An Externally-Heated Diamond Anvil Cell for Synthesis and Single-Crystal Elasticity Determination of Ice-VII at High Pressure-Temperature Conditions

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Abstract

The externally-heated diamond anvil cell (EHDAC) can be used to generate simultaneously high-pressure and high-temperature conditions found in Earth's and planetary interiors. Here we describe the design and fabrication of the EHDAC assemblies and accessories, including ring resistive heaters, thermal and electrical insulating layers, thermocouple placement, as well as the experimental protocol for preparing the EHDAC using these parts. The EHDAC can be routinely used to generate megabar pressures and up to 900 K temperatures in open air, and potentially higher temperatures up to ~1200 K with a protective atmosphere (i.e., Ar mixed with 1% H_2). Compared with a laser-heating method for reaching temperatures typically >1100 K, external heating can be easily implemented and provide a more stable temperature at ≤900 K and less temperature gradients to the sample. We showcased the application of the EHDAC for synthesis of single crystal ice-VII and studied its single-crystal elastic properties using synchrotron-based X-ray diffraction and Brillouin scattering at simultaneously high-pressure high-temperature conditions.

Introduction

The diamond anvil cell (DAC) is one of the most important tools for high pressure research. Coupled with synchrotron-based and conventional analytical methods, it has been widely used to study properties of planetary materials up to multi-megabar pressures and at wide ranges of temperatures. Most planetary interiors are under both high-pressure and high-temperature (HPHT) conditions. It is thus essential to heat the compressed samples in a DAC at high pressures in situ to study the physics and chemistry of planetary interiors. High temperatures are not only required for the investigations of phase and melting relationships and thermodynamic properties of planetary materials, but also help mitigate pressure gradient, promote phase transitions and chemical reactions, and expedite diffusion and recrystallization. Two methods are typically utilized to heat the samples in DACs: laser-heating and internal/external resistive heating methods.

The laser-heated DAC technique has been employed for high-pressure materials science and mineral physics research of planetary interiors[1,2]. Although increasing number of laboratories have access to the technique, it usually requires significant development and maintenance effort. The laser heating technique has been used to achieve temperatures as high as 7000 K[3]. However, long-duration stable heating as well as temperature measurement in laser-heating experiments have been a persistent issue. The temperature during laser heating usually fluctuates but can be mitigated by feed-back coupling between thermal emission and laser power. More challenging is controlling and determining the temperature for assembly of multiple phases of different laser absorbance. The temperature also has a considerably large gradient and uncertainties (hundreds of K), although recent technical development effort has been used to mitigate this issue[4,5,6]. Temperature gradients in the heated sample area sometimes may further introduce chemical heterogeneities caused by diffusion, re-partitioning or partial melting. In addition, temperatures less than 1100 K typically could not be measured precisely without customized detectors with high sensitivity in the infrared wavelength range.

The EHDAC uses resistive wires or foils around the gasket/seat to heat the entire sample chamber, which provides the ability of heating the sample to ~900 K without a protective atmosphere (such as Ar/H_2 gas) and to ~1300 K with a protective atmosphere[7]. The oxidation and graphitization of diamonds at higher temperatures limit the highest achievable temperatures using this method. Although the temperature range is limited compared with laser-heating, it provides more stable heating for a long duration and a smaller temperature gradient[8], and is well suited to be coupled with various detection and diagnostic methods, including optical microscope, X-ray diffraction (XRD), Raman spectroscopy, Brillouin spectroscopy and Fourier-transform infrared spectroscopy[9]. Therefore, the EHDAC has become a useful tool to study various material properties at HPHT conditions, such as phase stability and transitions[10,11], melting curves[12], thermal equation of state[13], and elasticity[14].

