Video Article

Simulation of the Planetary Interior Differentiation Processes in the Laboratory

Yingwei Fei¹

¹Geophysical Laboratory, Carnegie Institution of Washington

Correspondence to: Yingwei Fei at fei@gl.ciw.edu

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Abstract

A planetary interior is under high-pressure and high-temperature conditions and it has a layered structure. There are two important processes that led to that layered structure, (1) percolation of liquid metal in a solid silicate matrix by planet differentiation, and (2) inner core crystallization by subsequent planet cooling. We conduct high-pressure and high-temperature experiments to simulate both processes in the laboratory. Formation of percolative planetary core depends on the efficiency of melt percolation, which is controlled by the dihedral (wetting) angle. The percolation simulation includes heating the sample at high pressure to a target temperature at which iron-sulfur alloy is molten while the silicate remains solid, and then determining the true dihedral angle to evaluate the style of liquid migration in a crystalline matrix by 3D visualization. The 3D volume rendering is achieved by slicing the recovered sample with a focused ion beam (FIB) and taking SEM image of each slice with a FIB/SEM crossbeam instrument. The second set of experiments is designed to understand the inner core crystallization and element distribution between the liquid outer core and solid inner core by determining the melting temperature and element partitioning at high pressure. The melting experiments are conducted in the multi-anvil apparatus up to 27 GPa and extended to higher pressure in the diamond-anvil cell with laser-heating. We have developed techniques to recover small heated samples by precision FIB milling and obtain high-resolution images of the laser-heated spot that show melting texture at high pressure. By analyzing the chemical compositions of the coexisting liquid and solid phases, we precisely determine the liquidus curve, providing necessary data to understand the inner core crystallization process.

Video Link

The video component of this article can be found at <http://www.jove.com/video/50778/>

Introduction

Terrestrial planets such as the Earth, Venus, Mars, and Mercury are differentiated planetary bodies consisting of a silicate mantle and a metallic core. The modern planet formation model suggests that the terrestrial planets were formed from collisions of Moon-to-Mars-sized planetary embryos grown from km-sized or larger planetesimals through gravitational interactions¹⁻². The planetesimals were likely differentiated already once the metallic iron alloys reached melting temperature due to heating from sources such as radioactive decay of short-lived isotopes such as ²⁶Al and ⁶⁰Fe, impact, and release of potential energy³. It is important to understand how the liquid metal percolated through a silicate matrix during the early differentiation.

Planet differentiation could proceed through efficient liquid-liquid separation or by percolation of liquid metal in a solid silicate matrix, depending on the size and interior temperature of the planetary bodies. The percolation of liquid metal in the solid silicate matrix is likely a dominant process in the initial differentiation when the temperature is not high enough to melt the entire planetary body. The efficiency of percolation depends on the dihedral angle, determined by the interfacial energies of the solid-solid and solid-liquid interfaces. We can simulate this process in the laboratory by conducting high-pressure and high-temperature experiments on a mixture of iron alloy and silicate. Recent studies⁴⁻⁷ have investigated the wetting ability of liquid iron alloys in a solid silicate matrix at high pressure and temperature. They used a conventional method to measure the relative frequency distributions of apparent dihedral angles between the quenched liquid metal and silicate grains on the polished cross-sections for determination of the true dihedral angle. The conventional method yields relatively large uncertainties in the measured dihedral angle and possible bias depending on the sampling statistics. Here we present a new imaging technique to visualize the distribution of liquid metal in the silicate matrix in three dimensions (3D) by combination of FIB milling and high-resolution field-emission SEM imaging. The new imaging technique provides precise determination of the dihedral angle and quantitative measure of the volume fraction and connectivity of the liquid phase.

The Earth's core was formed in a relatively short time (<100 million years)⁸, presumably in a liquid state at its early history. Mars and Mercury also have liquid cores based on solar tidal deformation from the Mars Global Surveyor radio tracking data⁹ and radar speckle patterns tied to the planetary rotation¹⁰, respectively. Thermal evolution models and high-pressure melting experiments on core materials further support a liquid Martian core¹¹⁻¹². Recent Messenger spacecraft data provide additional evidence for a liquid core of Mercury¹³. Even the small Moon likely has a small liquid core based on recent reanalysis of Apollo lunar seismograms¹⁴. Liquid planetary cores are consistent with high accretion energy at the early stage of planet formation. Subsequent cooling may lead to formation of solid inner core for some planets. Seismic data have revealed

that the Earth consists of a liquid outer core and a solid inner core. The formation of the inner core has important implications for the dynamics of the core driven by thermal and compositional convections and the generation of the magnetic field of the planet.

