Six-axis multi-anvil press for high-pressure, high-temperature neutron diffraction experiments

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Six-axis multi-anvil press for high-pressure, high-temperature neutron diffraction experiments

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We developed a six-axis multi-anvil press, ATSUHIME, for high-pressure and high-temperature in situ time-of-flight neutron powder diffraction experiments. The press has six orthogonally oriented hydraulic rams that operate individually to compress a cubic sample assembly. Experiments indicate that the press can generate pressures up to 9.3 GPa and temperatures up to 2000 K using a 6-6-type cell assembly, with available sample volume of about 50 mm³. Using a 6-8-type cell assembly, the available conditions expand to 16 GPa and 1273 K. Because the six-axis press has no guide blocks, there is sufficient space around the sample to use the aperture for diffraction and place an incident slit, radial collimators, and a neutron imaging camera close to the sample. Combination of the six-axis press and the collimation devices realized high-quality diffraction pattern with no contamination from the heater or the sample container surrounding the sample. This press constitutes a new tool for using neutron diffraction to study the structures of crystals and liquids under high pressures and temperatures. © 2014 AIP Publishing LLC. [http://dx.doi.org/10.1063/1.4901095]

I. INTRODUCTION

The structure of crystals and amorphous materials are important for understanding their physical properties. Determining structures requires in situ observation because the variation under high pressure and high temperature is often unquerchable to ambient conditions. With respect to other probes, neutron diffraction is advantageous because it can locate light elements such as hydrogen and lithium, discern elements with low contrast in X-ray scattering, determine magnetic structures, and differentiate between isotopes. These features make high-pressure neutron diffraction an attractive tool for a wide range of sciences.

Many high-pressure devices have been developed for neutron diffraction experiments. Because the intensity of neutron sources is limited, large-volume samples and wide detector windows are essential for these devices. The Paris–Edinburgh press, which satisfies these requirements, has been widely employed in neutron and synchrotron facilities.1,2 A uniaxial load generates pressure between the two opposing anvils. The press generates a wide range of pressures up to 10 GPa with single toroid tungsten carbide anvils and up to 25 GPa with double toroid sintered diamond anvils. However, improvement is possible for high-temperature experiments. A furnace assembly used in high-PT experiments (high-PT cell) typically consists of a sample, a sample container, an internal resistive heater, electrodes, and a pressure-transmitting medium. Furthermore, a ceramic pressure-transmitting medium works as a thermal and electrical insulator. To achieve temperatures over 1000 K, this internal heating system requires sufficient thermal insulation. To date, the volume of the spheroid space in a Paris–Edinburgh press to accommodate a high-PT cell is limited to roughly 90 mm³ and the routinely available temperature and pressure ranges for neutron diffraction experiments are restricted to less than 1000 K and 10 GPa, respectively.

To generate 10 GPa and 2000 K with enough volume to accommodate the high-PT cell, we developed a large-volume multi-anvil press called ATSUHIME. The concept of the multi-anvil press is to apply a load to the sample using more than four anvils. The space to accommodate the high-PT cell depends on the size of the anvil; essentially, the space is larger than that in a Paris–Edinburgh press. In typical experiments at pressures up to 10 GPa, the space of a 7 mm cube surrounded by the truncated face of the anvil is available. This feature of the multi-anvil press enables us to scale up the size of the high-PT cell while maintaining sufficient thermal insulation.

Compression of a multi-anvil press requires synchronous movement of all the anvils to generate stable high pressure. Of the existing presses, a DIA-type press, in which six first-stage anvils are fixed on guide blocks and sliding wedges, is one of the most widely employed multi-anvil presses in laboratories and synchrotron facilities.3,4 Isotropic compression is mechanically attained via a pair of guide blocks and four sliding wedges. The uniaxial load applied to the guide blocks with one hydraulic ram is divided into six mutually perpendicular forces and concentrated on a high-PT cell. The attainable pressure and temperature condition of a 6–8 type (Kawai-type) cell assembly (MA6-8)5 is reaching to 80 GPa.