The BX-90 type DAC is a newly developed piston-cylinder type DAC with large aperture (90° at maximum) for XRD and laser spectroscopy measurements[9], with the space and openings to mount a miniature resistive heater. The U-shaped cut on the cylinder side also provides room to release the stress between the piston and the cylinder side caused by temperature gradient. Therefore, it has recently been widely used in powder or single-crystal XRD and Brillouin measurements with the external-heating setup. In this study, we describe a reproducible and standardized protocol for preparing EHDACs and demonstrated single-crystal XRD as well as Brillouin spectroscopy measurements of synthesized single-crystal ice-VII using the EHDAC at 11.2 GPa and 300-500 K.

Protocol

1. Ring heater preparation

1. Fabricating the ring heater base
    1. Fabricate the ring heater base by a computer numerical control (CNC) milling machine using pyrophyllite based on the designed 3D model. The dimensions of the heater are 22.30 mm in outer diameter (OD), 8.00 mm in inner diameter (ID) and

2.25 mm in thickness. Sinter the heater base in the furnace at 1523 K for >20 hours.

2. Wiring
    1. Cut Pt 10 wt% Rh wire (diameter: 0.01 inch) into 3 equal-length wires (about 44 cm each).
    2. Carefully wind each Pt/Rh wire through the holes in the heater base, leave about 10 cm wire outside of the heater base for connection to the power supply. When wiring, make sure that the wire is lower than the gutters of the base. If it is higher than the gutter, use a proper flat-head screwdriver to press it down.
    3. Wind more wires on the 10 cm extension wires to reduce the electrical resistance and thus the temperature of the extension wires during heating.
3. Adding insulators
    1. Use two small ceramic electrical insulating sleeves to protect the wires extending outside the ring heater base. Mix cement adhesive (e.g., Resbond 919) with water at a ratio of 100:13. Fix those tubes to the ring heater base using the cement mixture.

       NOTE: The cement needs 4 hours to be cured at 393 K or 24 hours at room temperature.
    2. Use the high-temp braid sleeving to protect the outside wires.
    3. Cut two mica rings using a CO_2 laser-cutting machine. To electrically insulate the wire, attach one mica ring to each side of the heater by UHU tac.

2. EHDAC preparation

1. Gluing diamonds
    1. Align the diamonds with backing seats using mounting jigs. Use black epoxy to glue the diamond to the backing seat. The black epoxy should be lower than the girdle of the diamond to leave some space for the high-temperature cement.
2. Alignment
    1. Glue mica or place the machined pyrophyllite rings under the seats to insulate the seats and DAC thermally. Put the seats with the diamonds into a BX-90 DAC. Align two diamonds under the optical microscope.
3. Preparing the sample gasket
    1. Place the rhenium gasket, which is smaller than the hole of the ring heater, between the two diamonds and pre-indent the gasket to approximately 30-45 µm by gently tightening the four screws of DAC. Drill a hole at the center of the indentation by electrical discharge machine (EDM) or laser micro-drilling machine.
4. Mounting thermocouple
    1. Fix two small pieces of mica with the cement mixture on the seat of the piston side of DAC to electrically insulate the thermocouples from the seat. Attach two K-type (Chromega-Alomega 0.005") or R-type (87%Platium/13%Rhodium–Platium, 0.005”) thermocouples to the piston side of the DAC, ensuring that the tips of the thermocouples touch the diamond and close to the culet of the diamond (about 500 µm away). Finally, use the high-temperature cement mixture to fix the thermocouple position and cover the black epoxy on both sides of the DAC.
5. Heater placement
    1. Cut the 2300 °F ceramic tape in the shape of the heater base by CO_2 laser drilling machine and place it on both sides of DAC (piston and cylinder sides). If

it is very easy to move around, use some UHU tac to fix it.

  2. Place the heater in the piston side of the BX-90 DAC. Use some 2300 °F ceramic tape to fill the gap between the heater and the wall of the DAC.

