# Experiment Report Form

| ESRF | **Experiment title:** A new pressure scale for ultrahigh pressure-temperature research | **Experiment number**: HS-2287 |
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| **Beamline**: ID9A | **Date of experiment**: from: 26-02-2004 to: 02-03-2004 | **Date of report**: 29-03-2004 |
| **Shifts:** 15 | **Local contact(s)**: Dr. Michael Hanfland | *Received at ESRF:* |

**Names and affiliations of applicants** (* indicates experimentalists):

Andrew P. Jephcoat*, Diamond Light Source Rutherford Appleton Laboratory, Chilton, Didcot, OX11 0QX, UK & University of Oxford, Department of Earth Sciences, Parks Road, Oxford OX1 3PR, UK.

Annette K. Kleppe*, University of Oxford, Department of Earth Sciences, Parks Road, Oxford OX1 3PR, UK.

Michael Drakopoulos*, Diamond Light Source, Rutherford Appleton Laboratory, Chilton, Didcot, OX11 0QX, UK.

## Report:

In experiment HS2287 (completed only a few weeks before this report) we obtained high-pressure Raman spectroscopic measurements of the $E_{2g}$ phonon in hcp-structured zinc with *simultaneous* x-ray diffraction patterns recorded on an image plate in order to provide a secondary pressure scale based on metal-only properties. We had recently shown that Raman spectroscopic measurements can be used to study k=0 phonons in various metals at pressure in the 100-GPa regime using the diamond anvil cell (DAC) [1-8].

During HS2287 beamtime, we obtained structural data (unit-cell volume) on Zn in solid He vs phonon frequency to over 100GPa (Fig. 1-3). We also measured a "sandwich" of Zn and Cu under nonhydrostatic conditions to 125 GPa and Raman spectra were obtained for Zn to the highest pressure. This experiment represents the *first use of an on-line raman system at ID9* and demonstrated its immediate success and potential for future work. The raman optics were part of an in-house system made available for special research only by permission of M. Hanfland. The raman system proved to be sensitive enough to detect the phonon mode to the highest pressures – alignment of the system under on-line conditions required careful work. Our DAC was loaded at Oxford with helium and in both the hydrostatic and nonhydrostatic run (the latter loaded at ESRF) sample sizes were of the order of 0.010 mm requiring the finest collimation of the x-ray beam. Most patterns were obtained without contamination of gasket reflections.

Of several available high-pressure scales, the standard ruby scale is the most widely used secondary scale in high-pressure research with the DAC. First established by Piermarini et al., in 1975 [9] and later extended to 100+ GPa under non-hydrostatic conditions [10], it continues to provide the basis for many experiments in the 150 GPa range and higher. But, ruby has distinct disadvantages in ultrahigh pressure experiments: the width of the R1-R2 fluorescence region is notoriously pressure sensitive and practically limits use under non-hydrostatic conditions above 100 GPa. Errors in location of the emission maximum contribute a correspondingly large error in the pressure calculated. As shown in Fig. 2 for Zn in He**,** the Zn phonon broadened much less with pressure than the R1 emission of ruby.

We are in the process of correlating the Zn shock-wave EOS (that provides link between volume and pressure) with the x-ray volumes measured in the experiment. Our result will then provide the basis for a new pressure scale with distinct optical advantages for both P and ultimately (with further work) P-T experiments.

We did not complete a similar set of experiments on rhenium because of the time taken for this first set of measurements. It is useful to make measurements with different metals to eliminate any systematic errors in the reduction of shock-wave data [10]. We will apply for a continuation experiment.

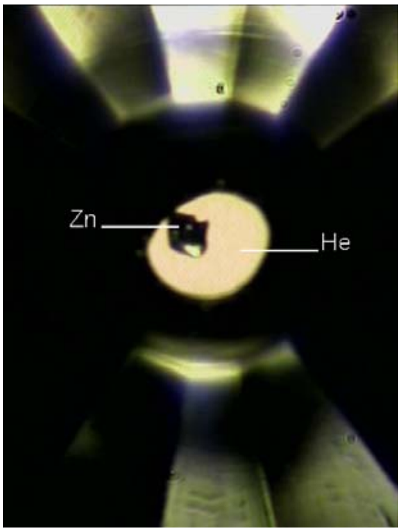


**Fig. 1: View into the DAC through the cylinder diamond onto the Zn sample in He at ~105.5 GPa. The central flat of the diamonds has a diameter of 100 µm, the Zn foil is 12 x 12 µm in size and 2.5 µm thick, and the hole size is ~35 x 60 µm.**

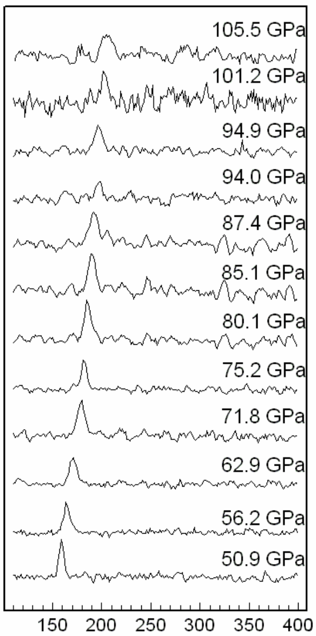


**Fig. 2: Selected Raman spectra (measured on-line at ID9) of the $E_{2g}$ mode of Zn as a function of increasing pressure.**

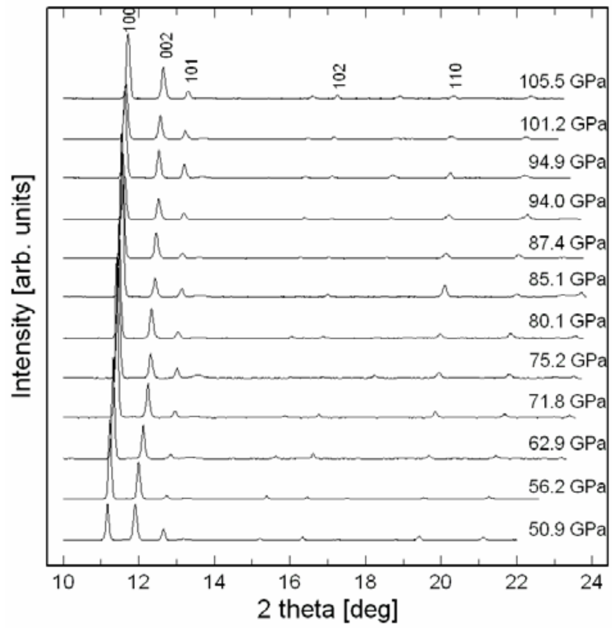


**Fig 3: High-pressure synchrotron powder X-ray diffraction patterns of hcp Zn foil (Fig.1) with He as a pressure-transmitting medium at 300 K. A 0.02 mm pinhole was used before the DAC.**


References

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