ESRF	**Experiment title:** Development of a new high pressure (30 GPa) high temperature (2500 K) apparatus for X-ray studies	**Experiment number:** HS2532
Beamline: ID27	**Date of experiment:** from: **14/05/2005 to 17/05/2005 and 30/11/2005 to 04/12/2005**	**Date of report:** 10/01/06
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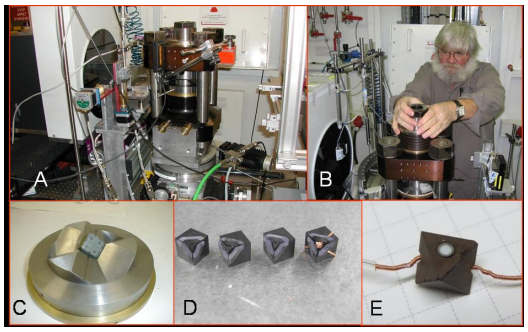
Introduction

All current applications of the PE press use an opposed anvils geometry. Experience shows that in such a geometry, the pressure and temperature range is limited to ~ 10 GPa and 2200 K, for various reasons. To overcome these limitations, we have designed a new device for *in situ* X-ray diffraction studies under HPHT. The system consists of a 450-ton V7 Paris-Edinburgh press combined with a Stony Brook "T-cup" multianvil stage. The V7 has a capacity of 450 tons, i.e. almost twice as the standard presses, but still a weight of less than 80 kg. The ~ 20% larger overall dimensions allow to accommodate a two-stage multi-anvil system similar to the Stony Brook "T-cup system" which operates routinely to 30 GPa and 2500 K. Figure 1 shows the set-up and its installation at ID27 (picture A,B). The first stage is a steel cylinder split into six parts enclosing a cubic cavity which contains the second stage anvil assembly (picture C). This second stage is assembled outside the press and consists of eight c-BN cubes (transparent to X-rays) of 10 mm edge length, separated by gaskets and spacers (picture D). Each cube has one truncated corner to give a triangular face; the eight truncations form an octahedral cavity in which the pressure medium is compressed. The high pressure set-up is hence an octahedron with a cylindrical hole where a cylindrical foil Re furnace, the electrically end plugs and the thermocouple are placed (picture E).



First year results

During the first year of this long term project we demonstrated :

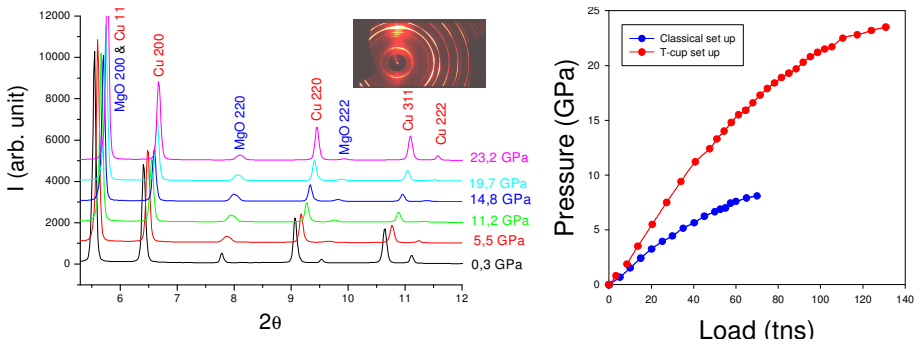
(i) The extreme mobilit of the setup. The press can be transported easily between laboratories and installed in the ID27 experimental hutch within ~2 hours.

(ii) The considerable advantage of being able to work with several presses simultaneously, i.e. carry out experiments with one cell whereas other presses are being prepared for the following measurements. It should be noted that multi-anvil systems of any kind need rather long decompression times of the order of several hours, which leads to a considerable loss of beamtime. In one run we have used two V7 cells simultaneously and hence proved the feasibility and usefulness of the approach.

(iii) The device has been adapted for the use with Sollers slits and large area CCD detectors for angle dispersive X-ray diffraction. This Soller slit system permits a significant improvement of the signal-to-background ratio and provides clean diffraction patterns (*Cf. Infra*).

Technical part

A considerable part of this first year beamtime was dedicated to testing the performance of the system under realistic conditions. Angle-dispersive powder diffraction with a MAR 345 image plate detector was carried out to test the pressure generating capacity for various gasket designs (nature and geometry of gaskets as well as the nature of the octahedron). For these experiments, the sample was simply a mixture of copper and magnesium oxide (typical spectra up to 23.2 GPa are shown in the figure) and the pressure efficiency (Cf. figure) was determined from the lattice constant of these compounds using their well-known equations of state (estimated error ~ 0.1 GPa). These measurements indicate that (i) The pressure efficiency is higher for Teflon gaskets close to the truncation than with pyrophyllite gaskets, or for a boron-epoxy gaskets, or even with no gasket. Teflon is however temperature-limited (melting point: ~ 600 K), whereas pyrophyllite gaskets can be useful in very high-temperature experiments. (ii) A 30% increase of the pressure/load capacity can be achieved by reducing the size of the gasket to 1.5 mm (instead of 2.3 mm). (iii) MgO octahedra seem to be slightly less efficient than octahedra made of amorphous boron epoxy. (iv) In the run using MgO and Cu samples, the differences between the observed and fitted d_{hkl} at high pressures were very small, which indicates homogeneous stress conditions. Compared to previous measurements done with the "classical" P.E. cell, one can conclude that the T-cup geometry helps considerably in providing a hydrostatic stress environment for samples at room temperature.



Finally, these experiments showed that the quality of diffraction patterns which can be obtained in the range 0 – 25 GPa and 300 - 1500 K is excellent, and that the data quality is comparable to that obtained with the standard opposed-anvil setup in the Paris-Edinburgh press.

Scientific part

We carried out a number of experiments which demonstrated the potential of this device. During the most recent run (30.11-4.12), the synthesis of potentially superhard phases in the ternary B-C-N system was studied *in situ* at HP-HT. Another experiment was devoted to the liquid Fe-S-Si immiscible system which is important for understanding the differentiation processes in the planets and the composition of their cores. The quality of the data was excellent, despite the sample being composed of low-Z elements, or being in the liquid state. This data are currently being analized and results in full will be presented in a complementary report.

Future beamtime

We intend to devote the next runs to test new shapes of cubes in order to reach higher pressure. A new assembly will be tested to prevent the frequent breakage of thermocouples during compression and to reach higher temperatures. New furnaces made of TiC will be used. We are aiming to investigate the efficacity of a new cooling system for the 1st-stage anvils as well. For the scientific programme, *in situ* studies of phase formation in the B-C-N system under HP-HT will be continued.

Publications arising from these measurements

- Y. Le Godec, G. Hamel, D. Martinez-Garcia, T. Hammouda, V.L. Solozhenko and S. Klotz, *High Pressure Research* **25**, 243 (2005).

- Y. Le Godec, G. Hamel, M. Mezouar, W. Crichton, V.L.Solozhenko *et al.*, to be submitted to *Journal of Synchrotron Radiation.*