

X-ray microtomography under high pressure

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X-ray computed microtomography (CMT) has various applications in geophysics, environmental and material sciences and is useful in imaging internal structures of objects, especially when samples are precious, fragile, or time-consuming to prepare. Data collection is currently conducted at ambient condition or at high temperature. It is expected to open new opportunities for tomography-based research if high pressure and temperature condition can be applied to the sample. As an internal collaborative work at Consortium for Advanced Radiation Sources, The University of Chicago, we have developed a technique for synchrotron microtomographic imaging under high pressure and temperature. The most critical issue in performing tomography experiments under pressure is the limited X-ray access to the sample due to the highly absorbing materials, such as tungsten carbide and tool steel, typically used in generating pressure. To avoid this problem, we employ an opposed-anvil high pressure cell, sometimes referred to as the Drickamer cell, which has some unique features compared to other high pressure apparatus such as diamond anvil cell (DAC) and conventional multi-anvil systems. The sample is compressed between two opposed anvils similar to the DAC. Relatively large volume of the sample chamber permits the use of resistive heating for melting experiments. Another difficulty in performing CMT experiment under pressure is to rotate the cell continuously from 0 to 180 degrees. This issue was addressed by employing two sets of Harmonic Drive gear reducer and thrust bearing on top and bottom side of the Drickamer cell. Diffraction capability and pressure efficiency of the Drickamer cell are examined up to 8 GPa using powdered NaCl as pressure standard material. Quality of X-ray diffraction patterns is good, showing no extra peak except for the NaCl sample and surrounding graphite heating material, benefited by less-absorbing materials in X-ray beam path. Anvils with larger diameters are less efficient in pressure generation, but allow us to apply more loads. As a result, the highest pressures achievable are greater than those using smaller anvils. Efficiency of pressure generation is better with larger tapered angle and smaller tip diameter of anvils, but the high efficiency cell sometimes experiences blow out. To examine the imaging capability, tomography data are collected for the entire cell assembly, including the outer containment ring and the pressure medium, using existing tomography setup at GSECARS beamline at the Advanced Photon Source (APS), USA, without applying any load. The quality of X-ray radiographs depends on the kind of the anvil containment ring because flat field is obtained through the containment ring and pressure medium. The highest noise level is observed with Al-alloy containment ring. Using the Al-alloy containment ring, a commissioning run of CMT was conducted up to 3 GPa. Volume change of sapphire sphere embedded in Fe-S mixture is measured with increasing load. The errors in the volume measurement are 0.3 to 0.7%, mostly due to shadowing by anvil deformation. Using the equation of state of sapphire, pressure is estimated from the volume change, showing general agreement with pressure efficiency obtained from diffraction measurements. Because of the noise, the quality of reconstructed images is not as good as that collected in the conventional tomography setup. These results indicate that high-resolution texture observation requests further improvement, but current setup is still useful to study, for example, density of fluid under pressure. Previous density measurements using X-ray radiography with only one-dimensional data assumes that the sample shape remains unchanged throughout the experiment. Using our technique, this assumption is no longer required and density of melts can directly be inferred from the sample volume even when the molten sample is distorted.