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Building In-situ Diamond Anvil Cell Sample Assemblies with Xe PFIB

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The diamond anvil cell (DAC) is an experimental tool that is commonly used to mimic the high-pressure conditions deep inside planets [1]. Materials, such as rocks and metals, are compressed to millions of atmospheres of pressure inside the cell, heated to a temperature of interest, and probed by diagnostics that measure material properties of interest to planetary scientists, such as density, electrical conductivity, and melting temperature.

In the six decades since the invention of the diamond anvil cell [2], most experiments have relied on human hands to prepare the approximately 10 micron-sized samples, and to arrange them inside the experimental sample chamber. An emerging alternative is to use automated methods.

Focused ion beams (FIBs) open up new possibilities to prepare samples in precisely-controlled shapes for diamond anvil cell experiments [3]. Recently, FIBs have been used to mill sample materials with sub-micron precision into the shapes needed for relatively precise electrical conductivity measurements [4-5]. However, the precisely milled samples were subsequently compressed inside hand-prepared sample chambers, allowing for substantial distortion of the sample's original shape. Here, we extend the FIB preparation method in order to control the starting shape of everything that presses against the sample during the experiment. We utilize a xenon (Xe) plasma focused ion beam (PFIB) to mill and construct a stacked sample assembly, including a custom-shaped trench in alumina and an inversely custom-shaped piece of Fe. Using an in-situ micromanipulator, we lift a piece of Fe out of the bulk and fit it into an alumina trench ("lift-in"). We then assemble the rest of the stack in-situ, eliminating the need for error-prone manual manipulation. The sample is compressed and heated in the full DAC assembly.

Not only does our method enable unprecedented control of sample shape during compression of samples, it also enables our experimental design and implementation cycle to more be efficient. We are optimistic that our method will lead to a major improvement in the accuracy of electrical conductivity measurements at high pressure.

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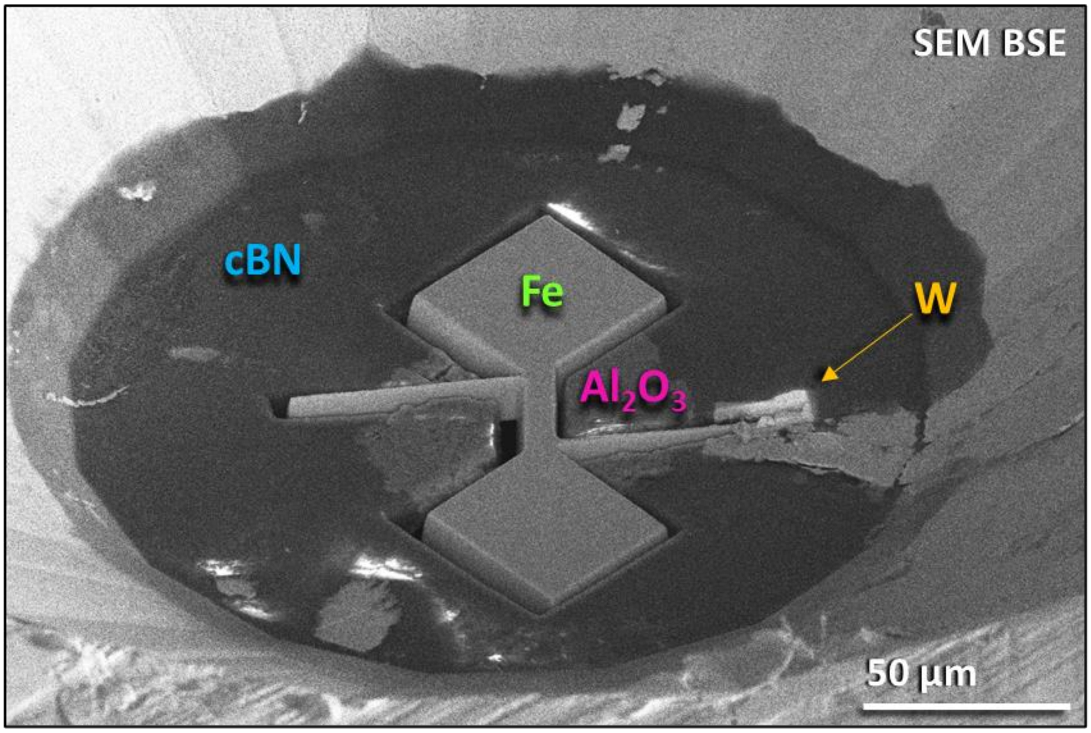

Figure 1. SEM BSE image of the Fe sample inside the alumina trench after the “lift-in” procedure is complete. The Fe is attached to the cBN with a small area of W deposition.

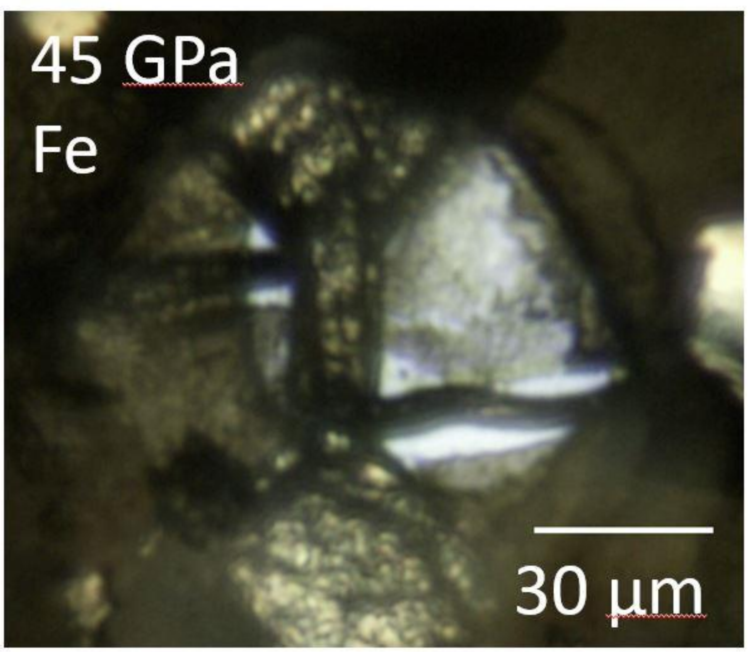

Figure 2. Transmission optical microscope image showing the sample compressed to 45 GPa inside of the diamond anvil cell.

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