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and 1500 K by using sintered diamond as the second stage anvils. Although this guide block system is a simple and excellent way to synchronize all the anvils, its bulk makes it difficult to maintain a wide window for neutron diffraction and prevents the collimator devices from being positioned close to the sample. To overcome these problems, we developed a six-axis-type multi-anvil press that compresses the sample assembly with six independent hydraulic rams without any guide blocks. To date, two types of six-axis presses have been developed that feature synchronous movement of the anvil in different ways. The first type is operated with a single hydraulic pump, and a servomotor in each ram precisely controls the anvil position. The second type is operated with six hydraulic pumps and the positions of the six anvils are controlled by individual plunger pumps. We adopted the latter setup because the servomotors in the former setup take up space and did not fit in the experimental hutch.

In this paper, we describe the design and performance of the six-axis ATSUHIME press, which is optimized for high-pressure time-of-flight (TOF) neutron diffraction measurements. This press has been installed in the high-pressure neutron beamline (PLANET) at BL11 of the spallation neutron source of the Materials and Life Science Experimental Facility (MLF) at the Japan Proton Accelerator Research Complex (J-PARC). The wide space around the sample allows us to not only collect diffraction over a wide scattering angle but also position the incident slit and radial collimators close to the sample. Combining the six-axis press and collimation devices results in a clean diffraction pattern with no contamination peaks from the surrounding high-PT cell components, which is an important factor for determining crystal structures.

II. DESIGN REQUIREMENTS

The design of the press has several requirements for achieving high pressure and temperature for neutron diffraction experiments. First, the dimensions of the press should be compatible with the shielding hutch and the detector banks of the beamline. The inner dimensions of the hutch are 4.4 m in width, 7.2 m in length, and 4.3 m in height. Helium-3 position-sensitive detectors make a pair of opposed 90° banks at 1.5 m from the sample position; therefore, the press should be less than 3.0 m wide. Additionally, the beamline provides a platform for experiments that use other high-pressure devices, such as a Paris–Edinburgh press, a Palm cubic press, or a nano-polycrystalline diamond anvil cell. To make space for experiments that use these devices, the press needs to be moved to downstream in the hutch.

The second design requirement is a wide aperture to detect neutrons scattered over a large solid angle. The detector banks cover the scattering angle of 90° ± 11.3° in the horizontal plane, and 0° ± 34.6° in the vertical plane. The body and other components of the press therefore need to be designed to leave the window for this detector coverage.

The third requirement is to have sufficient space to position the requisite devices close to the sample. Although internal heating can generate higher temperatures than external heating, neutron scattering from the internal heater is a problem. The overlap of these diffraction peaks with those of the sample complicate structure refinement. The beamline has a fine incident slit and radial collimators to eliminate scattering from the high-PT cell, and these devices become more effective when they are near the sample. The imaging camera used in radiography also needs to be close to the sample to avoid blurry images.

The final requirement is the synchronous movement of the six anvils. To reach our target PT condition of 10 GPa and 2000 K, we used two types of multi-anvil assemblies: an assembly with six second stage anvils (MA6-6) and an assembly with eight second stage anvils (MA6-8). For high-pressure experiments using MA6-6 and MA6-8, the first-stage anvils must be moved synchronously with a precision of several tens of microns.

III. APPARATUS

The six-axis multi-anvil press apparatus is composed of a main body, hydraulic pumps, alignment stages, a heating system, and a control unit. The details of each component are given below.

A. Main body

Figure 1 shows a technical drawing (a) and a photograph of the main body of the apparatus (b). The main body includes the frame, six hydraulic rams, and sensors to measure the anvil positions. The frame is built with six parts: the top and bottom blocks, and the four columns that are connected to the blocks by tie rods. The hydraulic ram is embedded in the frame, and three pairs of opposed rams are aligned orthogonally. The first stage anvils are made of tungsten carbide or tool steel and are attached on the top of each ram. The main body is 2.4 m wide, 2.4 m long, and 2.5 m high, so that the press fits with the other beamline components. The press, including the alignment stages, weighs approximately 30 tonnes.

