ESRF	**Experiment title:** Development of a new high pressure (30 GPa) high temperature (2500 K) apparatus for X-ray studies	**Experiment number**: HS2335
Beamline: ID30	**Date of experiment**: from: 08/10/03 to: 11/10/03	**Date of report**: 01/03/04
Shifts: 18	**Local contact(s): Wilson Crichton.**	*Received at ESRF:*

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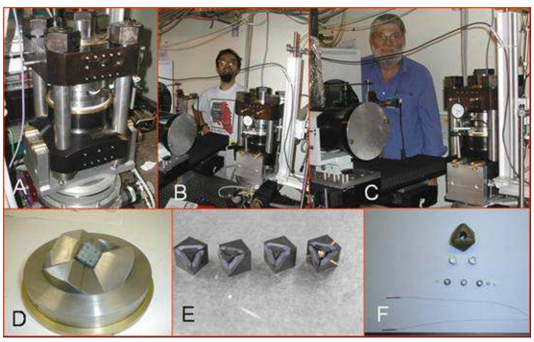
Preliminary report on HS2335

We had hoped to be able to report results from a first test experiment using our new Paris Edinburgh V7 cell at ESRF. In fact, we had obtained 18 shifts for this experiment and we had scheduled beam time for 3 days in November 8–11. Concerning the last 3 days, reconstruction of ID30 meant that our next experiment has had to be postponed until the beginning of July 2004. That's why we present in this preliminary report only some results that arise from these first 3 days of beamtime. We will forward a complementary report straight after the next experiment scheduled in July.

Introduction

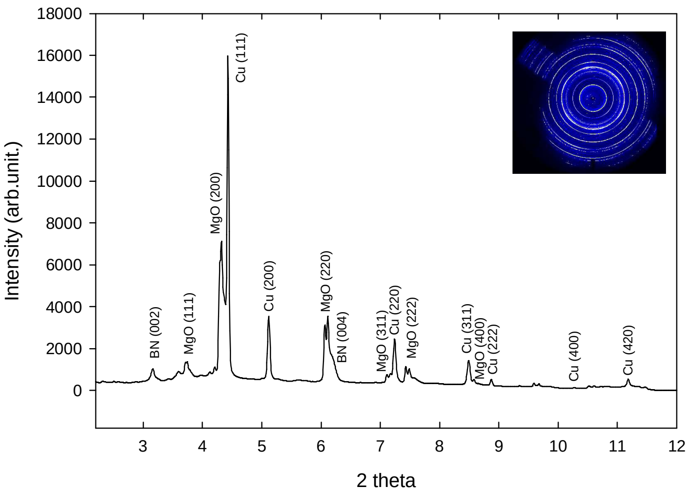
To date the Paris Edinburgh (PE) press is commonly in use at several ESRF beamlines (ID30 for diffraction experiments, BM29 and ID24 for high pressure - high temperature (HPHT) EXAFS experiments, ID15B for Compton scattering experiments and very recently ID16 for inelastic X-ray diffraction experiments). It is at the origin of many spectacular results obtained at the ESRF giving rise to at least a hundred publications during the last five years.

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 ~ 8 GPa and 2200 K, for various reasons. To overcome these limitations, we have designed a new Paris-Edinburgh press (V7) for *in situ* X-ray diffraction studies under HPHT. 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 (picture A) and its installation at ID30 (picture A,B,C). The first stage is a steel cylinder split into six parts enclosing a cubic cavity which contains the second stage anvil assembly (picture D). This second stage is assembled outside the press and consists of eight c-BN cubes (transparent to X-ray) of 10 mm edge length, separated by gaskets and spacers (picture E). 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 the tube graphite furnace, the electrically end plugs and the thermocouple are placed (picture F).



Preliminary Results

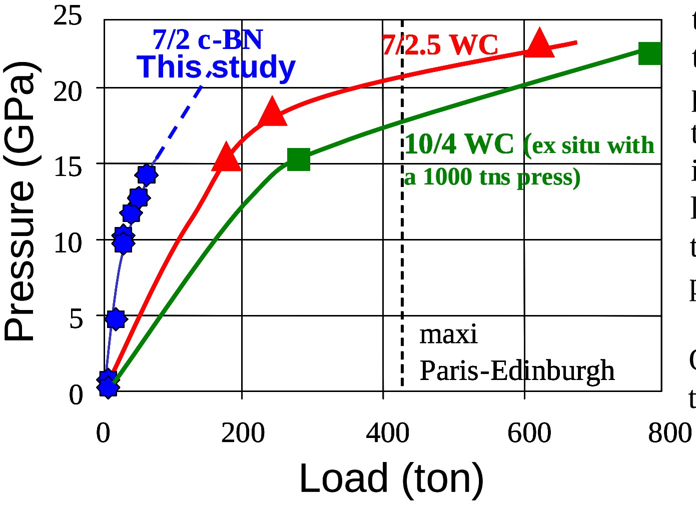
During this first-time experiment, we demonstrated (i) that this press can be transported easily between laboratories and installed in the ID30 experimental hutch within ~2 hours, (ii) the considerable advantage of being able to work with several presses simultaneously, i.e. carry out experiments with 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 our experiment, we have indeed operated using two V7 cells simultaneously and hence proved the feasibility and usefulness of the approach.



Angle-dispersive powder diffraction with MAR 345 image plate detector has been used to perform several tests (6 in total) of pressure generating capacity for various system designs (especially concerning the nature and the shape of gaskets as well as the nature of the octahedron). For these first tests, the sample was a mixture of copper, hexagonal boron nitride and magnesium oxide (a typical spectrum at 9.8 GPa is shown in the figure) and the pressure was determined from the lattice constant of these compounds using their well-known equation of state (We estimated that in our working (P,T) domain, the error in pressure measurement does not exceed 0.1 GPa).

We've made the following inferences: (i) The pressure efficiency is higher with Teflon gaskets near the truncation than with pyrophyllite gaskets, or with mixture of amorphous boron and epoxy resin (4:1 by weight) gaskets, or even with no gasket (we also tried this possibility). (ii) The improvement is greatest if the width of the gaskets is reduced to 1.9 mm (instead of 2.3 mm, its theoretical value). (iii) MgO octahedron seems to be slightly more efficient than amorphous boron epoxy octahedron. (iv) In the h-BN, 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 the sample at room temperature.

At high pressure, the transformation from h-BN to w-BN was recognized at about 15 GPa, as reported in the previous literature. We decided to stop the test at this pressure because the volume collapse induced by this transformation is very large and could have misaligned the cubes. So further experiments are required in order to probe the high-pressure limit of our cell. By looking at the opposite figure which shows the generated pressure in the present experiment as a function of the applied load, we can also conclude that replacing the common tungsten carbide cubes with c-BN anvils leads to a higher pressure efficiency.



Our last 3 days of beamtime will also be devoted to test the efficiency of our temperature generation system at high pressure.