer of Al foil (12.5 μm thick) is placed downstream of the outgoing window to insure that the thermal radiation exiting the chamber through the window does not heat the receiving Soller slit assembly (cf. Figure 6). Also, at higher temperature, we usually place a fan blowing on the window area to help keep the foil (and window) cool.

6. Heating Elements

After difficulties with short lifetimes of metal wire (W or Mo) heating elements, an alternative approach was desired. Options considered included carbon or carbon composite heaters as are compatible with running in vacuum or high-purity inert gas atmospheres, or MoSi₂-based heaters that are compatible with oxygen-containing atmospheres. Notably, while robust in an oxygen-containing atmosphere, reportedly, MoSi₂ heaters can degrade after extended use in vacuum. Therefore, resistive carbon-based heaters were chosen. Graphite heaters appeared to have an advantage in that they could easily be fabricated in 3D shapes tailored to specific applications. However, most designs tended to have rather low resistances (<1 Ω) so would need large current to achieve high power. It was also not clear how to attach the current leads so they would neither damage the graphite heater nor melt during use. Meanwhile, carbon composite heaters, essentially cut out of rigid sheet material, seemed to allow a simpler design, with modularly replaceable heater elements that were, in principle, stable to >2500 °C. Also, while significantly dependent on detailed design, it appeared a heater with a few (\( \approx 2-5 \)) ohms total resistance would be possible, relaxing the requirements on the current supply, and the current feed-through. Carbon composite heating elements were initially purchased from Thermic Edge (https://www.thermic-edge.com/heaters/) and then later from Thermocera (http://www.thermocera.com/). One heater plate is shown in Figure 3: the lower end of the heater, with the narrower, higher resistance, path, faces the Mo sample sleeve (see below) with one heater placed on each side of the sleeve (see Figure 4 below). The nominal resistivity of the material is \( \approx 0.0016 \text{ Ω} \cdot \text{cm at 1000 °C} \).
The measured resistance of two heater plates (two plates of the type shown in Figure 3) used in series was \( \frac{1}{25} \Omega \) at room temperature dropping to \( \frac{1}{2} \Omega \) at 1600 °C sample temperature—with most of the resistivity change occurring below 400 °C.

7. Thermocouples and Thermocouple Feedthroughs

Type C thermocouples of W/Re were used (W/5%Re–W/26%Re) as they can be operated to temperatures in excess of 2200 °C. These were either bought preassembled (e.g., from Omega), or, more typically, fabricated directly from 0.25 mm diameter wire bought from Ishikawa-Sangyo (http://www.ishikawasangyo.co.jp/) that was twisted and spot welded. When required, tubular ceramic insulators were used for the thermocouple wire (see Figure 4 and 5). Thermocouple feedthroughs (part number 9313006 from MDC, https://www.mdcvacuum.com/) required some modifications (Section 11) but then performed well. Usually two thermocouples are used at one time, placed on opposite sides of the Mo sleeve just outside the sample: one of these is used for temperature feedback and the other is used for additional monitoring. Sometimes an additional thermocouple is also mounted on the sample cage to monitor its temperature.

8. Temperature Control and Power Supplies

We use Yokogawa UT35A temperature controllers (https://www.yokogawa.com/solutions/products-platforms/control-system/controllers-indicators/temperature-controllers/). These were chosen because they were ethernet and socket I/O compatible, and had the required range of functionality and compatibility with type C thermocouples. They performed well after some initial time investment to learn the format of the ethernet commands. A notable positive point was a convenient limit on the power used for auto-tuning, making them easy to auto-tune even at high temperatures. The output from the controller (0–20 mA over a 500 \( \Omega \) resistor) was used to control the ITECH 6523D (0–200 V, 0–60 A, 3 kW, https://www.itechate.com/en/index_128.aspx) direct current (DC) power supply via its analogue (0–10 V) input. DC supplies were chosen to reduce noise: another chamber with alternating current (AC) power supplies (and grounding through the chamber body) was known to occasionally introduce noise in the IXS detectors, whereas there has been no evidence of noise with the DC power supplies used here.

9. Sample/Heater Cage Design

The heater cage is shown in Figure 4 and Figure 5. A cylindrical tube of beryllium copper is used as the main outer support. The
inner piece is a hollow rectangular BN block which is open at the top and bottom and has holes for the cylindrical molybdenum sample sleeve (see Figure 4). The Mo sample sleeve design is described in the work of Tamura et al.\textsuperscript{[14]} Two carbon composite heater plates are supported from above and are located on either side of the sample facing the sleeve. Outside the BN block, two pieces of alumina are used as insulators, with 50 μm-thick molybdenum foils placed between the BN and the alumina to act as a reflector (one such Mo foil can be seen in the left panel of Figure 4).