The main body is shaped to leave space for the collimators and to maintain a wide aperture for the scattered neutrons. The radial collimators and incident slit are positioned close to the press, as shown in Fig. 2(a). The distances from the center of the sample to the front of the radial collimators and the incident slit are 160 mm and 84 mm, respectively. The vertical angle of the aperture is 0° ± 60.5° (Fig. 1(a)), which assures that the vertical detector covers 0° ± 34.6°. However, the actual horizontal aperture depends strongly on the anvil gap of the first and second stage anvils (Fig. 2(b)). For MA6-6 with TEL = 10 mm, which we usually use, the horizontal angle is almost comparable with the detector coverage of 90° ± 11.3° before compression, whereas it decreases as the cell is compressed.

The allowable maximum load for each ram is 5000 kN. The load applied to the high-PT cell is equivalent to that of a DIA-type press with a maximum load of 15 000 kN; the different maximum load value arises from the different geometry of the compression. For the compression, the effective cross-sectional area of the ram is 80 817 mm². The stroke of the ram is 110 mm, which ensures sufficient working space around the
FIG. 1. (a) Technical drawing and (b) photograph of the six-axis press. Labels: (1) Top block, (2) tie rod, (3) column of frame, (4) first stage anvil, and (5) alignment stages. The vertical aperture angle is also shown.

Sample to remove the sample assembly when all the rams are retracted to their stroke ends.

Precise measurement of the anvil positions is indispensable to achieve isotropic compression with the six-axis press because the measured position is used in a feedback control of the anvil position. Figure 3 shows the cross-section of the hydraulic ram. To minimize uncertainty in the measurement and leave space for surrounding devices, the position is measured from the backside of the anvil through the hole along the center of the ram. The sensor (Magnescale Co. Ltd. SR74-012RSGL115) is attached on the frame of the press (Fig. 3(1)), and its probe is extended with a rod so that it touches the backside of the anvil base (Fig. 3(7)). The precision of the scale is 0.05 µm and the maximum stroke is 120 mm, which is sufficient to cover the full stroke of the ram. To compress the sample at the exact center of the press, the sensor is zeroed before the experiment by following the same procedure as described in Ito et al. The press frame where the sensor is attached, however, is subject to deformation during the high-pressure experiment. Thus, the measured value is not accurate because deformation of the frame shifts the sensor position relative to the center of the press. To correct this effect, displacements of the press frame relative to a reference point (i.e., the ground) were
measured beforehand for several loads. The displacement for 4500 kN was approximately 550 µm for the four horizontal rams and 100 µm for the top and bottom rams. Two more runs were conducted to verify reproducibility. By averaging the three runs, we obtained a linear function for the anvil positions for any loads up to 5000 kN.

B. Hydraulic pumps

Two types of hydraulic pump units were used depending on the purpose. The first type is an approach pump unit with a maximum oil pressure of 14 MPa. This pump is used for quick and rough positioning of the anvil at the beginning of experiments. The second type is a plunger pump unit with a maximum oil pressure of 70 MPa and is used to apply a high load and control the anvil position precisely during experiments. Each hydraulic ram is connected to individual plunger pumps. The full stroke of the plunger is 600 mm and the oil is automatically refilled when one of the plungers reaches the stroke end. In a typical experiment, one stroke reset suffices to reach the maximum load of 5000 kN.

C. Alignment stages

To align the sample to the incident neutron beam, the main body of the apparatus is placed on alignment stages. The x, θ, and y stages are stacked from the bottom, where the x and y are the lateral and vertical directions, respectively, with regard to the incident beam, and the θ stage provides rotation around the y axis. The travel range and precision are ±10 mm and 0.1 mm for the x stage, ±1° and 0.01° for the θ stage, and ±30 mm and 0.1 mm for the y stage, respectively. The main body of the press is supported by four posts on the stages at the same level as that of the center of the press to avoid shifting the sample with respect to the neutron beam by frame deformation. Once the sample is aligned to the beam, this setup requires no sample realignment during experiments. The whole stage unit sits on a rail laid along the beam axis and the press can be shifted out of the experimental position to make room for experiments using other high-pressure devices.

