ESRF	**Experiment title:** Sequence of phase transitions in KCl measured in a toroidal diamond anvil cell up to 600 GPa	**Experiment number**: HC-2783
Beamline: ID27	**Date of experiment**: from: 17/04/2016 to: 22/04/2016	**Date of report**: 20/02/2017
Shifts: 12	**Local contact(s)**: M. Mezouar	*Received at ESRF:*

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Report:

The aim of the proposal were: (i) to reproduce the test made in ME-1380, where a pressure of 590 GPa was reached in a gold sample using a toroidal shape of a diamond anvil, with the use of a pressure medium; (ii) to record the X-ray diffraction signal of lighter KCl in the same device (iii) to determine the sequence of phase transformations under ultra-high pressure in KCl, CsCl and CsBr. Phase transitions to lower symmetry phases (beyond the cubic CsCl-*Pm-3m* structure) have been predicted for these compounds.

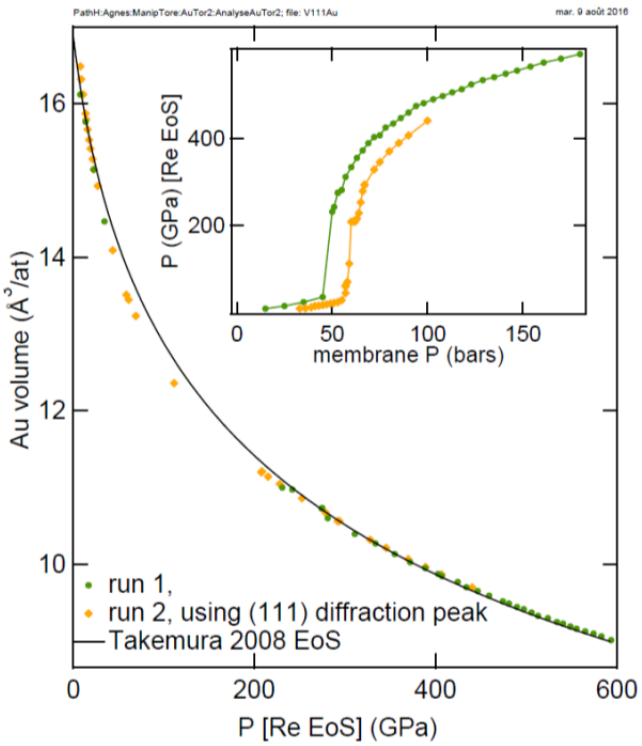
The conditions of the experiments are summarized in **Table 1**. For all these runs, the monochromatic x-ray diffraction signal of the sample assembly was recorded on a MAR-CCD detector. The pressure was estimated using rhenium gasket equation of state [1] in the first two runs; in the CDMX7 run, the pressure was measured using a ruby gauge; in the CDMX11 run, the pressure was measured using a tungsten sample equation of state.

Run name	Diamond tip & diameter	Sample	Pressure medium	Pressure range (GPa)
CDMX16	Toroidal (16 μm)	Au	Ar	0-150
CDMX26	Toroidal (16 μm)	Au	KCl	0-440
CDMX7	Standard (300 μm)	CsCl, CsBr	He	0-34
CDMX11	Standard (100 μm)	CsCl, CsBr	He	24-141

Table 1: Conditions of the experimental runs.

Two diamond anvils equipped with toroidal diamonds were prepared for this experiment. The first run failed at 150 GPa because of an unstability in the gasket due to a slight misalignment. In a second run, a pressure of 440 GPa was reached, significantly higher than the pressure achieved in standard diamond anvil cells (300 to 350 GPa). This confirms the potential of toroidal anvils to reach ultra-high static pressures. The equation of state of gold has been measured (see **Figure 1**). Unfortunately, the X-ray diffraction signal of KCl was too low to be distinghished from the gasket signal above ~200 GPa in this run. Therefore, the high pressure phase transformations of this alkali halide could not be followed.

The last two runs were dedicated to the study of heavier alkali halides (CsCl, CsBr) with standard diamond anvils. Helium pressure medium provided quasi-hydrostatic pressurization conditions. CsCl sample were single crystals while CsBr sample were powder samples. A transition to a *Pmma* phase around 60 GPa, as predicted in Ref. [2], was observed for CsBr (see **Figure 2**). In CsCl, a slight distorsion from a cubic lattice is observed above 70 GPa. This could be attributed to the phase transition predicted in this range, also to a *Pmma* structure [2]. However, the distorsion remains small and the higher pressure phase transformation is not observed.



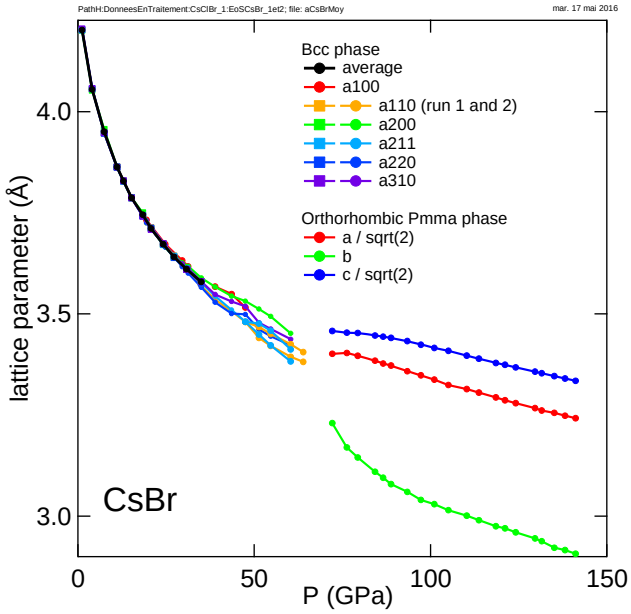


Figure 1 (left): Equation of state (EoS) of gold measured in this experiment as well as during ME-1380. The measurements are reproducible and in good agreement with the extrapolation of the EoS of gold measured at lower pressure [3]. **Inset:** Pressure measured at the center of the diamond tip, using rhenium gasket equation of state, vs membrane pressure.
Figure 2 (right): Lattice parameter of CsBr vs pressure. Below 60 GPa, the data corresponding to different X-ray diffraction lines for the cubic *Pm-3m* are plotted. Above 65 GPa, the lattice parameters corresponding to a *Pmma* orthorhombic phase are plotted.

Toroidal anvils are likely to open a new avenue for the study of statically compressed matter in the 300-600 GPa range. The results summarized here are will be published shortly. The phase transition measured here for heavy alkali halides (cubic *Pm-3m* -> orthorhombic *Pmma*) may be representative of similar simple ionic solid materials behaviour under high compression.

References:

[1] S. Anzellini et al., Equation of state of rhenium and application for ultra-high pressure calibration, J. Appl. Phys. 2014
[2] S. Wei et al., High-pressure phase transition of cesium chloride and cesium bromide, Phys. Chem. Chem. Phys. 16, 17924, 2014.
[3] K. Takemura and A. Dewaele, “Isothermal equation of state for gold with a He-pressure medium”, Phys. Rev. B 78, 104119, 1-13, 2008