ESRF	**Experiment title:** AgGe alloy phase diagram at high pressure	**Experiment number:** HS732
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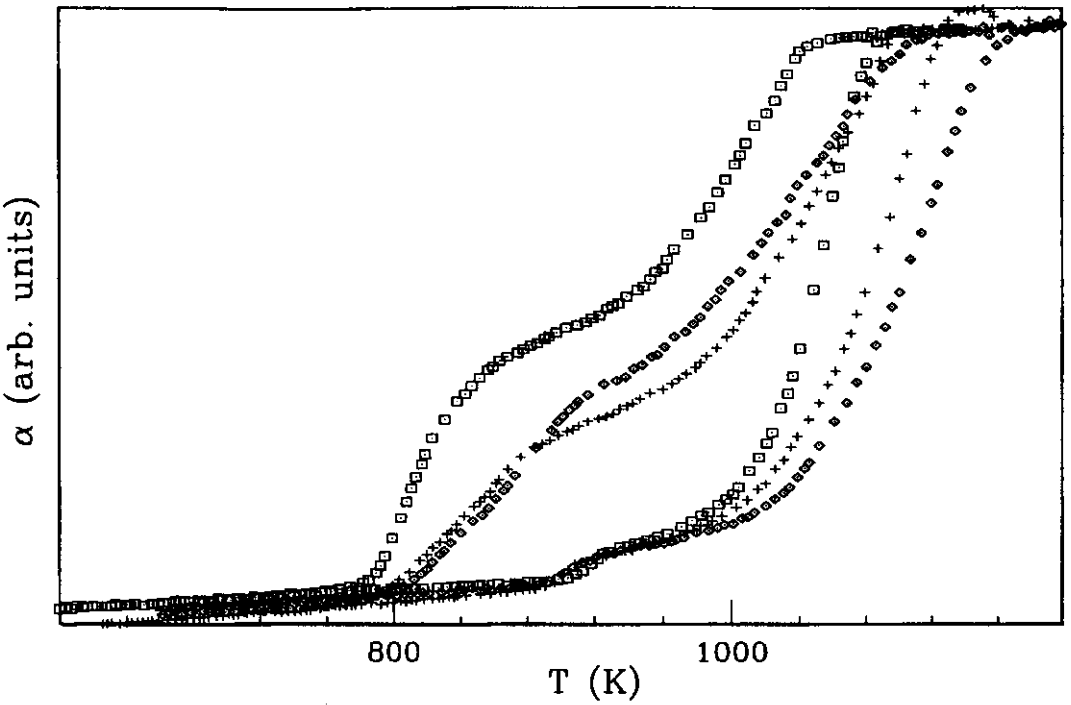
Report:

This experiment has exploited a complex setup involving the Paris-Edinburgh press for large-volume high-pressure high-temperature experiments [1] at BM29. For the first time an automatic temperature control was available through the acquisition system and it was possible to perform x-ray absorption temperature scans [2] at high pressure. The setup was also combining conventional x-ray absorption spectroscopy (XAS) and energy-scanning x-ray diffraction for sample characterization and pressure calibration, as detected through a 0.6 m long two-slit collimator at the fixed angle of $2\theta = 13.4°$.

$Ag_{1-x}Ge_x$ alloy samples were prepared by mixing macroscopic amounts of high purity substances in a graphite crucible under high vacuum conditions. The homogeneous melts were rapidly cooled to RT. The alloy lumps were kept in clean environment until the actual high pressure samples were prepared. The lumps were filed and the finest powder was separated by suspension and filtration procedure. The dried powder was finally mixed with BN (1:5) and pressed into 1.5 mm cylinders. These cylinders were directly inserted into the graphite (heaters) cylinders and eventually in the 7 mm sintered Boron gaskets of the large-volume HP setup.

A total of 4 samples were prepared during the experiment of which only 2 resulted successfully mounted as far as heather and thermocouple operation were concerned.



Each sample was subject to a complex experimental protocol involving cycles of pressurization, XRD scans, XAS measurements at the Ge and Ag K-edges, temperature scans, XRD scans. The Ge(220) Bragg peak was used for P calibration.

The full success of the experiment is illustrated in the above figure showing an example of Ge K-edge x-ray absorption temperature scans for sample (1), (composition x=0.84), at increasing pressures P (0.4 GPa ◇, 1.1 GPa +, and 1.7 GPa □). The curves show the progressive melting of the Ge component [2] in the alloy phase diagram. The major visible effect is the progressive bowing with pressure of the rising part of the curve just above the small step associated with the eutectic temperature (approximately constant in this P range). This phenomenon is connected with the known decrease of the Ge T_m with P, in this case, however, the present data will allow us to determine the variation of the whole liquidus curve of the alloy with P, using a single sample. The possibility to produce undercooled liquid samples under high pressure is also clearly demonstrated by the hysteresis in the curves.

Sample (2) with x=0.47 was pressurized up to 6 GPa while collecting similar temperature scans. The formation of the hexagonal inter-metallic phase was observed above ≈3 GPa.

All these data demonstrate the potential of the presently developed technique to provide unique information on the phase diagram of binary alloys at high pressure. More detailed publications are in preparation.

[1] Y. Katayama, M. Mezouar, J.P. Itié, J.M. Besson, G. Syfosse, P. Le Fevre, and A. Di Cicco, J. Physique IV **7**, C2/1011 (1997).

[2] A. Filipponi, M. Borowski, P. W. Loeffen, S. De Panfilis, A. Di Cicco, F. Sperandini, and M. Giorgetti, "Single energy x-ray absorption detection: a combined electronic and structural local probe for phase transitions in condensed matter.", J. Phys.: Condens. Matter **10**, 235 (1998).