6. Gasket placement
    1. Clean the sample chamber hole of the gasket using a needle or sharpened toothpick to get rid of the metal fragments introduced by the drilling. Use ultrasonic cleaner to clean the gasket for 5-10 min.
    2. Put two small balls of adhesive putty (e.g., UHU Tac) around the diamond on the piston side of the DAC to support the gasket. Align the sample chamber hole of the gasket to match the center of culet under the optical microscope.

3. Synthesizing single-crystal ice-VII by EHDAC

1. Loading sample
    1. Load one or more ruby spheres and one piece of gold into the sample chamber.
    2. Load a drop of distilled water in the sample chamber, close the DAC and compress it by tightening the four screws on the DAC to quickly seal the water in the sample chamber.
2. Pressurizing sample to obtain powder ice-VII
    1. Determine the pressure of the sample by measuring the fluorescence of ruby spheres using a Raman spectrometer.
    2. Carefully compress the sample by turning the four screws and monitor the pressure by ruby florescence until it reaches the stability field of ice-VII (>2 GPa). Watch the sample chamber under the optical microscope during compression. Sometimes the coexistence of water fluid and crystallized ice VI is visible if the pressure is close to the phase boundary of water and ice VI.
    3. Continue compressing the sample chamber until it reaches the pressure in the stability field of ice-VII. In order to melt the ice-VII later, the target pressure is usually between 2 GPa and 10 GPa at 300 K.
3. Heating sample to obtain single crystal ice-VII
    1. Put the EHDAC under the optical microscope with a camera connected to the computer. Thermally insulate the DAC from the microscope stage, without blocking the transmitted light path of the microscope.
    2. Connect the thermocouple to the thermometer and connect the heater to a DC power supply.
    3. Monitor the melting of ice-VII crystals upon heating to a temperature that is higher than the melting temperature of high-pressure ice-VII determined by the phase diagram of H_2O.
    4. Quench the sample chamber to allow the liquid water to crystallize, and then increase the temperature until some of the smaller ice crystals are molten. Repeat the heating and cooling cycles a few times until only one or a few larger grains remains in the sample chamber.
    5. Measure the pressure of the sample after the synthesis.

4. Synchrotron X-ray diffraction and Brillouin spectroscopy collection

1. Synchrotron X-ray diffraction

   1. Check if the ice-VII sample synthesized is polycrystalline or a single crystal by synchrotron-based single-crystal XRD[15]. If it is a single crystal, the diffraction pattern should be diffraction spots instead of powder rings.
   2. Obtain step scan single-crystal XRD images to determine the orientation and lattice parameters of ice-VII.
   3. Collect the XRD of pressure marker, i.e. gold, in the sample chamber to determine the pressure.
2. Brillouin spectroscopy
   1. Mount the EHDAC on a specialized holder which can be rotated within the vertical plane by changing the χ angles. Connect the thermocouples to the temperature controller and connect the heater to the power supply.
   2. Perform Brillouin spectroscopy measurements every 10-15° χ angle at 300 K for a total χ angle range of 180° or 270°[16]. Then heat the sample to high temperatures (e.g., 500 K) and repeat the Brillouin spectroscopy measurement.

Representative Results

In this report, we used the fabricated resistive micro-heater and BX-90 DAC for the EHDAC experiment (**Figure 1** and **Figure 2**). **Figure 1** shows the machining and fabrication processes of the ring heaters. The standard dimensions of the heater base are 22.30 mm in outer diameter, 8.00 mm in inner diameter and 2.25 mm in thickness. The dimensions of the ring heater can be adjusted to accommodate various types of seats and diamonds.