D. Heating system

We installed a heating system similar to that previously developed for a DIA-type multi-anvil press. The system consists of an AC power supply with a maximum power of 3 kW, a step-down transformer, a function synthesizer, and digital multimeters. All first-stage anvils are electrically insulated from each other, and electrical power is supplied to an internal heater in the high-PT cell through the top and bottom anvils. The transformer offers three step-down ratios: 4:1, 10:1, and 40:1; the ratio appropriate to the resistance of the heater may be selected.

IV. COMPRESSION MODES AND CONTROL OF ANVIL POSITIONS

Three compression modes are available: (1) isotropic compression in which all anvil positions are synchronized, (2) uniaxial compression in which only the top and bottom rams are operated, and (3) anisotropic compression in which the three sets of the opposing rams are controlled individually. In experiments, MA6-6 or MA6-8 is used for isotropic compression, whereas an opposed anvil assembly such as a Drickamer cell is used for uniaxial compression. For anisotropic compression that applies a deviatoric stress to the sample, MA6-6 is used. The maximum allowable load for uniaxial compression is 2000 kN, whereas, for safety, the maximum deviation of the horizontal axis is limited to 500 kN for anisotropic compression.

To achieve these compression modes, all plunger pumps are computer controlled. Each ram becomes a master or a slave of the control, depending on the compression modes. Master rams are controlled on the basis of the target load or anvil displacement, whereas slave rams are controlled to follow the anvil position of the master ram. The master and slave rams and the parameters used for their control in the three experimental modes are summarized in Table I. For example, for isotropic compression, the bottom and other rams become the master and slave rams, respectively. The load of the bottom ram is changed at the rate calculated from the target load and time and the other rams are moved so that their anvil positions equal that of the bottom ram.

<table>
<thead>
<tr>
<th>Axis</th>
<th>Isotropic compression</th>
<th>Uniaxial compression</th>
<th>Anisotropic compression</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bottom</td>
<td>Master/load</td>
<td>Master/load</td>
<td>Master/anvil position</td>
</tr>
<tr>
<td>Top</td>
<td>Slave</td>
<td>Slave</td>
<td>Slave (of bottom)</td>
</tr>
<tr>
<td>1</td>
<td>Slave</td>
<td>(Not used)</td>
<td>Master/anvil position</td>
</tr>
<tr>
<td>2</td>
<td>Slave</td>
<td>(Not used)</td>
<td>Master/anvil position</td>
</tr>
<tr>
<td>3</td>
<td>Slave</td>
<td>(Not used)</td>
<td>Slave (of 1)</td>
</tr>
<tr>
<td>4</td>
<td>Slave</td>
<td>(Not used)</td>
<td>Slave (of 4)</td>
</tr>
</tbody>
</table>

FIG. 4. Variation in the differences in anvil position between the bottom and other five rams during the experiment. The target load is also shown. Each period corresponds to (1) rough approach, (2) pre-press to 60 kN, (3) compression to 4500 kN, (4) stroke reset of plunger pump, (5) hold at 4500 kN, and (6) decompression to 0 kN.
FIG. 5. Variation in anvil positions in the anisotropic compression mode. Each period corresponds to (1) pre-press to 60 kN, (2) isotropic compression to 500 kN, (3) deformation, (4) duration after deformation, and (5) decompression to 0 kN.

V. SAFETY

High-pressure experiments are sometimes accompanied by blowouts of the sample from the high-PT cell due to the fracture of the anvils or an imbalance in sealing in the gaps between the anvils. Such blowouts are a serious issue within the neutron facility because the anvils and in some cases also the sample become radioactive once they are exposed to the neutron beam. To prevent scattering these materials inside the experimental hutch, the sample assembly is sealed with covers of aluminum rings attached to the first stage anvils. The six edge-truncated rings meet and seal the assembly during the experiment. To further increase safety, the experiment and heating is automatically stopped by an interlock system if a blowout is detected.

