High Pressure-High Temperature studies at the MSPD beamline of Alba synchrotron

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High temperature studies at high pressure in a diamond anvil cell (DAC) are very important to the high pressure community for the study of materials at extreme conditions existing in the interior of the planets or during high-energy density processes [1]. Such studies require precise control in time over the two thermodynamiques variables involved, pressure and temperature, which becomes extremely challenging. In order to achive this, two widely methods have been developed: resistive heating and laser heating. Eventhough the latter one has the advantage of reaching temperatures of thousands of Kelvin one of the drawbacks is the temperature discrepencies and instabilities below 1200K [2]. For this purpose the former method is the most stable and reliable method to be used at high pressure and temperatures abovementioned.

Our crystallographic studies at HP and HT conditions were performed at the Material Science Powder Diffraction Beamline (MPSD) of the Spanish synchrotron, Alba. Powder angle dispersive XRD measurements were performed using a wavelength of 0.4246 Å and x-ray beam of 15x15 μm (FWHM). The 2D XRD patterns were collected on a Rayonix chargecoupled device (CCD) detector with a rocking of (±4-6°) to insure the homogeinity of the Debye rings.

A dedicated vacuum chamber apparatus was developed for collecting HP-HT diffraction data, and which is designed to restrict the effects of convective heating and thermal expansion. The removal of convective heating prevents oxidation of the diamonds at high temperatures. Water cooling restricts the movement of the sample with respect to the X-ray micro-focus. We have used this setup during several experiments at MSPD beamline and have made further changes to the design in order to improve its performance. These changes include: a new spring loaded boron nitride heater, which should allow us to reach temperatures up to 1500+ K in a much faster time than is possible with our current resistive heaters (which are capable of reaching 900 K) and maintain good thermal contact with our sample; new spring loaded C-type thermocouples for good thermal contact at much higher temperatures; and internal water cooling for improved cooling efficiency. Several studies will be highlighted here through results obtained on various scientific cases: study of phase transitions in barocaloric materials, phase diagrams at HP and HT of elemental materials [4]. Finally, future technical developments of HT-HT sample environment of the beamline will be presented.

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