We heated the compressed H_2O sample in an EHDAC at about 6 GPa up to 850 K to synthesize single crystal ice-VII. The ice-VII synthesized from the liquid H_2O after several cycles of heating and cooling was a large single crystal (**Figure 3**). The synthesized single crystal ice VII was utilized for the synchrotron XRD and Brillouin spectroscopy at HPHT. The temperature-power relationship is determined during experiments (**Figure 4**). The single-crystal XRD data were collected as a set of step scans by rotating the omega angle from -110° to -71° at 0.5°/step. The single crystal ice VII had little lattice stress and retained its good quality after compression and heating, as indicated by the sharp Bragg diffraction peaks in synchrotron-based single crystal XRD images (**Figure 5**). The diffraction pattern can be indexed with a cubic structure (space group *Pnm*, $Z = 2$) with unit cell parameters $a = b = c = 3.1375(6)$ Å at 11.2(1) GPa, 300 K and $a = b = c = 3.1605(3)$ Å at 11.2(4) GPa, 500 K. The crystallographic orientation of the single-crystal ice-VII are determined to be (-0.105,0.995,0) at 300K and 500 K. The sound velocities and elastic moduli were obtained by high-pressure and high-temperature Brillouin scattering measurements (Fi**gure 6**). The obtained elastic moduli are: C_{11} =89.73(1) GPa, C_{12} = 55.72(1) GPa and C_{44} = 56.77(1) GPa, K_S = 67.8(1) GPa and G_{VRH} = 34(6) GPa at 11.2(4) GPa and 300 K; C_{11} =82.42(1) GPa, C_{12} = 49.02(1) GPa and C_{44} = 52.82(1) GPa, K_S = 63(1) GPa and G_{VRH} = 30(5) GPa at 11.2(4) GPa and 500 K.

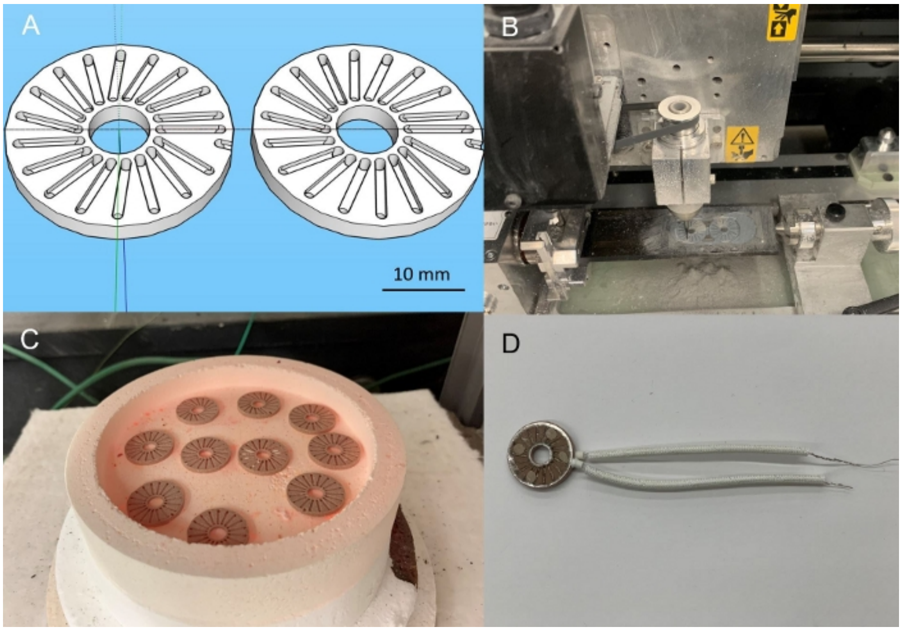

Figure 1: Fabrication of ceramic ring heater base and a micro heater with Pt/Rh wires.

(**A**) 3-D model of the heater base (**B**) Milling the pyrophyllite heater base by the CNC machine. (**C**) Heater bases sintered in the furnace at 1523 K. (**D**) Heater with Pt/Rh wires and insulators (mica, insulating tube and high-temp braid sleeving).