Solidification of the inner core is controlled by the melting temperature of core materials and the thermal evolution of the core. Core formation of terrestrial planets shared similar accretion paths and the chemical composition of the cores is considered to be dominated by iron with about 10 weight % light elements such as sulfur (S), silicon (Si), oxygen (O), carbon (C), and hydrogen (H)¹⁵. It is essential to have knowledge of the melting relations in the systems relevant to the core, such as Fe-FeS, Fe-C, Fe-FeO, Fe-FeH, and Fe-FeSi at high pressure, in order to understand the composition of the planetary cores. In this study, we will demonstrate experiments conducted in the multi-anvil device and diamond-anvil cell, mimicking the conditions of the planetary cores. The experiments provide information on the crystallization sequence and element partitioning between solid and liquid metal, leading to a better understanding for the requirements of the inner core crystallization and the distribution of light elements between the crystalline inner core and liquid out core. To extend the melting relationships to very high pressures, we have developed new techniques to analyze the quenched samples recovered from laser-heated diamond-anvil cell experiments. With precision FIB milling of the laser-heating spot, we determine melting using quenching texture criteria imaged with high-resolution SEM and quantitative chemical analysis with a silicon drift detector at submicron spatial resolution.

Here we outline two sets of experiments to mimic planetary core formation by percolation of metallic melt in silicate matrix during early accretion and inner core crystallization by subsequent cooling. The simulation is aimed to understand the two important processes during the evolution of planetary core.

Protocol

1. Prepare Starting Materials and Sample Chambers

1. Prepare two types of starting materials, (1) a mixture of natural silicate olivine and metallic iron powder with 10 wt% sulfur (metal/silicate ratios ranging from 4 to 30 wt%) for simulating percolation of liquid iron alloy in a solid silicate matrix during the initial core formation of a small planetary body, and (2) a homogeneous mixture of finely-grounded pure iron and iron sulfide for determining the planetary inner core crystallization.
2. Grind the starting materials to fine mixed powder under ethanol in an agate mortar for one hour and dried at 100 °C.
3. Load the starting material into a sintered MgO or Al₂O₃ capsule (typically 1.5 mm in diameter and 1.5 in length), and then place it in a high-pressure cell assembly for the multi-anvil experiments.
4. Load the Fe-FeS mixture into a small sample chamber (typically 100 μm in diameter and 25 μm in thickness) drilled in a preindented rhenium gasket for the laser-heating experiments in the diamond-anvil cell. Sandwich the Fe-FeS mixture between NaCl layers which serve as thermal insulators.

2. High-pressure and High-temperature Experiments in the Multi-anvil Apparatus

1. The multi-anvil high-pressure cell assembly consists of an MgO octahedron as a pressure medium, a ZrO₂ sleeve as the thermal insulator, and a cylindrical rhenium or graphite heater. The sample capsule fits inside the heater. A type-C thermocouple is inserted into the sample chamber to determine the sample temperature.
2. Place the high-pressure assembly in a multi-anvil high-pressure apparatus for pressurization.
3. The multi-anvil apparatus consists of a 1,500 ton hydraulic press and a pressure module which contains a retaining ring with six removable push wedges forming a cubic cavity in the center¹⁵. The cubic cavity houses eight tungsten carbide cubes with truncated corners. The truncated cubes, which converge on the octahedron cell assembly, are separated from one another by compressible gaskets. The hydraulic ram transmits the force effectively onto the sample assembly by a two-stage anvil configuration. **Figure 1** illustrates the experimental procedure for the multi-anvil experiment.
4. Pressurize the sample to a target pressure between 2-27 GPa at room temperature based on fix-point pressure calibration curve¹⁶, and then heat it to the experimental temperatures up to 2,300 °C by electrical resistance heating; maintain the experiment at a constant temperature for the duration of the experiment; and turn off the power to quench the sample to room temperature at the end of the experiment.
5. Release pressure slowly by opening the hydraulic oil valve and recover the experimental charge.

