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Single Crystal Preparation for High-Pressure Experiments in the Diamond Anvil Cell

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Introduction

Most research conducted in diamond anvil cells (DAC) is performed on polycrystalline samples. While data from polycrystalline samples are sufficient for determining the bulk properties, high-pressure experiments on single crystals allow for measurements on a range of tensor properties such as: thermal and electrical conductivity; magnetic susceptibility; elasticity; and plasticity. However, in order to achieve pressures above 1 Mbar in a DAC, single crystal samples must be <50 μm in diameter and <15 μm thick while maintaining a high degree of crystallinity. Thus, we have developed new procedures for producing extremely high-quality micro single crystal samples from commercially available material. Our sample preparation steps include cutting, classical metallographic polishing, and laser ablation. The key to our new process is the preservation of crystallinity during cutting and thinning. We have been successful in maintaining orientation, along with an extremely high degree of crystallinity in completed metal samples. To date, we have analyzed cobalt and molybdenum samples with both white-light interferometry and synchrotron x-ray diffraction, and are in the process of extending these methods to other metals and ceramics.

Sample Preparation Method

Step 1: Cutting

The initial sample is oriented and a wire saw is used to cut a thin section (~1 mm thick) from the as-received sample.

Step 2: Polishing

The cut sample is embedded in an epoxy resin matrix that is used to hold the sample, and to prevent damage during thinning. The sample is then mounted on a polishing fixture used to polish both surfaces to a high degree of parallelism in order to maintain sample orientation. The sample is polished using descending grits of alumina slurry or diamond paste (paste selection is dependent on sample material). An appropriate chemical solution is used as a micro etchant between every polishing step. After each step, the surface is examined for etch pits. Etching proceeds until the disturbed surface layer is removed[1]. The final step before laser cutting is to polish the sample to a mirror surface using chemical polish.

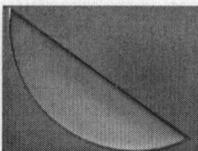


Figure 1 : Top view of a Molybdenum plate as seen after polishing.

Step 3: Laser Cutting

The polished face is cut using a laser ablation facility. Ultra short pulse (1.5×10^{-13} seconds) laser beams can be focused to extremely small spot sizes and then used to ablate materials. Unlike longer pulsed lasers, ultra short pulse lasers allow low-heat machining near the ablation zone. Thus, this laser technology is attractive for precision cutting of any material, such as single crystals, that are sensitive to heat or deformation produced during the cutting process. Figure 2 shows a schematic view of this system.

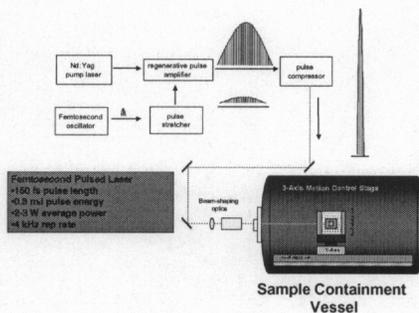


Figure 2 : Schematic view of the laser ablation bench used for cutting the circular shape of the samples.

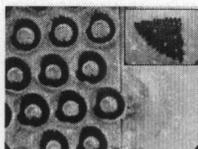


Figure 3 : Top view of a cobalt plate after laser cutting. The diameter of the inner cores are ~60 μm.

Step 4: Thinning

Once the sample has been cored, holes of a known depth (final sample thickness) are drilled to serve as thickness indicators. The sample is inverted and the polishing sequence described above is applied to the opposite surface until the desired thickness indicator is exposed.

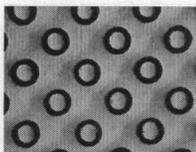


Figure 4 : Top view of a molybdenum plate as seen during the thinning step. The diameter of the inner cores are ~45 μm.

Crystal Quality Preservation

We have performed measurements of the crystal quality of our samples at high-pressure diffraction beamlines ID09A and ID30 of the ESRF. Fig. 5 shows a typical diffraction pattern of a Mo sample after the sample manufacturing process. The typical rocking curves associated with these reflections are less than 0.05°, which is close to the as-received crystal value of 0.02°.

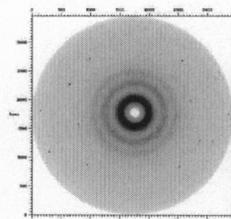


Figure 5 : Typical angle dispersive diffraction pattern of a 45 μm in diameter by 17 μm thick Mo sample. This image was recorded while varying continuously θ from -15° to +15°. Thirteen Mo reflections are seen. The circular shapes of the spots are a first indication of the quality of the crystal.

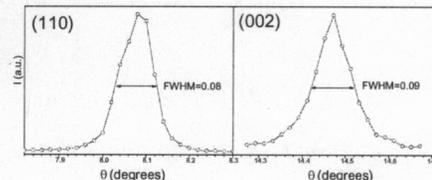


Figure 6 : Rocking curves of a Mo sample at 3.2 GPa recorded at IXS-dedicated beamline ID28 of the ESRF prior to phonon energy measurements. Their full width half maxima (FWHM) are less than 0.1 degree.

Inelastic X-ray Scattering (IXS) Experiments on Single Crystals at High Pressure

Measurements of the pressure dependence of the dynamical properties of Mo have been performed at the IXS-dedicated beamline ID28 of the ESRF to 37 GPa. Figure 6 shows a typical spectrum. The weakness of the quasi-elastic line illustrates the outstanding crystalline quality of the sample.

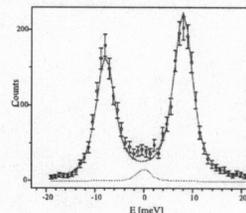


Figure 7 : Typical IXS spectrum recorded at 3.2 GPa. Three peaks are seen: the faint quasi-elastic line (green curve) surrounded by the Stokes and Anti-Stokes peaks (blue curve) corresponding to the creation and annihilation of acoustic phonons.

Conclusion

We have produced single-crystal samples with dimensions compatible with diamond anvil cells, while maintaining orientation and crystallinity. We are currently manufacturing Co samples less than 35 μm in diameter. These dimensions allow us to access experiments in the Mbar range. Working significantly above a Mbar (using helium as a pressure transmitting medium) requires samples less than 20 μm in diameter and less than 10 μm thick. We are presently extending our techniques to produce crystals with these dimensions.

References

- [1] G. Petzow, Metallographic Etching, Am. Soc. for Metals (1978).