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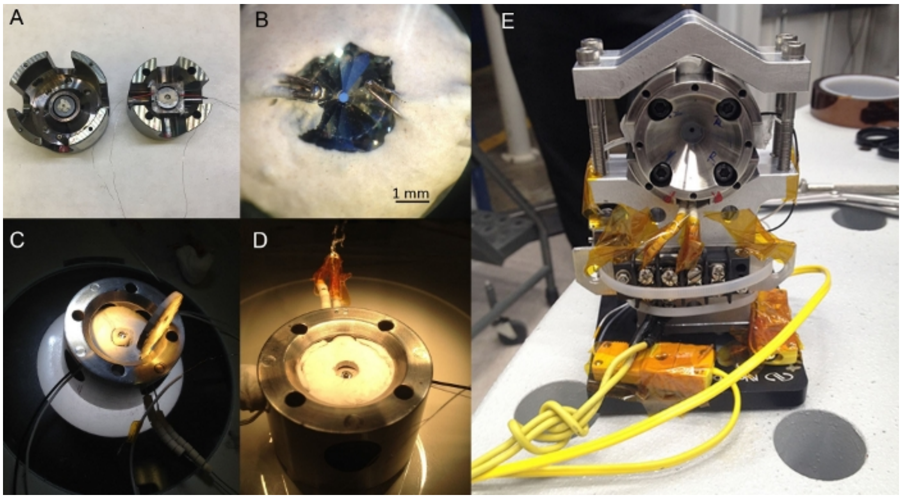

Figure 2: Preparation of EHDAC for high-pressure and high-temperature experiments.

(**A**) BX-90 DAC with thermocouple installed. (**B**) Zoom-in view of the placement of thermocouples near the diamond culet. (**C**, **D**) The placement of micro-heater in the EHDAC. (**E**) EHDAC on the cell holder with the heater connected to a DC power supply and thermocouples connected to a thermometer. Please click here to view a larger version of this figure.

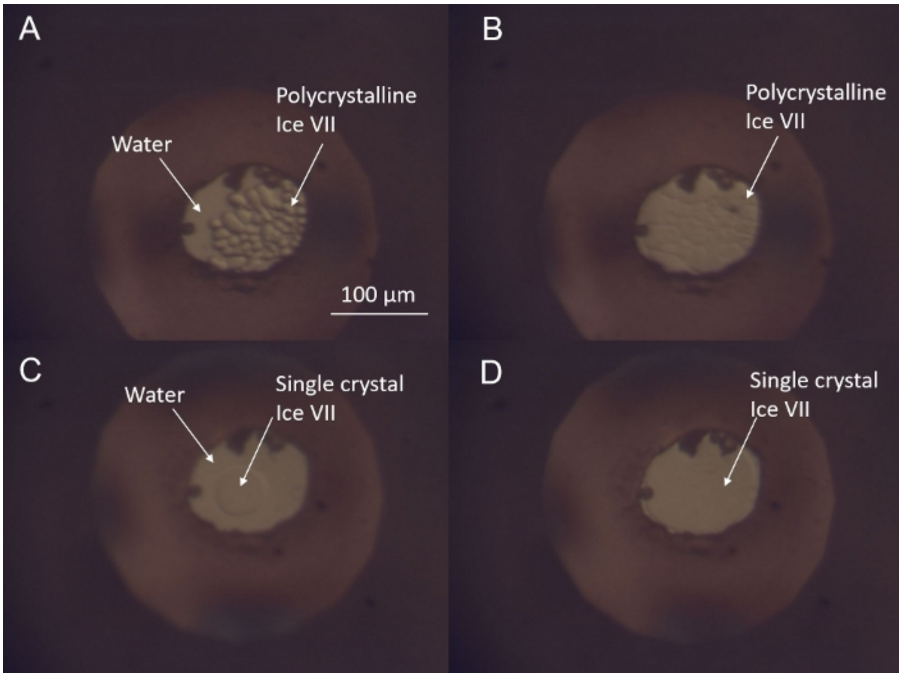

Figure 3: Synthesis of single crystal ice-VII in an EHDAC at about 6 GPa up to 850 K.

