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Title: 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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Author Questionnaire

1. Microscopy: Does your protocol involve video microscopy, such as filming a complex dissection or microinjection technique? **YES**

If **Yes**, can you record movies/images using your own microscope camera?

YES

If **No**, JoVE will need to record the microscope images using our scope kit (through a camera port or one of the oculars). Please list the make and model of your microscope.

Leica Stereo Microscope, Model MZ16

2. Software: Does the part of your protocol being filmed include step-by-step descriptions of software usage? **NO**

3. Filming location: Will the filming need to take place in multiple locations? **NO**

Introduction

1. Introductory Interview Statements

REQUIRED:

1.1. **Xiaojing Lai:** Externally-heated diamond-anvil cell can generate simultaneously high-pressure and high-temperature conditions to simulate the conditions in Earth's and other planets' interiors.

1.1.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera. *Videographer: Author will film this and send it to JoVE directly.*

1.2. **Feng Zhu:** The main advantage of this technique is that it can be combined with various spectroscopic techniques such as optical microscopy, X-ray diffraction, Raman spectroscopy, and Brillouin scattering.

1.2.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera.

OPTIONAL:

1.3. **Xiaojing Lai:** This technique is used to study interiors of rocky planets and moons. It can also be used to investigate materials' properties under extreme conditions in solid state physics and chemistry.

1.3.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera.

1.4. **Xiaojing Lai:** The most challenging part of the protocol is the placement and fixture of the thermocouples to the diamonds. It is important to carefully follow the instructions when performing this step.

1.4.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera. *Videographer: Author will film this and send it to JoVE directly.*

1.5. **Feng Zhu:** This protocol involves many hands-on steps, so visual demonstration is critical in order to provide sufficient details for the audience to follow.

1.5.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera.

Protocol

2. Ring Heater Preparation

2.1. Begin by cutting the platinum rhodium wire into 3 equal lengths, approximately 44 centimeters each **[1]**. Carefully wind each wire through the holes in the heater base **[2]**, leaving about 10 centimeters outside of the heater base for connection to the power supply **[3]**. *Videographer: This step is important!*

2.1.1. WIDE: Establishing shot of talent cutting wire.

2.1.2. Talent winding a wire through a hole in the heater base.

2.1.3. The wire outside of the base.

2.2. 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 **[1]**. Wind more wires on the 10-centimeter extension wires to reduce the electrical resistance **[2]**. *Videographer: This step is important!*

2.2.1. Talent pressing the wire down to the appropriate position.

2.2.2. Talent winding the wire around the extension.

2.3. Use two small ceramic electrical insulating sleeves to protect the wires extending outside the ring heater base **[1]**. Mix cement adhesive with water at a ratio of 100 to 13 **[2]** and use the mixture to fix those tubes to the ring heater base, then allow the cement to cure **[3-TXT]**.

2.3.1. Talent putting an insulating sleeve on the wire.

2.3.2. Talent mixing the cement adhesive with water.

2.3.3. Talent fixing the tubes to the ring heater with the cement mix. **TEXT: Cure for 4 hours at 393 K or 24 hours at room temperature**

2.4. **[1]**, **[2]**. *Videographer: This step is important!*

~~2.4.1. Talent attaching mica rings to the side of the heater.~~

3. EHDAC Preparation

3.1. Use mounting jigs to align the diamonds with backing seats **[1]**, then glue the diamond to the backing seat with black epoxy. The black epoxy should be lower than the girdle of the diamond to leave some space for the high-temperature cement **[2]**.

3.1.1. Talent aligning diamonds with the backing seats.

3.1.2. Talent gluing the diamond to the backing seat.

3.2. To thermally insulate the seats and diamond anvil cell, or DAC *(pronounce 'dek')*, glue mica or place mica rings under the seats **[1]**. Put the seats with the diamonds into a BX-90 DAC *(pronounce 'dek')* **[2]** and align two diamonds under the optical microscope **[3]**.

3.2.1. Talent placing mica rings under seats.

3.2.2. Talent putting the seats into the DAC.

3.2.3. Talent at the microscope, aligning the diamonds.

3.3. Place the rhenium gasket between the two diamonds **[1]** and gently tighten the four screws of the DAC to pre-indent the gasket to approximately 30 to 45 micrometers **[2]**. Drill a hole at the center of the indentation with an electrical discharge machine or a laser micro-drilling machine **[3]**.

3.3.1. Talent placing the rhenium gasket between the two diamonds.

3.3.2. Talent tightening the screws.

3.3.3. Talent drilling the hole.

3.4. Fix two small pieces of mica with the cement mixture on the seat of the piston side of the DAC to electrically insulate the thermocouples from the seat **[1]**. Attach two K-type or R-type thermocouples to the piston side of the DAC, ensuring that the tips of the thermocouples touch the diamond close to the culet **[2]**. *Videographer: This step is difficult and important!*

3.4.1. Talent fixing the mica on the seat.

3.4.2. Talent attaching the thermocouples.

3.5. Then, use the high-temperature cement mixture to fix the thermocouple position and cover the black epoxy on both sides of the DAC **[1]**.

3.5.1. Talent fixing the thermocouple position with the cement.

3.6. Use the carbon dioxide laser drilling machine to cut the 2300-degree Fahrenheit ceramic tape in the shape of the heater base and place it on both sides of the DAC, fixing it with adhesive putty if necessary **[2]**.

