EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

ESRF

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office using the **Electronic Report Submission Application:**

http://193.49.43.2:8080/smis/servlet/UserUtils?start

Reports supporting requests for additional beam time

Reports can now be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** High pressure EXAFS studies on Fe and Ge bearing amorphous and quasicrystalline materials	**Experiment number:** HD-258
Beamline: ID24	**Date of experiment:** from: 18 June 2008 to: 24 June 2008	**Date of report:**
Shifts: 18	**Local contact(s):** Dr. Sakura Pascarelli	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Dr. Ho-Kwang David MAO, Geophysical Laboratory, Carnegie Institution of Washington*
Prof. Wendy MAO, Stanford University*
Dr. Sakura PASCARELLI, ESRF*
Dr Guoyin SHEN, HPCAT, Carnegie Institution of Washington*
Dr. Wenge YANG, HPCAT, Carnegie Institution of Washington*

Report:

EXAFS is a powerful technique for measuring element-specific bonding changes, We conducted an extremely productive pilot study using high pressure energy-dispersive EXAFS techniques to study two archetypal 4th row elements: Ge and Fe. We looked at a number of model materials: Ge, GeO_2 glass, and $FeGeO_3$. Crystalline Ge undergroes a number of pressure induced transitions including structural and electronic changes. We studied this material up to 60 GPa (Figure 1). Variation of the energy of the edge position of Ge as a function of pressure confirms the transition from an insulator to metal at around 10 GPa (see Figure 2). The known crystal structures for Ge from XRD provide an additional validation of the EXAFS analysis.

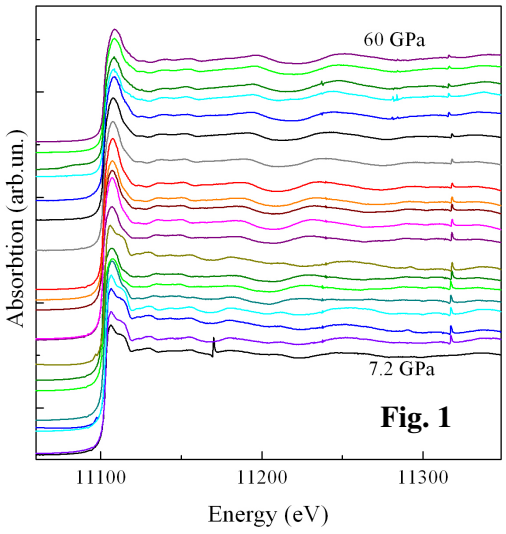

Fig. 1

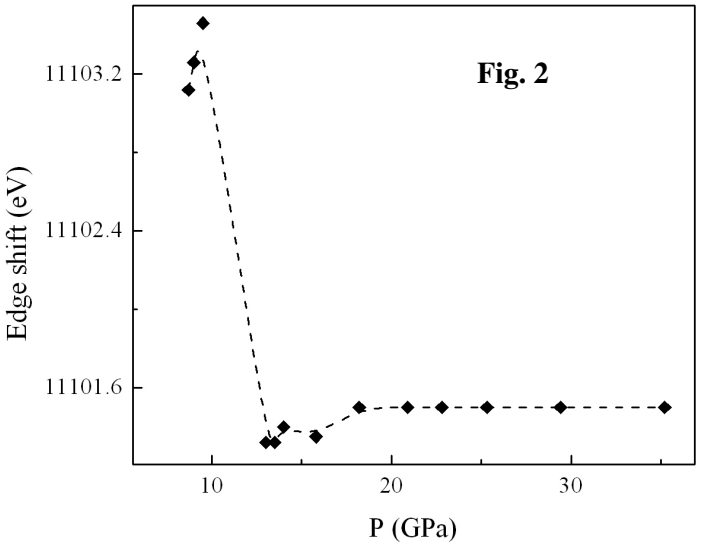

Fig. 2

A preliminary quantitative analysis has been performed up to 60 GPa, The backscattering amplitude and the photo-electron mean free path were calculated by FEFF6 and the three first Ge-Ge bond lengths were used to

fit experimental data in R space (range 1.5-3.3 Å) with the Artemis software. Considering a constant evolution of the Ge-Ge bond lengths with pressure we define each Ge-Ge distance as $R_{Ge-Ge} = R_{Ge-Ge}(calc) + a$ R_{Ge-Ge} (calc) and the EXAFS Debye-Waller factor as $\sigma^2_1 = \sigma^2_2 = \sigma^2_3$. The fit were performed using "*a*" and σ^2_1 as free parameters .The decrease in the Ge-Ge distances, described by the pressure behavior of the "a" parameter (see Figure 3), is in good agreement with the XRD results. The anomaly in the pressure behavior of *a* between 10-13 GPa is the signature of the structural transition from the diamond to the β-Sn phase which occurs in the system contemporaneously with the electronic transition. The pressure evolution of the σ^2_1 suggests the onset of a new structutal phase transition above 60 GPa (see Figure 4).