(**A**) Polycrystalline ice-VII crystallized from the supercooling water at high pressure and high temperature. (**B**) Growth of polycrystalline ice-VII by decreasing the temperature. (**C**) Growth of a large single-crystal ice-VII and melting of other smaller crystals after multiple heating and cooling cycles. (**D**) Growth of one single-crystal ice-VII to fill the sample chamber by further decreasing the temperature. Please click here to view a larger version of this figure.

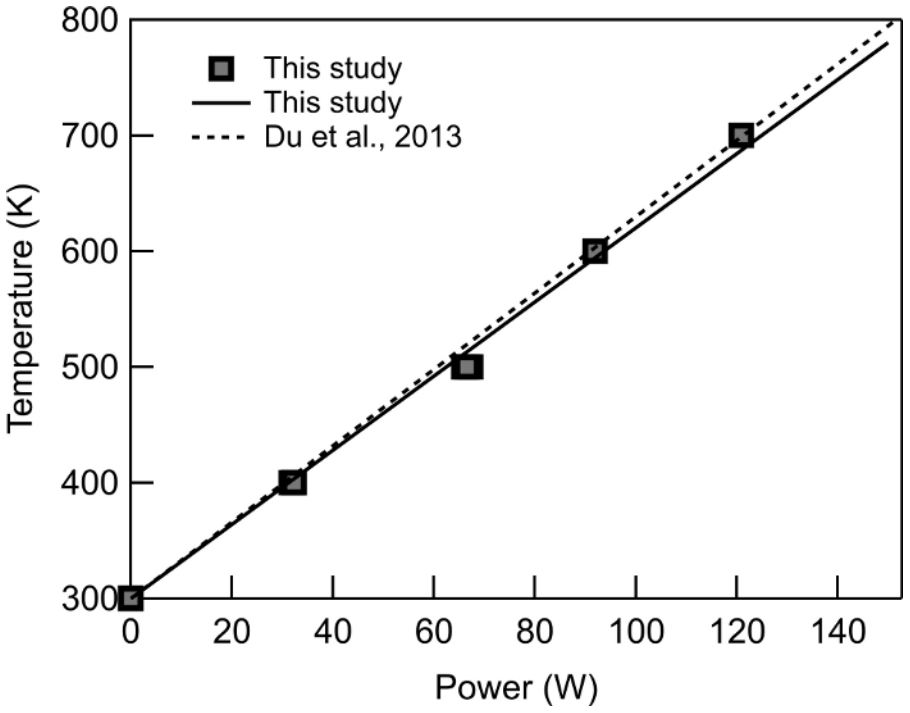

Figure 4: The temperature-power relationship of the EHDAC experiments.

Solid squares represent the temperature-power data in this study, which can be linearly fitted (solid line). This is consistent with the relationship (dashed line) in previous work[7] . Please click here to view a larger version of this figure.

