Experiment title: EXAFS at extremely high temperatures at BM29	**Experiment number:** MI124

Beamline: BM29-BL18	**Date of Experiment:** from: 4-Dec-1996 to: 9-Dec-1996	**Date of Report:** 22-Feb-1996
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Report:

We have completed the installation of an automated high-temperature sample environment system for x-ray absorption measurements.

The high temperature vessel is based on the evolution of a previously developed furnace [l]. The samples are confined in a x-ray transparent crucible and the temperature can be measured by a variety of probes including Chromel-Alumel, Pt-PtRh10%, WRe5%-WRe26% thermocouples, and a pyrometer for the range 1000-3000 °C with a small spot sensitivity. The probe readouts are converted into 420 mA current signals and fed to ADC channels of the acquisition system. Two temperature readings can be simultaneously recorded on the data files, they usually correspond to a suitable thermocouple which probes the internal temperature just above the beam path, and the pyrometer which probes the surface temperature of the crucible. Coincidence between the two readings and calibration with known phase transitions assures a correct temperature readout.

The crucible is heated by a **0-100** A DC power supply which is remotely controlled by a O-5 V voltage source. This is generated by a 12 bit DAC channel corresponding to a pseudomotor in the SPEC application. The acquisition system allows to control the sample temperature with an excellent time stability (fluctuations below 10 K for continuous operation at 1500 K). This is essential for the acquisition of low noise x-ray absorption spectra at high temperature. At the same time it is possible to perform regular temperature cycles in a wide range of temperature intervals and heating and cooling rates from 0.01 to 50 ***K/s.*** This is essential for single energy x-ray absorption detection experiments where the occurrence of phase transition can be monitored at specific points with particularly high sensitivity.

Several prototype experiments have been performed during the allocated beamtime, including studies of **Rh** and Ir at high temperature. **Rh** has been investigated as foil and powder samples in a wide temperature range up to 2600 K in the liquid phase. The possibility to measure liquid Rh is demonstrated by the single energy temperature scan reported in Fig. 1. The energy has been chosen on a point in the EXAFS region of high sensitivity to the melting transition. The sharp decrease of absorption indicates the occurrence of melting at T_m = 1966 **°C** and the presence of the hysteresis loop is a clear signature of the possibility to undercool the liquid state.

The excellent performances of the beamline allow to collect spectra with noise equal or better than 10^{-4} even for powder samples at high temperature. As an example in Fig. 2 we report three Rh EXAFS spectra collected in the range 1200-1900 K. The thermal damping of the structural oscillations is quite evident.

[I] A. Filipponi and A. Di Cicco, Nucl. Inst. & Methods for Phys. R.es. B 93, 302 (1994).
[2] A. Filipponi, J. Phys.: Condensed Matter 8, 9335 (1996).

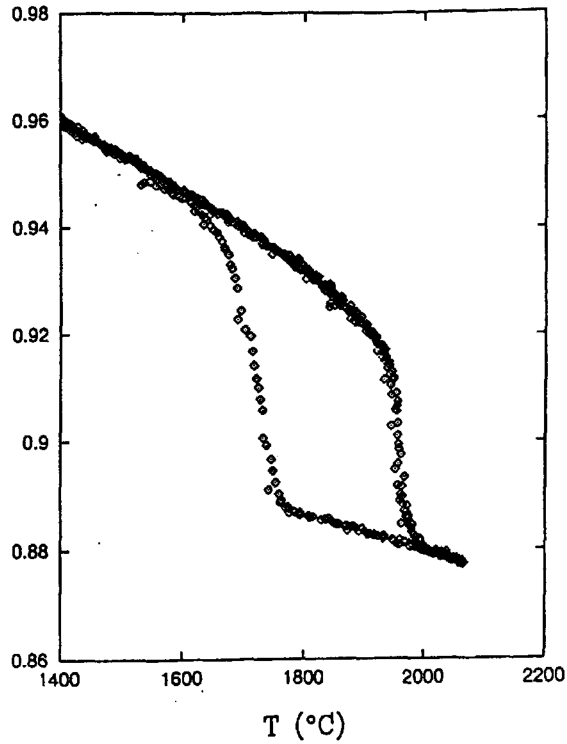

Fig. 1

Fig. 2