3. Laser-heating Experiments in the Diamond-anvil Cell

1. Pressure in a diamond-anvil cell is generated between two gem-quality single-crystal diamond anvils (about 0.25 carats each). We use a symmetric diamond-anvil cell to drive the perfectly aligned opposite anvils with a piston-cylinder system. The cell is capable of generating pressures corresponding to the pressure conditions of the Earth's core¹⁷. High temperature is achieved by laser heating in the diamond-anvil cell. We use a system at the Advance Photon Source (APS), which is based on a double-sided laser heating technique and consists of two fiber lasers, optics to heat the sample from both sides, and two spectroradiometric systems for temperature measurements on both sides¹⁸. The system is designed to generate a large heating spot (25 μm in diameter), minimize the sample temperature gradients both radially and axially in the diamond anvil cell, and maximize heating stability. **Figure 2** shows schematics of the experimental configuration for the laser-heating experiment in the diamond-anvil cell with an image of the laser-heating spot.
2. Align the diamond anvils with 300 μm culets and preindent a rhenium gasket to a thickness of 30 μm from an initial thickness of 250 μm.
3. Drill a hole in the preindented gasket with a diameter of 120 μm at the center, and load the sample in the hole.
4. Pressurize the sample to a target pressure at room temperature, and then heat the sample by increasing the laser power while taking temperature measurements and *in situ* X-ray diffraction measurements at the synchrotron facility.
5. Turn off the laser power to quench the sample when partial melting is detected by a change in thermal radiation and from the diffraction pattern.

6. Recover the heated sample for *ex situ* characterization.

4. Sample Recovery and Analysis

1. Mount the retrieved multi-anvil sample in epoxy resin and polish its surface using a suite of diamond powder grit from 150 μm to 0.25 μm .
2. Carbon-coat the surface of the sample and load it into the sample chamber of a Zeiss Auriga FIB/SEM crossbeam instrument (**Figure 3A**) for analysis.
3. Align the sample to the coincident point of the FIB and SEM at a working distance of 5 mm (**Figure 3B**), and then premill the sample to expose a volume of $15 \times 20 \times 20 \mu\text{m}^3$ (**Figure 3C**).
4. Take SEM images at an interval of 25 nm using the slice&view function on the Zeiss Auriga FIB/SEM instrument (automatically record a series of images after ion-beam milling with typical image resolution of about 35 nm).
5. Input the image data files to a visualization software and reconstruct 3D images to visualize the melt distribution and connectivity in the quenched sample (**Figure 3D**).

Representative Results

We have conducted a series of experiments using mixtures of San Carlos olivine and Fe-FeS metal alloy with different metal-silicate ratios, as the starting materials. The S content of the metal is 10 weight % S. Here we show some representative results from high-pressure experiments performed at 6 GPa and 1,800 °C, using well-calibrated multi-anvil assemblies¹⁵. Under the experimental conditions, the Fe-FeS metal alloy is completely molten and the silicate (San Carlos olivine) remains crystalline. The purpose of the experiment is to examine how liquid metal would percolate through crystalline silicate. The efficiency of removal of liquid metal alloys from a solid silicate matrix strongly influences the timing of core formation and the composition of the core through mantle-core interaction. It depends on the percolation threshold and the dihedral angle. For samples with the melt fraction below the minimum percolation threshold, interconnected melt can exist only when the dihedral angle is below 60°. **Figure 4** shows 3D reconstruction of the quench sample. The measured dihedral angle for the Fe-FeS melt in the olivine matrix is above 100°, larger than the critical angle (60°) that divides the non-connected and interconnected networks. The calculated melt percentage is about 3.3 volume%, which is below the minimum percolation threshold. The image clearly shows the metallic melt pockets were trapped at the silicate grain corners because of the large dihedral angle. This study along with previous studies¹⁹⁻²⁰ shows that the dihedral angle for Fe-FeS melts in the olivine matrix is above the critical dihedral angle at high pressures. The Fe-FeS melt evenly distributes in the olivine matrix without forming an interconnected melt network.