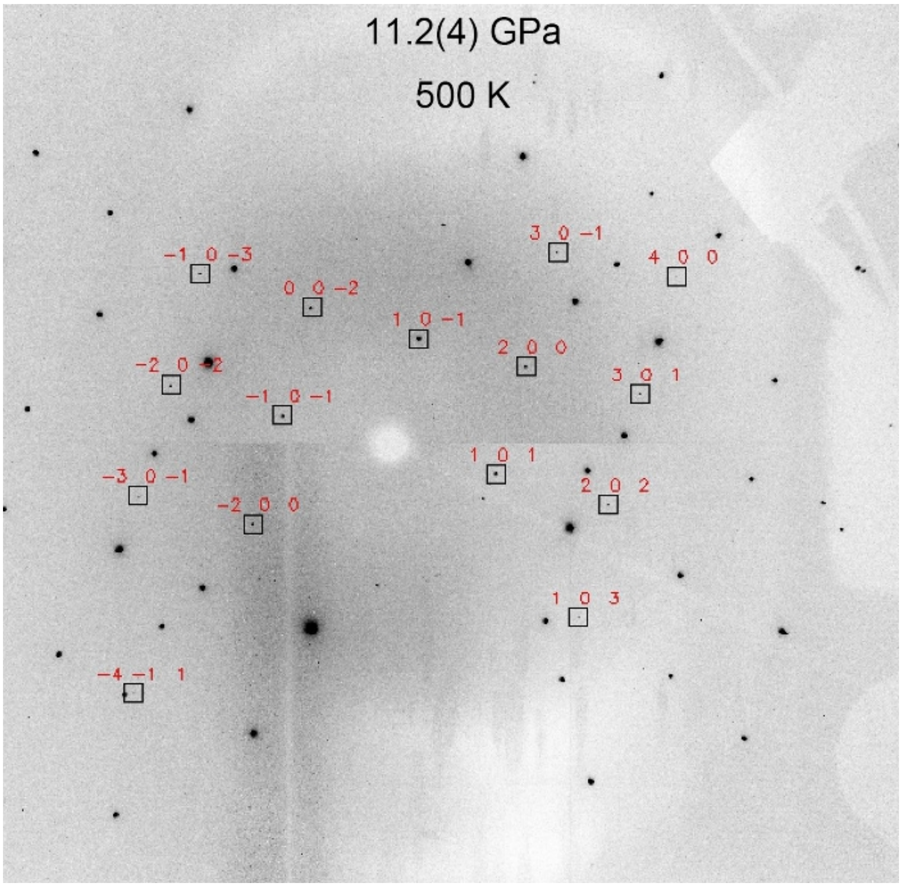

Figure 5: Single crystal XRD pattern of ice-VII at 11.2 GPa and 500 K.

Diffraction peaks of single crystal ice-VII were marked by black boxes. Red labels correspond to Miller indices (*hkl*) of the diffraction peaks. Other single-crystal peaks are from single-crystal diamond anvils used in the EHDAC. Please click here to view a larger version of this figure.

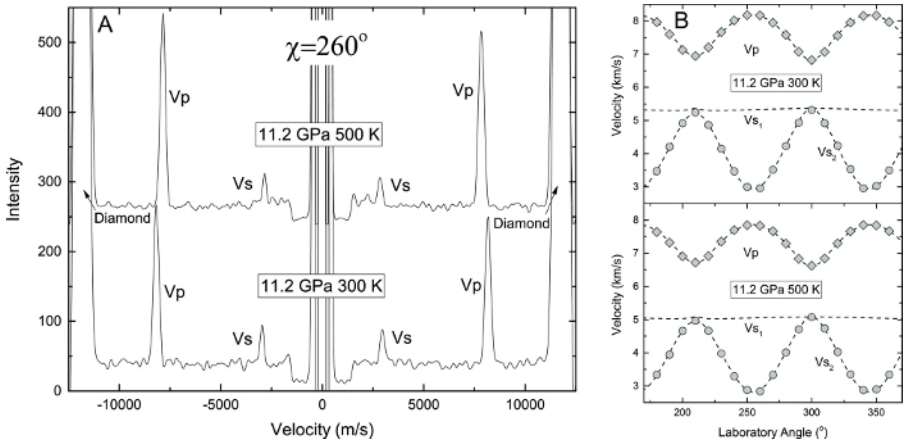

Figure 6: Sound velocities of single crystal ice-VII at 11.2(1) GPa, 300 K and 11.2(4) GPa, 500 K. (**A**) Representative Brillouin spectra of ice-VII at χ angle = 260 ° (**B**) Sound velocities of ice-VII as a function of rotational χ angles. Solid symbols represent the measured velocities by Brillouin spectroscopy. Dashed lines represent the calculated velocities from the best-fit single-crystal elasticity model. Please click here to view a larger version of this figure.