The sample space is covered above and below by disks of BN (inner layer) which are then covered by disks of alumina (BN and alumina parts were fabricated by Krosaki, https://www.krosaki.co.jp/). The upper disks have slots for the heater elements and holes for the thermocouples.

Cooling is provided through four copper “fins” which, with thermal expansion, contact the water-cooled sides of the chamber (the four fins are shown in Figure 4 and 5). Considering the coefficient of thermal expansion of the copper beryllium to be about 17 ppm K\(^{-1}\) and the outer diameter of 75 mm, one expects an expansion of about 130 μm in diameter per 100 °C of temperature increase in the cylinder assembly. In fact, copper fins were carefully thinned by hand so they are a tight fit against the cooled chamber walls—with ≈50 μm of clearance on each side. In operation, one finds that with the sample position at 1600 °C, the temperature of the copper cylinder is about ≈170 °C, so this cooling works well.

The assembled heater cage is shown in Figure 5 just before insertion into the water-cooled vacuum chamber of Figure 1. The horizontal slot for the scattered X-rays is visible in the center, partially covered by a 50 μm thick Mo foil over the alumina insulator. The foil is held in place on the cage by the two copper wires. The top of the heater assembly is visible above the cage, with the two cylindrical current leads visible at the very top of the figure.

The tops of the two thermocouples (in ceramic insulators) are also visible at the upper right of the cylinder. Threaded rods are used to adjust the position (height) of the cylinder in the chamber to match that of the chamber windows.

10. Performance

The performance of the system has been relatively robust. Figure 6 shows the system mounted in the spectrometer. Operating in vacuum, one set of heaters has been used for multiple runs at temperatures of >≈1500 °C at the sample: the same heaters and thermocouples now have been used for a total of >18 days in these operating conditions (eight cycles from room temperature to >1500 °C and back) and continue to perform well. The pressure of the system measured at the pump cart (a system with a small, 50 L s\(^{-1}\), turbo pump) typically is <5 × 10\(^{-3}\) Pa during the runs at 1500 °C, indicating the vacuum level is fairly good. The two thermocouples placed in the Mo sample sleeve read the same within about 25 °C (worst case) near 1500 °C, with a corresponding estimated thermal gradient over the illuminated sample volume of <1 K. We typically calibrate the sample temperature by observing the melting point of the sample (e.g., pure iron melts at 1538 °C) using the change in the X-ray transmission. The transmission change is easy to observe on the first melting of a new sample. It is more difficult to see when remelting a sample that has already been melted (transmission changes on remelting are sometimes only ≈0.2%) but usually can be observed using high-rate detectors. Doing so shows that the sample temperature is typically about 50 °C lower than the average thermocouple temperature. This difference is

![Figure 5. Sample cage mounted on the flange ready for insertion in the vacuum chamber. The left panel shows just the cage and leads, whereas the right panel shows also the upper part of the flange including 4 current feedthroughs, one feedthrough for a pair of thermocouples, and the pressure gauge (see text for discussion).](image)

![Figure 6. Furnace in use for IXS at BL43. The X-ray beam is incident from the right. Thermal radiation from the heater is visible coming through the windows. A piece of Al foil on the downstream side acts as a reflector to keep from heating the receiving Soller slit assembly. A cleanup slit and flux monitor for the incident beam are visible on the right side of the figure.](image)
reasonable given that the design of the sample holder allows some additional cooling for the sample (part of the sapphire sample cell—where it is sealed—extends well out of the molybdenum sleeve so remains relatively cool). When the heaters are turned off (during cooling), the two thermocouples read the same to within about 5°C—suggesting the thermocouples are well matched and the difference in temperature observed at higher temperature is due to a real thermal gradient in the molybdenum sleeve. However, as this difference has not been worse than 25°C (and in fact is often <10°C) in our measurement conditions, this is not considered serious. It probably originates from a slight difference in the performance of the two heaters or a slight asymmetry in their position relative to the sleeve.

Typical temperature and power curves are shown in Figure 7. The system is usually first heated to ≈400°C using a constant (but manually adjusted) voltage on the power supply. Then the system is ramped up to ≈100°C below the sample melting point at a rate of ≈20°C min⁻¹. The heating is interrupted every 300–500°C to retune the system feedback parameters. Then the sample is usually slowly heated through the melting point (2–5°C min⁻¹) while observing the transmission. The melting point, as is usually observed by a sharp change in transmission, then becomes the reference for working at higher temperature. Cooling down usually occurs at 25–40°C/min⁻¹ to ≈600°C. At that point, the heaters usually have near zero current, and a small amount of high purity He is introduced in the chamber to speed cooling. The control of the chamber is fairly easy above ≈700°C, but, below that, limited cooling for the system in vacuum slows the response time. (If temperatures <≈700°C are needed, operation in a low pressure He atmosphere is probably straightforward.)