The Fe-FeS system with eutectic melting behavior and preferential S partitioning to liquid iron has been used as a model system to explain the basic observations of the Earth's core system, including the liquid outer core and solid inner core configuration and the large density jump at the inner core boundary (ICB). It is also applicable to the cores of terrestrial planets such as Mars and Mercury. In order to definitely evaluate the role of S during core formation and evolution of the core, we must have full knowledge of the phase relations in the Fe-FeS system as a function of pressure up to core pressures. High-pressure experiments on Fe-FeS melting relations using piston-cylinder apparatus and multi-anvil device have provided fundamental knowledge of the phase relations in the system up to 25 GPa²¹⁻²⁵. However, detailed mapping of the liquidus curves in the Fe-rich region has only been reported up to 14 GPa²⁴⁻²⁵. We have developed an efficient way to map the phase relations in the Fe-rich region that can be extended to pressures up to at least 27 GPa. **Figure 5** shows a melting experiment at 21 GPa with two sample chambers loaded with two different starting compositions (3 wt% and 7 wt% sulfur). The total length of the two samples is still less 500 μm , limiting to small thermal gradient within the sample chambers. At 21 GPa and 2,023 K, the starting sample with 7 wt% S was molten completely indicating condition above the liquidus temperature, whereas the sample with 3 wt% S forms Fe and Fe-S melt indicating condition within the solid iron + liquid two-phase region. By analyzing the compositions of the solid and melt phases, the liquidus curve and the S partitioning between solid and melt phases are precisely determined.

In order to extend the measurements on the melting relations to even higher pressure (>27 GPa), it is necessary to use the laser-heating technique in the diamond anvil cell. The key aspects of the experiment are (1) recovering the laser-heated sample and specifically polishing the heating spot with FIB; (2) obtaining high-resolution images of the heated spot and establishing melting criteria; and (3) analyzing chemical compositions of the coexisting phases with a silicon drift detector (SDD). We use both *in situ* X-ray diffraction measurements and *ex situ* chemical analyses of the recovered samples to determine melting and chemical compositions of the coexisting phases. The recovered samples are prepared and analyzed with a Zeiss Auriga FIB/SEM crossbeam system installed at the Geophysical Laboratory. The crossbeam system integrates a FIB system and a field-emission scanning electron microscope (FE-SEM) in one powerful instrument. It equipped with an analytical silicon drift detector for chemical analysis. **Figure 6** shows the quenched sample from 53 GPa, with laser-heated spots heated to different temperatures. We have milled the heated spots to obtain melting texture information. **Figure 6C** shows clear melting textures, similar to that of the quenched multi-anvil sample, but at a much smaller scale. By analyzing the compositions of the two coexisting phases, we can determine the liquidus curve and S partitioning between solid and liquid. The study demonstrated that we have established a reliable experimental procedure to obtain high-quality melting data from the recovered laser-heating DAC samples, providing necessary data to understand the inner core crystallization process.

Figure 1. The experimental procedure includes preparing the starting materials (A), loading a sample into the multi-anvil assembly (B), assembling the second-stage anvils into the pressure module (C), and setting up for pressurization in the hydraulic press (D). [Click here to view larger image.](#)

Figure 2. Schematics of the experimental configuration for the laser-heating experiment in the diamond-anvil cell. An image of a laser-heated spot (20 μm) is shown. *In situ* diffraction pattern can be collected at high pressure and temperature at a synchrotron radiation facility. [Click here to view larger image.](#)

Figure 3. Schematics for 3D data collection. (A) FIB/SEM crossbeam instrument; (B) Sample stage inside FIB/SEM; (C) Set-up for 3D slicing and viewing; and (D) 3D reconstruction using Avizo software. The size of the bounding box is $4\ \mu\text{m} \times 6\ \mu\text{m} \times 5\ \mu\text{m}$. [Click here to view larger image.](#)

Figure 4. 3D reconstruction of Fe-FeS melt in an olivine matrix. The size of the bounding box is $5\ \mu\text{m} \times 6.1\ \mu\text{m} \times 7.2\ \mu\text{m}$. The highlighted volume represents the Fe-FeS melt whereas the crystalline olivine occupies the transparent volume. [Click here to view larger image.](#)

Figure 5. Melting experiment result in the Fe-FeS system at 21 GPa and 2,023 K. Two sample chambers loaded with two different starting compositions (3 wt% and 7 wt% sulfur) yielded precise determination of liquid curves and S partitioning between solid and liquid phases. [Click here to view larger image.](#)

Figure 6. Milling and imaging of the laser-heated spot. (A) Picture of the sample in the diamond anvil cell at 53 GPa based on NaCl pressure scale 30. The laser-heated spots are visible in the reflective light. (B) SEM image of the quenched heating spot. Three milling areas are shown to expose the laser-heated spots. (C) High-resolution SEM image of the partially molten area at the spot heated to 2,300 K. The melting texture is very similar to that of the quenched multi-anvil sample, but at a much smaller scale. The scale bar represents 400 nm. [Click here to view larger image.](#)