~~3.6.1.~~

3.6.2. Talent placing the tape on the side of the DAC.

3.7. Place the heater in the piston side of the BX-90 DAC **[1]** and use some ceramic tape to fill the gap between the heater and the DAC wall **[2]**. Clean the sample chamber hole of the gasket with a needle to get rid of the metal fragments introduced by the drilling **[3]**, then use ultrasonic cleaner to clean the gasket for 5 to 10 minutes **[4]**. *Videographer: This step is important!*

3.7.1. Talent placing the heater.

3.7.2. Talent filling the gap between the heater and DAC wall with ceramic tape.

3.7.3. Talent cleaning the hole on the sample chamber.

3.7.4. Talent using the ultrasonic cleaner.

3.8. Put two small balls of adhesive putty around the diamond on the piston side of the DAC to support the gasket **[1]**. Then, align the sample chamber hole of the gasket to match the center of culet under the optical microscope **[2]**.

3.8.1. Talent putting the adhesive putty around the diamond.

3.8.2. SCOPE: sample chamber hole aligning.

4. Single-crystal Ice-VII by EHDAC Synthesis

4.1. Load one or more ruby spheres and one piece of gold into the sample chamber **[1]**, then load a drop of distilled water in the sample chamber **[2]**, close the DAC and compress it by tightening the four screws **[3]**.

4.1.1. Talent loading the sample chamber.

4.1.2. Talent loading water into the sample chamber.

4.1.3. Talent closing the DAC and tightening the screws.

4.2. Determine the pressure of the sample by measuring the fluorescence of ruby spheres with a Raman spectrometer **[1]**. Carefully compress the sample by turning the four screws and monitor the pressure until it reaches the stability field of ice-VII *(pronounce 'ice-7')*. The target pressure is usually between 2 and 10 gigapascal at 300 Kalvin **[2]**.

4.2.1. Talent measuring the fluorescence.

4.2.2. Talent compressing the sample and monitoring the pressure.

4.3. Put the externally heated DAC under the optical microscope with a camera connected to the computer **[1]**. Thermally insulate the DAC with the microscope stage, without blocking the transmitted light path of the microscope **[2]**.

4.3.1. Talent putting the EHDAC under the microscope. NOTE: 4.3.1 and 4.3.2 in one shot

4.3.2. Talent insulating the DAC.

4.4. Connect the thermocouple to the thermometer **[1]** and connect the heater to a DC power supply **[2]**. Monitor the melting of ice-VII crystals upon heating to a temperature that is higher than the melting temperature of high-pressure ice-VII **[3]**. *Videographer: This step is important!*

4.4.1. Talent connecting the thermocouple to the thermometer.

4.4.2. Talent connecting the heater to the power supply.

4.4.3. SCOPE: Crystals melting. *Video Editor: This will be supplied by authors.*

NOTE: JOVE_SCOPE_61389_step4.4.3

4.5. Quench the sample chamber to allow the liquid water to crystallize **[1]**, then increase the temperature until some of the smaller ice crystals are molten **[2]**. Repeat the heating and cooling cycles a few times until only one or a few larger grains remain in the sample chamber **[3]**.

4.5.1. Talent quenching the sample chamber.

4.5.2. Talent increasing the temperature.

4.5.3. SCOPE: Sample as it goes through the heating and cooling cycles. *Video Editor: This will be supplied by authors.*

JOVE_SCOPE_61389_step4.5.3

Results

5. Results: Synthesis of Single Crystal ice-VII in an EHDAC

5.1. The compressed water sample was heated in an externally heated DAC at about 6 gigapascal up to 850 Kalvin to make single crystal ice-VII *(pronounce 'ice-seven')*. A large single crystal was synthesized after several cycles of heating and cooling **[1]**.

5.1.1. LAB MEDIA: Figure 3. *Video Editor put the images A – D in one line, starting with A and ending with D, to indicate that the sample started out as A and ended as D.*

5.2. The synthesized single crystal ice VII was utilized for synchrotron X-ray diffraction and Brillouin spectroscopy at high pressure and high temperature. The temperature-power relationship was determined **[1]**.

5.2.1. LAB MEDIA: Figure 4.

5.3. The crystal 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 X-ray diffraction images. The diffraction pattern can be indexed with a cubic structure **[1]**.

5.3.1. LAB MEDIA: Figure 5.

5.4. The sound velocities and elastic moduli were obtained by high-pressure and high-temperature Brillouin scattering measurements **[1]**.

5.4.1. LAB MEDIA: Figure 6.

Conclusion

6. Conclusion Interview Statements

6.1. **Xiaojing Lai:** When attempting this protocol, the placement of the thermocouple is important. The thermocouple should be electrically insulated from the seats and DAC and it should be close to the culet of the diamond.

6.1.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera. *Suggested B-roll: 3.4.2. Videographer: Author will film this and send it to JoVE directly.*

6.2. **Feng Zhu:** The EHDAC is often combined with Raman, FTIR and numerous synchrotron radiation spectroscopic methods such as X-ray diffraction to measure the properties of materials in-situ at HPHT conditions.

6.2.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera.

6.3. **Feng Zhu:** For those who are familiar with DAC, this technique can be easily learned to enable performance of not only high pressure but also high temperature measurements in future studies.

6.3.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera.