	Experiment title: Crystallization kinetics of chalcogenide alloys for high density optical data storage.	**Experiment number**: HS1942
Beamline: ID24	**Date of experiment**: from: 13/02/03 to: 18/02/03 from: 16/12/00 to: 18/12/00	**Date of report**: 28/02/03
Shifts: 15	**Local contact(s)**: S. Pascarelli	*Received at ESRF:*

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Report:

We have performed time-resolved studies of crystallization kinetics at high temperature and pressure on chalcogenide alloys of interest for high density optical data storage. Understanding the structural transformation from the amorphous to the crystalline state is essential because it represents the time limiting step of rewritable optical data storage processes. Since crystallisation is accompanied by a large density decrease, the possibility to study this kinetics at different pressures helps disentangle the contribution of nucleation and growth to the crystallisation process.

The phase change materials used in the rewritable DVD's are based on ternary $Ge_xSb_yTe_{1-x-y}$. During our HS1942 shifts we performed several test runs on $Ge_4Sb_1Te_5$. The aim of the experiment was twofold:

- a feasibility experiment
- the study of the kinetics of the transition between the amorphous and the crystalline states.

This experiment tested a newly developed Diamond Anvil Cell (DAC) equipped with a double stage furnace that allows to reach 730 C (1000K). An external heating sleeve is used to heat the body of the DAC up to ~ 400 C. An additional Cu wire (0.2 mm diameter) coil fixed in a ceramic support at the base of the diamonds allows to heat the diamonds locally up to above 700 C. The temperature was measured using a Chromel Alumel thermocouple positioned at the base of the ceramic support. The temperature on the sample is estimated to be within 10 % of the measured value.

After different attempts, and despite difficulties encountered during the setup and the tests of the new furnace, we were successful in recording the kinetics of crystallisation of $Ge_4Sb_1Te_5$ in a high pressure time resolved experiment. The kinetics of crystallization has been studied at room pressure at a fixed temperature and under pressure (10 GPa) at a slowly increasing temperature. Typical acquisition rates were ~ 1spectrum/second. We clearly see a regular evolution of the local structure, as shown in the Figure for the 10 GPa run.

We performed several runs at ambient pressure and at high pressure (up to 10 GPa). The maximum temperature reached by the furnace was 835 C. A summary of the performed experimental runs on sample Ge_4SbTe_5 is presented below:

1 - Ambient pressure run. T from 20 C to 810 C and back to 550 C.
2 - Ambient pressure run. T from 20 C to 658 C and back 78 C. Some time resolved scans recorded with variable T.
3 - 10 GPa run with T from 20 C to 835 C. Some time resolved scans recorded at variable T.
The Figure below shows Ge K-edge XAS on Ge_4SbTe_5 at 10 GPa and temperature from 150 C to 225 C. 300 spectra were recorded in this temperature range at a rate of ~ 1 spectrum per second.
4 - Ambient pressure run. T from 20 C to 180 C. Problem with thermocouple.
5 - Ambient pressure run. T from 20 C to 185 C. Some time resolved scans recorded at constant T.
6 - High pressure run from ambient to 10 GPa.
7 - 10 GPa run with T from 20 C to 456 C. Some time resolved scans recorded at variable T and constant T.