VI. PERFORMANCE TESTS

The performance of the press was evaluated from the standpoints of (1) precision in feedback control of anvil positions, (2) degree of isotropy in compression, and (3) pressure and temperature generation.

A. Precision in feedback control

To evaluate the precision of the feedback control of the anvil positions, we isotropically compressed an edge-truncated 55 mm cube of stainless steel and examined the
deviation of the anvil positions up to 4500 kN. The first stage anvils were made of tool steel with TEL = 50 mm. Figure 4 shows the time dependence of the load and the differences in anvil positions between the master ram and bottom rams during the experiment. The positions of all slave rams follow that of the master ram within 5 µm throughout the entire load range up to 4500 kN, which suggests that the feedback control performs as desired at any load.

The anisotropic compression mode was tested by the MA6-6 with the second stage anvils of TEL = 10 mm and with a pressure-transmitting medium consisting of a 17 mm cube made of zirconia ceramic. The first stage anvil was made of tungsten carbide with TEL = 27 mm. The resulting variations in anvil position are shown in Fig. 5. The load first increased to 500 kN in isotropic compression mode (Fig. 5(2)), then anisotropic compression started (Fig. 5(3)). During anisotropic compression, the top and bottom anvils were advanced 0.6 mm, while the others were retracted 0.3 mm. Each anvil was successfully controlled as scheduled. To simultaneously measure axial and transverse strain with neutron diffraction, one of the two rams in the horizontal plane is selected as the principle axis of deformation, in the same manner as for analyzing strain in engineering materials with opposed 90° detector banks.

FIG. 9. Cross-sectional view of high-PT cell for MA6-6 with TEL = 10 mm and MA6-8 with TEL = 5 mm. (a) The cell for the high-PT generation test with MA6-6, (b) that for the high-PT-generation test with MA6-8, and (c) that for the experiment on lawsonite with MA6-6.
B. Compression isotropy

The dimensions of the cubes recovered from several loads were measured to evaluate the degree of compression isotropy. Figure 6 shows the differences between the face-to-face dimensions of the cube. Below 2500 kN load, the difference was within 25 μm and, up to 4500 kN load, the difference was within 70 μm, which is acceptable for high-pressure experiments. This result also demonstrates that the correction of the anvil stroke by the measured frame deformation works successfully.

C. Generating high pressures and temperatures with MA6-6

We tested the capability of MA6-6 to generate high pressures and temperatures. Figure 7 shows the pressure generated at ambient temperature. We used the second stage anvils of tungsten carbide with TELs of 10 and 7 mm. A cube of zirconia was used as the pressure medium because it is highly transparent to neutrons and offers good thermal insulation. The cubes had 17 and 12 mm edge lengths for the anvils with TELs of 10 and 7 mm, respectively. In both cases, the samples consisted of 4-mm-diameter NaCl pellets. Neutron diffraction patterns were collected for 10–30 min at every 100–200 kN. The pressure was estimated from the unit cell volume of NaCl on the basis of the equation of state. The maximum pressure generated was 7.5 GPa at 1400 kN with the 10-mm-TEL anvils and 9.3 GPa at 950 kN with the 7-mm-TEL anvils.

At high pressure, the actual aperture of the six-axis press depends on the anvil gap. Figure 8 shows the variation in the second stage anvil gap versus the applied load in the experiment with MA6-6 and TEL = 10 mm. Assuming negligible deformation of the anvils, the gap is calculated from the anvil position. The anvil gap, which is 4.95 mm before compression, decreases to 44% at 200 kN, and 20% at 1400 kN. The narrowing of the anvil gap results in a decrease in the counts of neutron scattering. The integrated intensity of the NaCl 220 reflection as a function of load is also presented to show the change in the intensity at high pressure. The change in the peak intensity is almost comparable with the change in the anvil gap; the peak intensity decreases to 47% at 200 kN and 16% at 1400 kN. This result implies that the exposure time required to obtain the same number of counts at 1400 kN is approximately five times that required at ambient pressure.