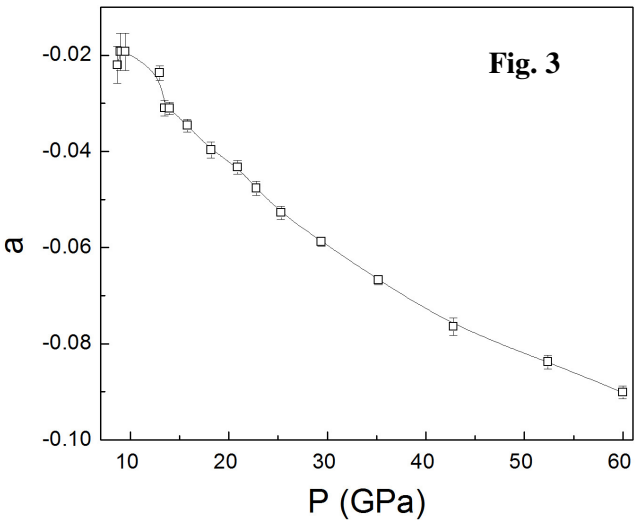

Fig. 3

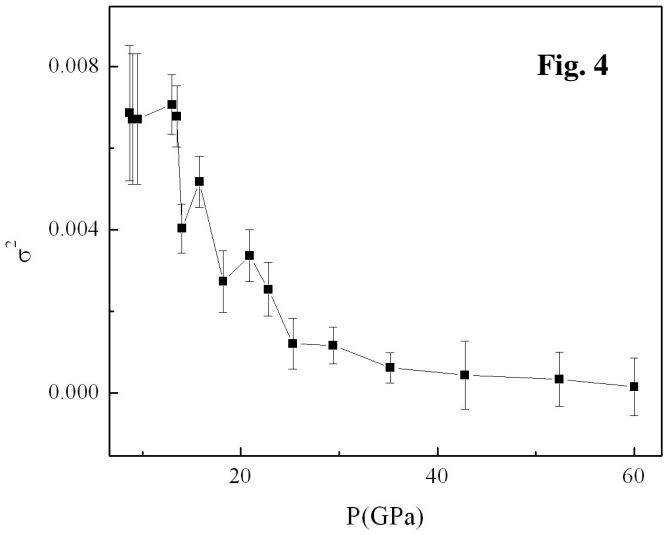

Fig. 4

The structure of amorphous GeO_2 has previously been studied by EXAFS to 29 GPa and XRD to 15 GPa. We extended the study of GeO_2 to 44 GPa (see Figure 5) over several increasing and decreasing pressure cycles. A preliminary analysis of data was performed up to 44 GPa. The Ge-O bond lengths were calculated by FEFF6 using the quartz and the rutile structure previously used to describe the structural phase transition in crystalline GeO_2. The GeO_2 data were fit over the range from 0.6 to 1.9 A with the mean value of the R_{Ge-O} and σ^2 as free parameters. The pressure evolution of the R_{Ge-O} distance was found to increase up to 30 GPa (see Figure 6) suggesting that the transition from a quartz-like to a rutile-like structure is complete at 30 GPa in amorphous GeO_2, whereas it occurs above 8 GPa in the crystalline compound.

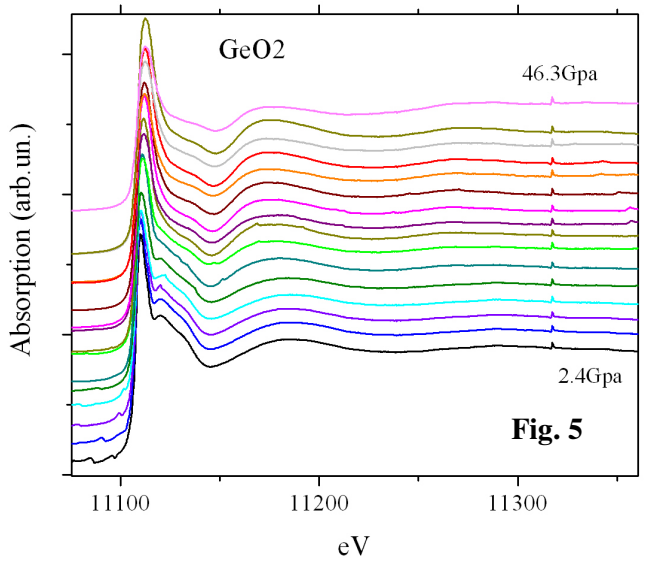

Fig. 5

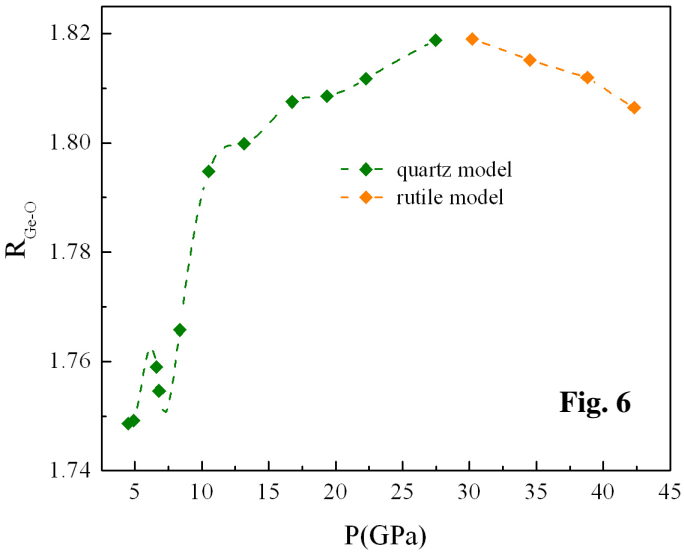

Fig. 6