Figure 7. Design of five sample chambers in a SiO_2 glass plate loaded in a Re gasket. Each chamber is 15 μm in diameter (smaller than the laser spot) and 15 μm depth. Each chamber confines the sample individually, which is critical to prevent melt migration after melting. The individual sample is imaged after recovering from the high-pressure experiment. Heating spots at 2,000 K and 2,200 K are shown as inserts. [Click here to view larger image.](#)

Discussion

The techniques for the multi-anvil experiments are well established, generating stable pressure and temperature for an extended period of run time and producing relatively large sample volume. It is a powerful tool to simulate the interior processes of planets, especially for experiments, such as melt percolation, that require certain sample volume. The limitation is the maximum achievable pressure, up to 27 GPa with tungsten carbide (WC) anvils, reaching the core pressures of Mars and Mercury, but far too low pressure to reach the cores of the Earth and Venus. The maximum achievable pressure can be extended to about 100 GPa by using expensive sintered diamond as anvils²⁶. We are testing less expensive new anvil material made of sintered diamond and silicon carbide. Our test results showed efficient pressure generation with great potential. We use 25-mm cubes as anvils instead of the conventional 14-mm cubes to maximize sample volume in the same pressure range achieved by the conventional WC anvils, which opens a new research opportunity for experiments that require large sample volume, such as measurements of transport properties and synthesis of large samples for industrial applications at high pressure.

The 3D imaging utilizes the combined capabilities of FIB and SEM to produce high-resolution volume rendering at the nano-scale. It is complementary to the X-ray tomography²⁷⁻²⁹, but provide much high spatial resolution. It provides a new, powerful tool to precisely determine the true dihedral angle. The method is far more superior than the traditional technique¹⁹⁻²⁰ based on the measurements of the relative frequency distributions of apparent dihedral angles between the quenched liquid metal and silicate grains on polished 2D cross-sections. It further provides the details of each interface, allowing examination of the wetting ability of liquid in the matrix with multiple crystal phases. Through quantitative calculations, we can obtain volume fraction, surface area ratio, and connectivity. The 3D network through reconstruction can also be used as a realistic import 3D model for other calculations of transport properties such as permeability and conductivity.

Because of its high spatial resolution, the 3D imaging is limited to rendering of small volume (typically 20 μm x 20 μm x 20 μm). This is ideal for imaging the laser-heating spot in the diamond-anvil cell. We have imaged the laser-heated spot of iron from the recovered sample in 3D to illustrate melting of iron at high pressure. For the measurement of dihedral angle in the recovered multi-anvil sample, it is necessary to prevent large crystal growth in order to obtain representative 3D data. We perform experiments in a small confined sample chamber and have observed significant crystal size reduction with small sample chamber for the same run conditions, comparing to large sample chamber. The small sample volume is preferred when we try to reach extreme pressure conditions, but we do need to ensure texture equilibrium and representative chemical composition and homogeneity. To evaluate texture equilibrium, we performed experiments for 6 and 12 hr, and did not observe significant changes of texture in these experiments.

It is important to prepare homogeneously mixed starting materials for the laser-heated DAC experiments because the laser-heating spot is only about 20 μm in diameter. Typically, we mechanically mix Fe and FeS powder to make starting materials with different S contents. It is difficult to breakdown Fe powder to micron-size grains with mechanical grinding. We often see compositional variations from heating spot to spot within the same DAC sample. This affects not only the ability to control the starting compositions, but also uniform laser-coupling with the sample. Through many tries, we now make homogeneous starting mixtures by melting the Fe-FeS mixtures and then regrounding the charges to fine grains and sintering them again. This procedure can produce a homogeneous composition at the 2-3 μm scale. Homogeneity at a fine spatial scale is imperative for achieving uniform heating and tightly controlling the starting composition.

Large temperature fluctuations upon melting are commonly observed, which prevents accurate determination of the melting temperature. The temperature fluctuations are due to melt convection and migration when there is no physical container for the heated sample. We designed small sample containers with the diameter (15 μm), smaller than the laser spot (**Figure 7**). Such containers reduce thermal gradients and prevent melt migration during heating. In addition, samples in each container can be heated to well-controlled different target temperatures, dramatically increasing the efficiency of the experiments. Such design is only becoming possible with FIB micro-fabrication and the samples can be recovered by FIB technology and analyzed with high-resolution SEM.

Disclosures

No conflict of interest declared.

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