



	Experiment title: Structural properties of HF and DF at high pressure	Experiment number: HS2294
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Shifts:	Local contact(s): Dr. Michael Hanfland	<i>Received at ESRF:</i>
Names and affiliations of applicants (* indicates experimentalists): G. Weck, CEA/DAM Bruyères-le-Châtel, France P. Loubeyre, CEA/DAM Bruyères-le-Châtel, France P. Lalanne, CEA/DAM Ripault, France P. Hebert, CEA/DAM Ripault, France Christian Henriquet, ESRF.		

Report:

Introduction

Hydrogen fluoride is the simplest molecule that displays a strong intermolecular hydrogen bond. For this reason HF, has been widely studied at ambient pressure in its gas and liquid phases. Particularly, it has been shown that HF molecules form, in the liquid phases a structure of zig-zag chains of 6 or 7 molecules [1]. This one dimension zig-zag chain structure has been also observed in the solid at low temperature (HF crystallizes in the C^{12}_{2v} structure) [2]. To extend the comprehension of the hydrogen bond, pressure is a unique tool since it allows to continuously change the distance between molecules. But, because HF is strongly corrosive only one experiment has been performed at high pressure and low temperature by Raman spectroscopy [3]. The observed Raman modes confirm the C^{12}_{2v} structure for solid HF up to 6 GPa. Above this pressure, strong evidences of transition to a symmetric H-bond structure are observed. The aim of the present experiment was to determine the structure of the solid below and above 6 GPa, to confirm the transition to a symmetric H-bond structure and to determine the equation of state of HF up to 40 GPa.

Experimental Method

Because HF is a dangerous gas, special care must be paid for the loading of the sample. The loading was performed at the ID16 beamline, following a similar procedure as the one that had been implemented for loading a vessel for a previous inelastic experiment [4]. The loading system and procedure described below was developed in collaboration with Christian Henriquet and validated by the ESRF safety group.

To pressurize the sample we use a membrane diamond anvil cell with high X-ray aperture ($2\theta_{\max} = \pm 37^\circ$, $\varphi = 360^\circ$). The sample is confined between two diamonds and a Rhenium gasket with a hole drilled in it. In order to circumvent any possible reaction a thin layer of Platinum (2-3 μm) is deposited on the gasket by plasma projection. To load the sample we fill the “loading cavity” with HF. Two stainless steel supports and an o-ring gasket define the loading cavity around the diamond. The cell is covered with ice in order to condensate HF. A force is applied with the metallic membrane to close the loading cavity and the sample chamber.

e.g.: the pressure needed to close the loading chamber (10-20 bars) is lower than the pressure needed to close the sample chamber (~ 50 bars).

During the loading, we have experienced great difficulties to condensate HF in the loading chamber. For this reason, only one of the two cells prepared for the experiment could be loaded. Moreover, even for this cell we couldn't avoid a strong shrinkage of the sample chamber (the sample diameter was about 30 μm after the loading).

The experiment was done on the ID9 beamline. We performed angle dispersive X-ray diffraction with a focussed monochromatic beam at $E = 29.7$ keV. The X-ray diffraction images were collected with an on-line image plate detector (MAR3450).

Results and Conclusion

Twenty X-ray diffraction patterns were collected for pressures in between 0.6 GPa and 11 GPa. Unfortunately, due to the small sample size all spectra exhibit diffraction rings of platinum. The analysis of the patterns and complementary Raman spectroscopy measurements performed in our laboratory, indicate that the HF sample had reacted probably with platinum forming a platinum fluoride ionic compound. This result is surprising since Platinum is known to be chemically compatible with HF. The 22 observed diffraction lines are well indexed in an orthorhombic cell with possible space group $P4_2NM$ or $P4_2/MNM$. The EOS of the compound is presented in figure 1 and compared to the equation of state of Platinum and water, showing that the bulk modulus of the compound is by about 10 times higher than the bulk modulus of water.

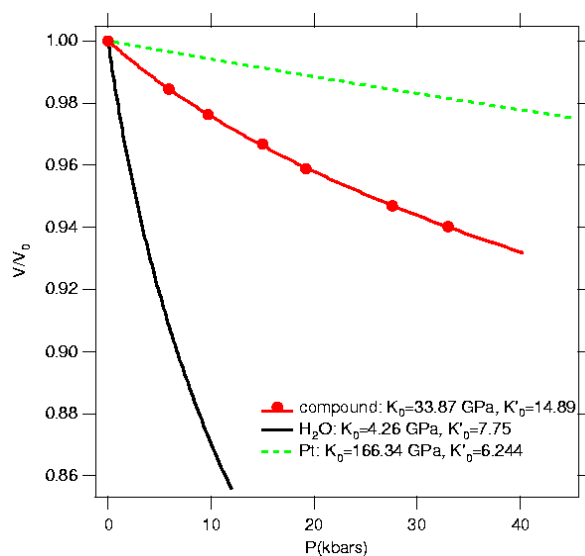


Figure 1: Comparison between the equation of state of HF, H_2O and Pt

Before performing new experiments at the ESRF, several tests should be done with different gasket materials in order to select a material chemically inert with HF for the sample chamber.

References

1. B. Desbat et al, J. Chem. Phys 78, 6377 (1983).
2. M.W. Johnson et al, Acta Crystallogr. B31, 1998 (1975).
3. D.A. Pinnick et al, Phys. Rev. B39, 8677 (1989).
4. R. Angelini et al, Phys. Rev. Lett. 88, 255503 (2002)

