ESRF	**Experiment title:** Pressure measurements at high temperature in X-Ray diffraction studies: periclase (MgO) and platinum (Pt) as secondary standards	**Experiment number:** HS-503
Beamline: ID30	**Date of Experiment:** from: 01-27-98 to: 02-03-98	**Date of Report:** 08-27-98
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Report:

We present here high pressure (HP) and high temperature (HT) volume measurements, carried out in-situ on a mixture of periclase and platinum, in a laser-heated diamond-anvil cell. The compressional behaviors of these materials have been cross checked between 0 and 50 GPa and 300 and 2400 K. At ambient T, we showed that existing data on MgO and Pt incompressibilities are mutually consistent. The HP/HT Equation of State (EoS) of MgO has also been determined.

The aims of this experiment were:

(1) to confront the HP / ambient T compressional behavior of periclase and platinum. Indeed, platinum is used as a pressure calibrant by our experimental team (see reference (1)). We wanted to confirm by static compression measurements the only available EoS of platinum, which has been calculated by reduction shock-wave Hugoniot data (see Jamieson et al., 1982, reference (2)).

(2) to determine the HP/HT EoS of periclase. This EoS is of first interest to geophysics because MgO is the pure magnesian endmember of magnesiowüstite, which is the second most abundant material in the Earth's lower mantle. This EoS has never been experimentally measured at high temperature in the pressure range of the lower mantle (25-120 GPa).

The experimental setup was the same as previously used by the same team (see reference (1)), on the ID30 high pressure beamline: both monochromatic X-Ray beam and CO_2-laser beam are focused on the center of a diamond-anvil cell (20x10 μm for the X-Ray spot, and around 80x80 μm for the laser-heated spot). The diffracted signal is collected during heating on an image plate (in around 30s to 1 min), and readed using the FastScan detector.

The X-Ray diffraction spectra, after integration, have been treated with the GSAS program package. Lebail refinement allowed an accurate determination of the volume of each phase present in the diamond-anvil cell: Pt, MgO, and the pressure transmitting media: a mixture of argon and nitrogen (see figure 1). Relative uncertainties in Pt and MgO volumes were of the order of 0.05 %. Temperatures have been determined by the analysis of thermal emission spectra recorded during the acquisition of the X-Ray diffracted signal.

At ambient T, the elastic parameters of MgO have already been widely studied (see reference (3), for example). They are used to obtain the incompressibility of platinum: $K_T(P = 0, T = 300K) = 266$ GPa; $K'_T(P = 0, T = 300K) = 6.2$, with a third-order Birch-Murnaghan Equation of State. These parameters are in excellent agreement with the EoS derived by Jamieson et al, 1982, (2).

Since no data under HP and HT (except shock-wave studies) is available for MgO, the inverse approach has been used to further analyse the high temperature measurements. Platinum has been used as a pressure calibrant, using the Jamieson et al. EoS with has been showed to be reliable, at least at ambient temperature. Both manual (see figure 2) and numerical global inversion of the P-V-T data of MgO give consistent results concerning K_T, α (the thermal expansion coefficient) and their first pressure and temperature derivatives. These parameters are:

$\alpha(P,T) = (3.0(3).10^{-5} + 1.3(5).10^{-8}\,T)(1 - 0.024(2)\,P + 2.2.(1.0)10^{-4}\,P^2)$ (P in GPa, T in K) in K^{-1}, and $K_T(P = 0,T) \simeq 265 + (\partial K_T/\partial T)_P (T - T_0)$ with $T_0 = 300$ K and $(\partial K_T/\partial T)_P (P = 0) = -0.023(5)$ GPa.K^{-1}, and $K'_T(P = 0, T = 300K) = 4.2$.

However, the pressure range scanned appears to be too weak to constrain temperature derivative of K'_T (4).

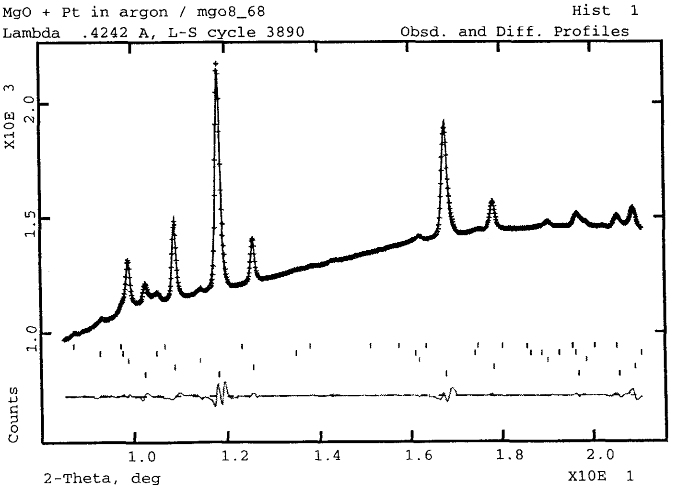

Figure 1: Typical X-Ray diffraction pattern after integration and LeBail refinement. Present phases are MgO, Pt, fcc-Ar, hcp-Ar and δ-N_2. P/T conditions are 2110 K and 24 GPa.

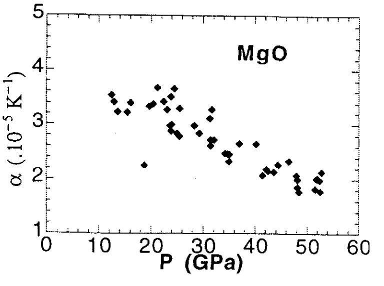

Figure 2: Thermal expansion cœfficient of MgO versus P.

References:

(1) G. Fiquet et al., Phys. Earth. Planet. Inter, 105, 21-31, 1998;

(2) J.C. Jamieson et al., in High Pressure Research in geophysics, edited by S. Akimoto and M. Manghnani, Reidel, Boston, pp 27-48, 1982;

(3) Duffy et al., Phys. Rev. Lett., 74, 8, 1371-1374, 1995;

(4) Dewaele et al., P-V-T Equation of state of magnesiowüstites under lower mantle conditions, in prep.