The operation of the system is mostly automatic. It is usually monitored during the initial heating phase, and during the determination of the sample melting point, but then the controller is set at the desired operating point, and the furnace control is not touched over typical run times of >24 h. During that period, the temperature at the control sensor usually remains very stable (within ±0.3 K of the set point), whereas the drift of the second sensor varies—in a good run the full range of this drift is <2 K, and, in the worst run to date, the full range was <10 K. Sometimes, we also see jumps in both sensors (1–2 K in magnitude), that are quickly corrected by the control system. Thus, we take the real sample temperature to be stable to ±5 K, or better, over ≈24 h time scales.

An IXS spectrum taken with this furnace is shown in Figure 8 from a sample of liquid iron (≈0.1 mm thick) at T ≈ 1560°C (about 22° above the melting point) in a Hiroshima style sapphire cell[14] and measured using a Soller slit and specially designed analyzer masks.[15] This is an interesting sample for discussion about the nature and interpretation of the dynamics of liquids[2,3,5,6] and from the viewpoint of being a starting point to understand geological issues.[16] The red points show the background (scaled by the sample transmission) and the blue points the measured scattering from the sample—the quasielastic and phonon contributions from the liquid iron are clearly visible (measurement at 25.7 keV with 0.75 meV energy resolution).

11. Comments for Future Research and Development

The furnace has performed well within the boundaries described earlier. However, through the process of constructing the furnace and making it work, some experience was gained that may be useful to others considering such a system in the future. This includes issues with thermocouple feedthroughs and also some issues related to going to higher temperature. The thermocouple feedthroughs caused some difficulties. While their vacuum performance was good, the feedthroughs are "compensated" in that the conductors are not W/Re material, but something that is designed to be compatible over a limited temperature range (this was not so obvious from the description.
on MDC’s website). These compensated wires extended far (≈15 cm) beyond the vacuum interface. While the long extent facilitates the physical connections to the thermocouple leads, it also forced the connection points to be far enough inside the chamber that they viewed the furnace heating elements, at least partially. Thus, in initial work, the interface to the compensated wire was probably heated above the nominal operating limit and initial test runs with the chamber suffered from very poor and nonreproducible thermal control. After this issue was recognized, the long interior compensated leads were cut very close to the vacuum feedthrough and replaced by proper type C (W/Re) wires that were spot welded to the feedthrough. The interface between the compensated wire and the proper wire was then much further from (and out of view of) the heating elements. After doing this, the temperature control and reproducibility improved to the level discussed earlier.

With respect to attaining higher temperature, we note that during our first online usage of the chamber, we attempted to go to 1900 °C at the sample, and this led to the heaters failing in about 4 h (they were replaced), even though they had been very stable at ≈1600 °C. This has not been explored further, as, so far, the samples of interest have melted below 1600 °C. Also, using a slightly different design (with three heater plates), we melted the molybdenum sample sleeve (T_m = 2623 °C) during offline tests (this over-power event was probably the result of using the compensated thermocouple feedthrough in its original configuration). Thus it appears the carbon composite heating elements may indeed function over 2000 °C, but achieving >1600 °C required some additional work.

We note that at higher sample temperatures the current feedthroughs become warm. The flange is cooled, but, apparently, more cooling at the feedthrough might be good. The source of the heat is not the direct resistive heating by the current flowing through the feedthroughs, but the heat conducted from the heater region inside the chamber back to the feedthrough: a low-resistance current path is usually also a low-resistance heat path. The temperature of the feedthrough was reduced by putting a small section of W wire (0.6 mm diameter, x ≈ 10 mm long) between the feedthrough and the main heater body, increasing the thermal isolation of the feedthrough from the heater. At a sample temperature of ≈1600 °C, the feedthroughs are then warm to touch. This heating might become an issue for operation at extended times at temperatures >1600 °C.

12. Conclusions

This article has described the design and operation of a furnace for IXS that operates robustly up to temperatures of 1600 °C. A pair of carbon composite heating elements have, as of the time of the writing of this article, survived >8 heating cycles (room temperature to >1500 °C) with an integrated time at T > 1500 °C of more than 18 days. The use of Hiroshima-type single crystal sapphire cells along with single crystal windows and a careful IXS setup, allows measurements to be conducted even to sub-inverse-nm momentum transfers at high temperature.

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Conflict of Interest

The authors declare no conflict of interest.

Keywords

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