Discussion

In this work, we described the protocol of preparing the EHDAC for high pressure research. The cell assemblies including a micro-heater and thermal and electrical insulating layers. Previously, there are multiple designs of resistive heaters for different types of DACs or experimental configurations[7, 17, 18, 19, 20]. Most of the heaters are machined by individual investigators or purchased from industry which are typically designed for other purposes. Fabricating micro-heaters in a normal machine shop can be time consuming and not always reproducible. In most occasions, the micro-heaters of different designs from individual groups are not optimized and thoroughly tested. The heaters supplied from industry typically are not designed and optimized for EHDAC experiments. Custom designed and machined heaters are mostly pricy due to the requirement of bulk order by industrial machine shops. Therefore, the infrastructure development of heaters for EHDAC experimentation would benefit the entire community with standardized and thoroughly tested heater assemblies, and well documented preparation procedures. In addition, the design and standardization of thermal and electrical insulating layers can help improve the success rate and temperature stability of the EHDAC experiments. The new EHDAC setup allows routine high-temperature DAC experiments for the broad high-pressure community[13].

We have also designed other variations of heaters. The thickness of the heater can be increased to 4.65 mm for the BX90 EHDAC, when backing plates (or seats) with stepped thickness are used. We also designed heaters with varying thickness along the radial direction. They are thinner at the center and thicker near the rim, thus can be used in the EHDAC with short diamonds anvils of Boehler-Almax (BA) design. The DAC with BA diamonds has large opening angles, which is optimal for high-pressure single-crystal XRD experiments.

There are some pros and cons of this technique. The highest achievable temperature is typically limited to 900 K in the open air due to the oxidation and graphitization of diamonds compared with laser-heated DAC. However, higher temperatures above 1200 K have been achieved for a BX90 EHDAC housed in a newly designed and fabricated water-cooled enclosure with protective atmosphere/vacuum and membrane for pressurization. The thermal gradient in the sample chamber of the EHDAC is smaller and the temperature can be stable for a long time (several hours to days) with an easy feed-back control between power and temperature. In this work, the temperature was stable at 500°±2 K for about one day for each Brillouin scattering data collection and multiple heating-cooling cycles can be achieved. Another challenge for the EHDAC is that the pressure sometimes would increase significantly upon heating especially at low pressures (<20 GPa). This could be mitigated by untightening the screws for pressurization before heating or tuning the membrane gas pressure during heating when a membrane pressurization system is used.

There are several critical steps for the EHDAC experimentation. Regarding the placement of the thermocouple for accurate temperature measurements, the thermocouple should be first electrically insulated from the metallic seats and body of the DAC. The junction of the thermocouple should be secured to touch the surface of the diamond's pavilion and <1 mm away from the culet, in order to determine the temperature of the sample. Regarding heater preparation, ensuring good thermal insulation surrounding the micro-heater is critical, and it is necessary to wind more spare wires around the wires extending from the heater to reduce the electrical resistivity and thus the temperature of the extension wires during heating.

Here we showcased the utilization of the EHDAC to synthesize single-crystal ice-VII of good quality from liquid H_2O at HPHT. Combined with the accurately determined single-crystal orientation by single crystal XRD, the elastic moduli with small uncertainties were determined from Brillouin scattering measurements. The elastic moduli at 300 K of ice-VII were close to the previous data[21,22] and the elastic moduli at 500 K was the first HPHT Brillouin results of single-crystal ice-VII reported. The sound velocities and elastic moduli decrease as a function of temperature at 11.2 GPa (**Figure 6**). Experiments at different pressures and temperatures should be performed to understand the temperature effect on the elastic moduli of ice-VII at elevated pressures. In this case, the EHDAC can be used to synthesize high-pressure phases with low melting temperature, and can also be used to simulate the HPHT conditions in the Earth's and planetary interiors. Combined with various detection methods, such as synchrotron XRD and Brillouin spectroscopy, physical properties of planetary materials in deep interiors of planets or moons can be obtained and compared with the geophysical models.

Disclosures

The authors declare no conflict of interest.

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