| ESRF | Local structure in liquid matter under extreme conditions of high-pressure and/or high-temperature | **Experiment number:** HS1197 |
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| **Beamline:** BM29 | **Date of experiment:** from: Jan 2000 to: Jun 2002 | **Date of report:** 30 Aug 2002 |
| **Shifts:** 120 ( 40 16b) | **Local contact(s):** M. Borowski, G. Subias-Peruga, S. Ramos | *Received at ESRF:* |

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Report:

According to the original plan, the long term research project has involved both instrumentation development and advanced research efforts. The project has been supported by a grant ( 45 keuro) from the Istituto Nazionale per la Fisica della Materia (INFM) for the construction of a multi-channel detector collimator to reveal powder diffraction patterns at energy scanning x-ray absorption beamlines (Project PURS008). In spite of late funds availability and minor technical problems the instrument has been constructed, tested and exploited within the hs1197 project. The instrument is composed of rear CZT detector/slit units and motorized front slit units that can be mounted with a large freedom on the experimental bench using suitable Al structural frames. Each channel probes a fixed $2\theta$ scattering angle and the diffraction pattern is collected by performing monochromator energy scans. This detector can be configured in vertical or horizontal configurations around experimental chambers such as the L'Aquila Camerino oven or the Paris-Edinburgh (PE) press for high-pressure and high-temperature (HPHT) experiments. The detector is able to reveal the high-resolution powder diffraction pattern from the crystalline solid components of the sample. This information is essential in HPHT experiments for the pressure determination in the cell and, in general, to assess the sample nature. In addition to the hs1197 LTP the instrument has been used also during other ESRF projects in the framework of a preliminary usage agreement. Details on the instrument can be found at the URL: www.aquila.infn.it/PURS008/.

An additional experimental achievement has involved the manufacturing and optimization of the sample mounting components for the 10 *mm* gasket technique associated with the usage of the PE press. This is very important for the BM29 setup due to the larger sample size exploitable for HPHT studies above 15 keV.

Several experiments have been performed using the BM29 setup a part of which has been fully analyzed and published. The scientific efforts have been focused on the physics of fluid Ge. High purity Ge samples have been studied under high vacuum and under high-pressure conditions up to about 10 GPa both in the liquid and in the solid cubic and $\beta$-Sn phase. The experiments involved not only XAFS measurements but also x-ray diffraction for the pressure and phase assessment, collected with the instrument previously described, and x-ray absorption temperature scans.

With this technique it has been possible to demonstrate the possibility to undercool the sample also at high pressure and confirm the occurrence of major changes in the fluid Ge nature in the investigated range. All this matter will be subject of detailed publications. Additional scientific topics tackled within the present project include:

- Measurements of the thermal expansion effect in the $Ag_{1-\theta}Ge_{\theta}$ terminal solid solution [6].
- Accurate simultaneous measurement of the lattice parameter of Au and NaCl in a wide pressure temperature range, as a test for the ESXD technique for HPHT studies.
- Detailed investigation of the AgGe phase diagram by XAFS and XRD to understand the mechanism for the stability onset of the intermetallic hexagonal phases.
- A combined investigation of pure Au by XAFS and XRD to correlate the changes in the first neighbor distribution and in the lattice spacing as a function of the thermodynamic state.
- An attempt to investigate liquid $GeO_2$ another candidate for polyamorphism in the liquid phase.

The ESRF HS1197 beamtime has been also at the basis of several Laurea degree theses (in Italian) written by students from the Universities of L' Aquila and Camerino, in particular:

**a)** A. Trapananti, Camerino "Studio della struttura e delle transizioni di fase nel CuI tramite assorbimento e diffrazione di raggi X"

**b)** E. Principi, Camerino "Studio del disordine strutturale nella lega ternaria RbBr(1-x)I(x) in fase solida e liquida"

**c)** V. Giordano, L' Aquila: "Misure dell' equazione di stato di solidi cubici in condizioni estreme di pressione e temperatura mediante diffrazione di raggi x a scansione di energia."

**d)** M. Malvestuto, L' Aquila: "Studio del fenomeno della recalescenza nel Ge liquido sottoraffreddato."

Publications


1. A. Filipponi, *"EXAFS for liquids."*, J. Phys.: Condensed Matter **13**, R23-R60 (2001).
2. A. Filipponi, A. Di Cicco, S. De Panfilis, A. Trapananti, J. P. Itiè, M. Borowski, and S. Ansell, *"Investigation of undercooled liquid metals using XAFS, temperature scans and diffraction."*, J. Synchr. Rad. **8**, 81-86 (2001).
3. A. Filipponi, *"Structural studies of condensed matter under extreme conditions of high pressure and temperature combining x-ray absorption spectroscopy, powder diffraction and temperature scans."*, Notiziario Neutroni e Luce di Sincrotrone **6**, 23-29 (2001).
4. S. De Panfilis, A. Di Cicco, A. Filipponi, and M. Minicucci, *"Solid and liquid AgI at high pressure and high temperature: a x-ray absoprtion spectroscopy study."*, High Pressure Research **22**, 349-353 (2002).
5. A. Di Cicco, E. Principi, and A. Filipponi, *"Short-range disorder in pseudobinary ionic alloys."*, Phys. Rev. B **65**, 212106 (2002).
6. A. Filipponi, V. M. Giordano, and M. Malvestuto, *"Lattice expansion and Ge solubility in the $Ag_{1-\vartheta}Ge_{\vartheta}$ terminal solid solution."*, physica status solidi (b) **234**, 496-505 (2002).
7. A. Filipponi, V. M. Giordano, S. De Panfilis, A. Di Cicco, E. Principi, A. Trapananti, M. Borowski, and J. P. Itie, *"A multi-channel detector-collimator for powder diffraction measurements at energy scanning x-ray absorption spectroscopy synchrotron radiation beamlines for high-pressure and high-temperature applications."*, Rev. Sci. Instr. (to be published 2003).