ESRF	**Experiment title:** Undercooling phenomena and Short Range Order in Liquid Metals	**Experiment number:** HE-259
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Names and affiliations of applicants (*indicates experimentalists):

*†S. De Panfilis, *†A. Filipponi and *‡C. Meneghini

† E.S.R.F. - B.P. 220, F-38043 Grenoble, France
$ I.N.F.N. - Via Enrico Fermi 40, I-00044 Frascati (Rome), Italy

Report:

During beam time allocated for experiment HE-259 we have performed a successful experiment to investigate the interaction between liquid tellurium (ℓ-Te) droplets and several different confining matrices.

To obtain out results, we used the newly developed XAS technique [1], based on single energy detection during temperature scans and revealed, for the first time, a strong interaction occurring upon freezing of the ℓ-Te droplets when they were confined in graphite.

Combining the results of this measurements together with a powder X-ray diffraction (XRD) experiment performed at ESRF-BM16 (prop. n. SC-360), we show that this behaviour is due to the entangled broken chain structure of the liquid phase. This is consistent with our previous X-ray absorption spectroscopy experiment [1], indeed, ℓ-Te is largely made of two-fold coordinated atoms, partially retaining its peculiar chaine-like crystalline structure upon melting.

To prepare suitable sample for XAS measurements, particular care was devoted to achieve the optimal thickness ($\sim 20\ \mu m$) and requested homogeneity; samples were prepared as follows: fine precursor high-purity 99.995% compound (TeO_2) powder was ball-milled together with inert matrix powder (graphite, BN, Al_2O_3, NaCl) in 1:20 dilution, and then pressed into pellets. *In situ* reduction to crystalline Te was performed by means of thermal treatment under controlled atmosphere during several cycles and was monitored looking at the near edge structures.

The powder XRD patterns were taken using the same samples that were used in this experiment, verifying *a posteriori* that complete reduction was achieved during the sample thermal treatment, and demonstrating the absence of any crystalline phases other than c-Te and the particular matrix phase – excluding any chemical interaction during the thermal treatment.

Complete K-edge spectra were recorded in the solid (from RT to 400°C) and liquid phase (at 470 °C) for each matrix, to tune the photon energy to a spectral feature that is highly sensitive to the phase transition (Fig. 1). XAS measurements were performed in transmission mode with Kr filled photodiode

detectors in the energy range 31.5 - 32.8 keV and with constant k spacing of 0.02 Å^{-1}.
High temperature measurements were performed in an X-ray transprent graphite resistive crucible in high vacuum oven, particularly suitable for these ranges of temperatures and energies. Several melting and freezing cycles were performed on each sample.
Single energy temperature scans in the range RT-800 K were performed and are shown in Fig 2 for the graphite and NaCl cases. The absorption coefficient shows an hysteresis cycle with discontinuity at the known Te melting point on the heating stroke and reveals an undercooled state during the cooling period. The occurrence of a sharp melting transition is also evidence of a high purity bulk sample.
On the cooling stroke, we reveal a dramatically different freezing behaviour of ℓ-Te droplets confined in the two matrices, showing complete different crystallization processes.
Main results of this work will be more extensively presented in a forthcoming paper.

References:

[l] S. De Panfilis and A. Filipponi, Europhys. Lett. 37, 397 (1997).
[2] A. Filipponi, M. Borowski, P. W. Loeffen, S. De Panfilis, A. Di Cicco, F. Sperandini, M. Minicucci and M. Giorgetti, J. of Physics: Cond. Matt. 10, 235 (1998).

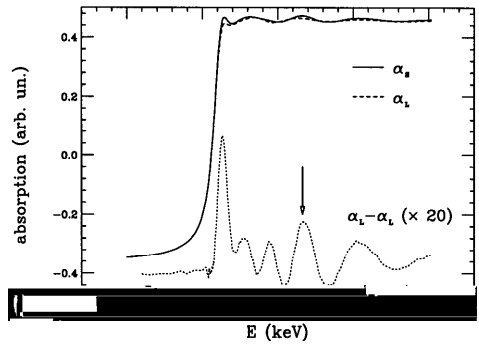

Figure 1: K-edge of solid (α_S, solid curve) and liquid (α_L, dashed curve) Te in graphite at 400°C and 470°C respectively. The choosen energy point for temperature scan is indicated by the vertical arrow on the difference curve.

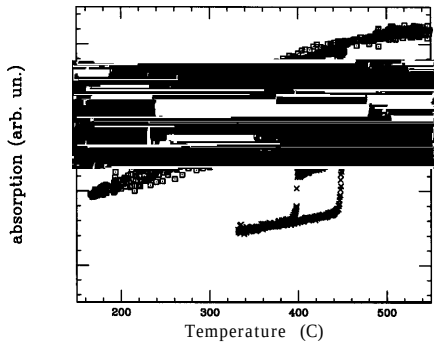

Figure 2: Single energy X-ray absorption scan vs. temperature for Te particles confined in NaCl (upper curve) and graphite (lower curve).