The heating system was tested at 3 GPa by MA6-6 with the second stage anvils with TEL = 10 mm. Figure 9(a) shows a cross-sectional view of the high-PT cell used in the test. A graphite tube was used as an internal heater and electric power was supplied through Ta electrodes. The temperature was measured with a W97%Re3%–W75%Re25% thermocouple placed at the center of the sample. A temperature of 2000 K was confirmed for an applied electric power of 520 W (Fig. 10).

D. Generating high pressures and temperatures with MA6-8

To extend the accessible PT range beyond 10 GPa, we tested the capability of MA6-8 to generate high pressures and temperatures. We used the 5-mm-TEL second stage anvils made of SiC sintered diamond cubes and with edge lengths of 15 mm for the MA6-8 assembly. Pyrophyllite gaskets were placed at the edges of the octahedron of the (Mg,Cr)O pressure medium. The cylindrical heater was made of LaCrO3 and a sample of NaCl with a diameter and height of 2.0 mm was placed in the graphite sample container (Fig. 9(b)). Scattered neutrons were detected through the second stage anvils. The exposure time at each load was 10–30 min and a pressure of 16 GPa was generated at 1600 kN (Fig. 7). The heating test was also conducted up to 1273 K and the pressure decreased to 14 GPa upon heating to 1073 K.

VII. NEUTRON DIFFRACTION EXPERIMENTS

To demonstrate the use of the six-axis press for TOF neutron diffraction measurements, we conducted an experiment on a hydrous mineral of lawsonite (CaAl1.95Fe0.05Si2O7(OD)2-D2O) using MA6-6 with the 10-mm-TEL anvils. The sample was 4 mm in height and diameter and was sandwiched between boron nitride neutron-shielding disks to reduce scattering from the parts of the container above and below the sample (Fig. 9(c)).

Figure 11 shows the diffraction pattern obtained at 4.6 GPa and 1073 K. The data were acquired over 10 h at an accelerator beam power of 200 kW. This exposure time was sufficient to obtain the required counting statistics for Rietveld refinement. Thanks to the radial collimators and incident slits, no scattering from the sample container and heater was observed and all the peaks were assigned to the sample.

This experiment emphasizes the potential usefulness of the six-axis press for investigating material structure by neutron diffraction. Currently, the press can create pressures up to 16 GPa with enough sample size. The available pressure is limited by the strength of the material of the second stage anvils. Further progress on anvil material and design
for MA6-6 and MA6-8 should expand the available pressure range in the future. In that case, the anvil gap that defines the aperture for the scattering angle is an important point in the development. Other promising applications are deformation experiments to investigate the rheological properties of materials and neutron radiography to observe the macroscopic structure of materials.

VIII. CONCLUSIONS

The six-axis multi-anvil press ATSUHIME was developed for high-pressure, high-temperature TOF neutron diffraction experiments at PLANET at the MLF at J-PARC. Precise control of the anvil positions enabled highly isotropic compression of the sample and well-controlled anisotropic compression. The design of the press, which leaves a relatively large space near the center, allows us to detect diffraction over wide scattering angles and place an incident slit, radial collimators, and a neutron imaging camera near the sample. From the samples, we obtained neutron diffraction patterns of sufficient quality with no contamination due to scattering from the high-PT cell with MA6-6; this is important for precise structural determinations. MA6-6 generated pressures up to 9.3 GPa and temperatures up to 2000 K. MA6-8 expands the available pressure and temperature limits to 16 GPa and 1273 K, respectively. This press provides a new window into the structure of crystals, glasses, and liquids under extreme conditions and should contribute to advancing various fields of science, such as earth and planetary science, material engineering, and condensed matter physics.

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