ESRF	**Experiment title: X-ray diffraction of diamond and cubic BN to 1 Mbar and 1000 K : equation of state and thermodynamics at high temperature**	**Experiment number**: HS-2514
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Report:

Experiments: The aim of this proposal was to measure the high pressure and high temperature equation of state (EoS) of diamond and c-BN, under carefully controlled and measured pressure and temperature conditions. In four diamond anvil cells, diamond, c-BN, a metal (gold or copper), the pressure gauge ($SrB_4O_7:Sm^{2+}$) have been loaded in neon (**see figure 1**). High temperatures were generated using resistively heated diamond anvil cells (global heating up to 750 K coupled to local heating up to 950 K). We have scanned the following pressure and temperature ranges: 0-80 GPa and 300-750 K. Lattice parameters of diamond **(Figure 2**), c-BN (**Figure 3**), gold or copper, and neon (**Figure 4**) have been measured for each pressure and temperature steps at the ESRF ID27 (first experiment on this new beamline !). This has been done in parallel with the measurement of the Raman phonon of c-BN and diamond under the same P-T conditions, at the ESRF or in separate experiments in the laboratories of the authors. Out of the 5 pressure cells used in these experiments, three prematurally failed before achieving the P-T conditions that we aimed (100 GPa-1000 K), due to diamond anvil failure. This is more likely the result of neon diffusion inside the anvil tip at high P and T. This behavior is not predictible (one of our cell could sustain 80 GPa at 600 K without failure while another one failed at 50 GPa and 500 K).

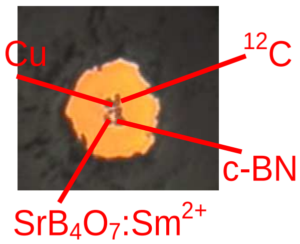

Figure 1: Photograph of a typical pressure chamber (diameter 50µm).

Data recorded: The cold (298 K) compression curves we measured are in excellent agreement with available published data (in [1] for C, in [2] for c-BN, but the latter reference contains only a few data between 0 and 80 GPa). These reference curves allowed us to evaluate thermal expansion coefficients of c-BN and C with an accuracy not achieved so far.

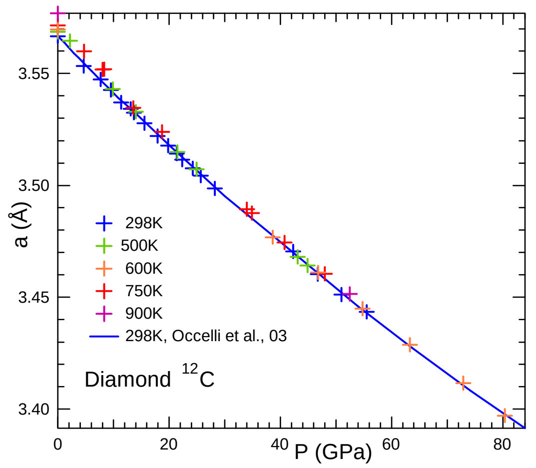

Figure 2: Equation of state of diamond.

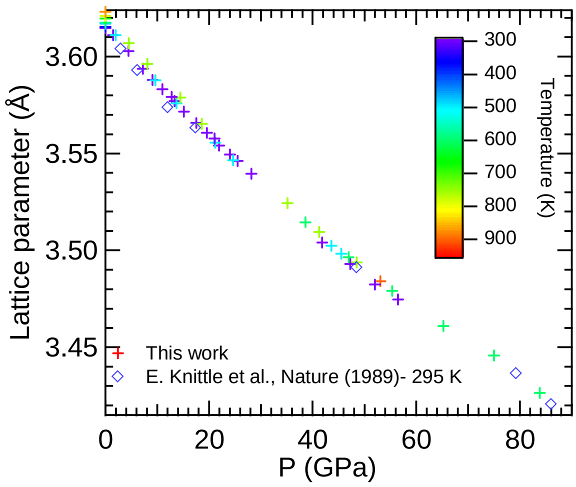

Figure 3 : Equation of state of c-BN

For C, at P>30 GPa, the thermal expansion coefficient was much lower than calculated by density functional perturbation theory [3]. On Figure 2, it can be seen that at high pressure, the lattice parameter is almost insensitive to temperature in the scanned range. This observation is strengthened by Raman measurements. This very low thermal expansion coefficient could be explained by negative Grüneisen coefficients for TA branches. By contrast, there is still a measurable thermal expansion in c-BN at 50 GPa. The analysis shows that a Mie-Grüneisen model with a volume dependent Grüneisen parameter (quasi-harmonic approximation) satisfactorily reproduce our data. In both cases, data at higher P and T would be valuable in order to extract the precise variations of thermodynamic Gruneisen parameter.

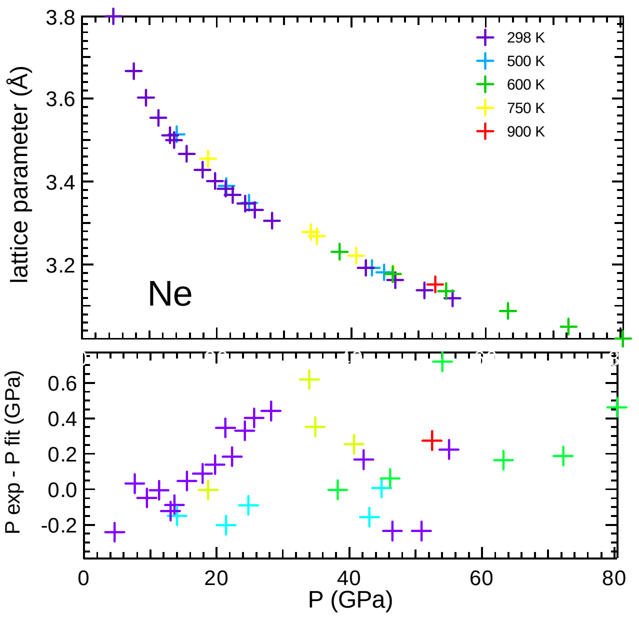

Figure 4: top: EoS of neon; bottom: residuals after Fitting of this EoS by a Mie-Grüneisen formulation.

As neon was used as the pressure transmitting media, we could also extract the high pressure and high temperature EoS of neon. The latter could be very well fitted with a Debye-Grüneisen formulation [4], as evidenced in Figure 4: the residuals after fitting the experimental data are small and do not depend on the temperature. Since our experiments have been performed at temperatures large compared to the Debye temperature of neon (75 K), we are confident that this EoS can be extrapoled at higher temperatures without any large errors. Also, this expected behavior validates our high pressure and high temperature metrology. Using this equation of state, neon can be used as a high pressure and high temperature pressure gauge in diamond anvil cells.

References:

[1] Occelli et al., Nature Mat., 2, p. 151, 2003

[2] Knittle et al., 337, p. 349, Nature, 1989

[3] Xie et al., Phys. Rev. B, 60, p. 9444, 1999

[4] Finger et al., Appl. Phys. Lett., 39, P. 892, 1981