Advanced Photon Source Activity Report for 2003

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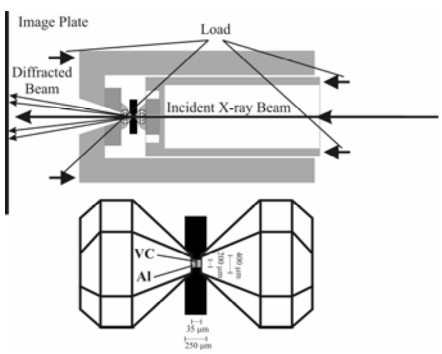
Summary of experiments conducted at *GSECARS*.

During 2003 we visited GSECARS twice for a total of 18 shifts. The data that were collected resulted in one publication and one paper that is in preparation.

1) VC-project.

This project was designed to develop and test a new technique to measure the compression of very hard materials under quasi hydrostatic conditions using Al as solid pressure medium.



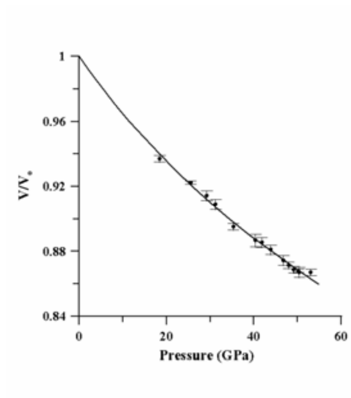


Fig. 1:

Right: Experimental setup of compression measurements in the DAC under quasi hydrostatic conditions using Al as a pressure medium.

Left: Volume change of VC as a function of pressure derived from the cell parameters of the diffraction pattern collected in the diamond anvil cell. Fitting of the data to a Birch-Murnaghan Equation of State resulted in a bulk modulus and pressure derivative of Ko=258±11, K'=4.5±0.6, respectively.

We successfully published the technique and measured the bulk modulus of $VC_{0.85}$ as an example. The technique has been subsequently used to measure compression of other binary and ternary carbides under quasi hydrostatic conditions.

Liermann, H. P., Singh, A. K., Saxena, S. K., Manoun B., Prakapenka V. B., Shen G. (2004). Compression behavior of $VC_{0.85}$ up to 53 GPa. International Journal of Refractory Metals and Hard Materials, 22, 129-132.

During 2003 we also conducted experiments to determine the compression strength of $VC_{0.85}$. Data are currently being processed and will be published in 2005.

2) EoS project for P determination at high T in a resistive heated cell (13 IDD 03 87, GUP1450).

Introduction

This is an ongoing project to determine the EoS of internal standards such as Pt, Fe and MgO at simultaneous high-pressure and -temperature in the resistively heated DAC. In the last three years we have successfully developed a resistively heated DAC that can reach up to 1 Mbar and 1500 K. We continue to collect data to develop a comprehensive dataset that can be used to determine pressures at high-temperatures using the cell parameter of multiple standards.

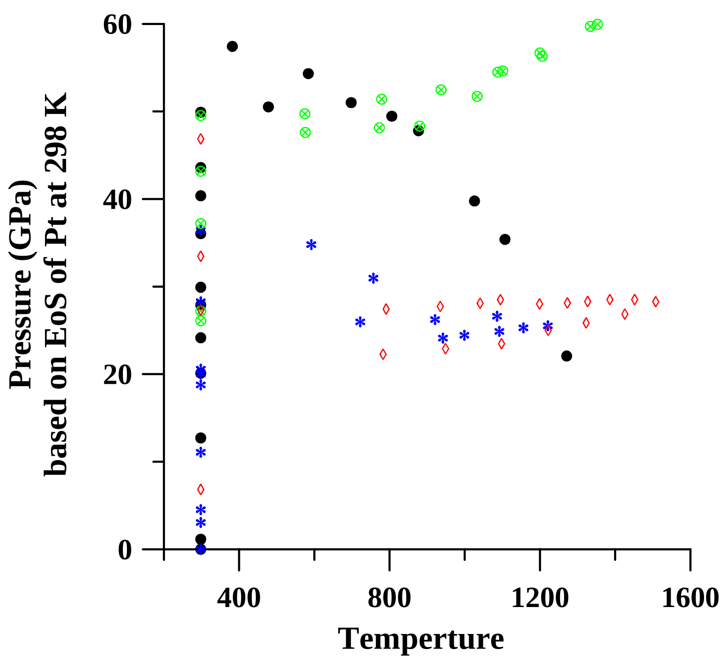


Fig. 2: Approximate pressure and temperature space investigated during in situ X-ray diffraction experiments in the resistive heated diamond cell (DAC).

Results

During 2003 we conducted X-ray diffraction experiments with Pt and Fe up to ~ 60 GPa and ~1500 K (Fig. 2). The data have been processed and cell parameter refined, but additional data are needed to cover the PT space to 1 Mbar and 1500 K.

3) Enstatite + H_2O (13 IDD 03 88, GUP1502)

Introduction

$MgSiO_3$ and its polymorphs are major constituents of the Earth's mantle and its phase stability has been studied extensively with the multi-anvil press up to the perovskite transition [1]. Because evidence for the presence of H_2O in the mantle is mounting [2-3] it is necessary to reevaluate the effect of water on the $MgSiO_3$ phase diagram. Previous research in this system has been restricted to quenched experiments in the multi-anvil apparatus at 15 GPa and 1500 °C [4] and in the laser heated DAC at 15-19.3 GPa and 1200 +/- 300 °C [5]. The results show that $MgSiO_3$ clino-enstatite is stable in the presence of H_2O below the transition zone, but transforms to hydrous or super hydrous phase B, plus some hydrate β-phase and stichovite at higher P-, T-conditions. There have been many experiments concerning the hydration and dehydration of Mg_2SiO_4, but little has been done on the system $MgSiO_3$, in part due to the breakdown of high-P clino-enstatite to β-phase with stichovite and the formation of various hydrous phases and their subsequent breakdowns to perovskite, periclase, and water [6]. However, the overall stability of clino-enstatite, phase B or hydrate β- (or γ-) phases and the transition to perovskite have not been established.

Experimental

In 2003 we conducted two experiments. In both experiments we mixed pure natural enstatite with small amount of H_2O and filled it in a resistive heated DAC. The presents of H_2O was established by Raman spectroscopy. The during the experiments the sample was pressurized to 30 GPa and heated to 1400 K for 2-3 hr at a time. During pressurization and heating X-ray diffraction patterns were collected in 10 min intervals.

Results and Discussion

Initial evaluation of the diffraction data indicates that enstatite transforms into an amorphous phase that slowly reacts to form a new phase. However, the duration of the heating part of the experiment was insufficient to induce enough crystallization to obtain a high quality diffraction pattern of the new phase. The data are currently awaiting more detailed processing and evaluation. We are also planning to collect Raman spectra of the quenched sample. After this detailed evaluation we like to repeat the experiment and combine the resistive heating setup that is currently been developed for ID-B at sector 16 with laser heating.

Acknowledgment

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References

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