ESRF	**Experiment title:** Investigation of Icosahedral Short Range Order (ISRO)-mediated nucleation in deep undercooled aluminium alloys	**Experiment number**: A30-2-1164
Beamline: BM 30	**Date of experiment**: from: 22/02/2023 to: 28/02/2023	**Date of report**: 14/03/2023
Shifts: 18	**Local contact(s)**: Jean-Louis Hazemann, Isabelle Kieffer	*Received at ESRF:*

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Objective of the project:

The experimentations carried out in this project aim to understand the mechanism of Icosahedral Short Range Order (ISRO)-mediated nucleation in aluminium alloys. This theory proposes the existence in the liquid metallic alloy of locally ordered zones in an icosahedral shape (hence the name). These icosahedra then promote nucleation of alpha-aluminium phase. As the locally ordered zones are only a few atoms in size, the EXAFS/XANES technique was chosen to characterize them.

Transition metals as alloying elements are thought to promote ISRO in liquid aluminium; this project focuses on the role of iron (Fe), alone or in combination with either silicon (Si) or zirconium (Zr). The aluminium icosahedral atomic clusters are expected to form around iron atoms, therefore the Fe K-edge was chosen for the EXAFS analysis (7.112 keV). Three main aluminium alloy compositions were tested: Al+1%wt Fe, Al+1%wt Fe+1wt Si, Al+1%wt Fe+0.2%wt Zr.

The analysis were performed on small metallic spheres (few mm wide), in fluorescence. Several sample temperatures were tested, from 850°C to below the alloy liquidus temperature, as undercooling promotes the ISRO. The heating of the samples was carried out with a portable induction furnace, in a controlled reducing atmosphere, to avoid oxidation. This apparatus levitates the sample while heating it, thus avoiding any solid contact likely to induce undesired nucleation when the sample is undercooled. For reference, some measurements have been made on solid samples at room temperature.

Preliminary results:

Preliminary results show no visible differences in the XANES signal for the different alloys in the liquid state at high temperature (figures 1 and 2). Some differences seem to exist for the solid materials at room temperature. The XANES signal could bring some valuable information, but some higher resolution measurements in this energy range would be required.

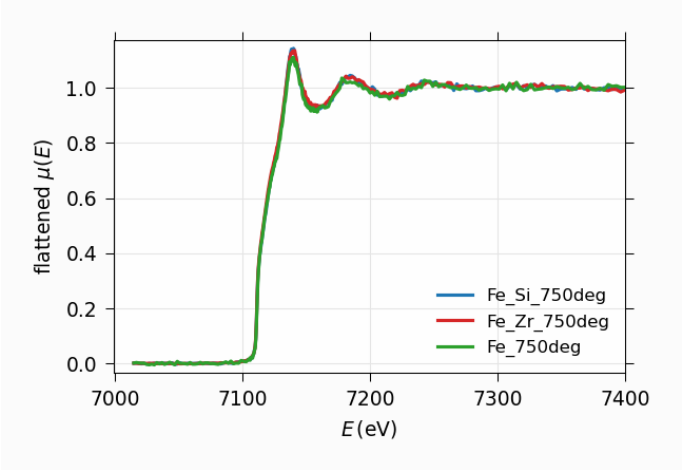

Figure 1: Normalised EXAFS signals for liquid samples at 750°C

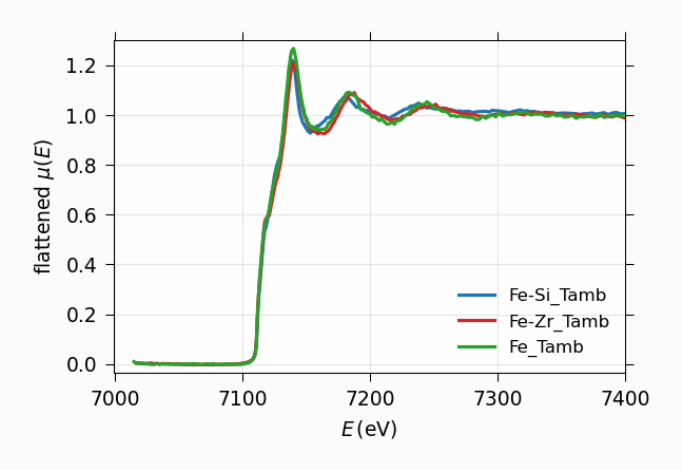

Figure 2: Normalised EXAFS signals for solid samples at room temperature

The role of the alloying elements in creating a potential local order in the liquid metal remains to be thoroughly analysed. The first results tend to show an effect of the addition of Si in the Al-1%wt Fe alloy (Figures 3 and 4). The order in Si-containing alloy in the liquid seems to be closer from the order of the solid, compared to Al-1%wt Fe without Si.

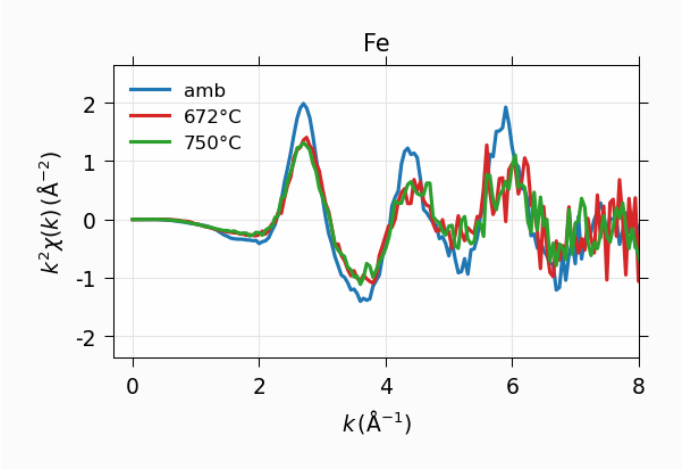

Figure 3: Reduced EXAFS signals for Al-1%wt Fe samples at several temperatures.

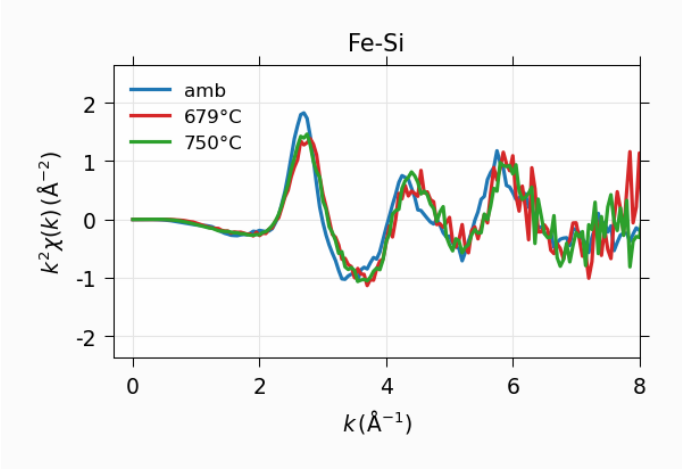

Figure 4: Reduced EXAFS signals for Al-1%wt Fe-1%wt Si samples at several temperatures.

Measurements issues:

The experimental setup has caused some problems with the EXAFS detector (Canberra 13 elements). The high-frequency electromagnetic field (132 kHz) generated by the induction coil disturbs the electronics of the photon detector, and consequently, causes some noise in the measured signals. Despite these disturbances, usable data were obtained by taking some precautions during the experimental setup installation and the measurements. Nevertheless, a detector setup immune to the radiofrequency-induced perturbations, such as a crystal analyser, would vastly improve our measurements.