Experiment HC-2595, ID26

Title: Role of interface in bcc-fcc phase transition of Fe nanostructures: an in-situ XAS-XES experiment.

Participants: Chiara Maurizio, Angela Trapananti, Alessandro Patelli

Experimental conditions: HR-XAS, heating in vacuum the sample, nominally up to 900 C

Samples: Fe films and nanoclusters embedded in silica and Au. Films with different thickness and clusters of various size were prepared

Report

The aim of the experiment was to investigate the bcc-to-fcc transition of selected Fe nanostructures. The transition occurs in the bulk at 912C. To successfully perform the experiment we needed a furnace that allowed to heat the sample in vacuum up to 900C minimum.

The idea was to test first a thick (≈100 nm) film, then move to thinner Fe films and then to nano clusters, always embedded in either amorphous silica or in gold. The two substrates are expected to influence the phase transition.

The furnace available at the beamline worked at a maximum temperature of about 900C-950C on the heating filament, but the temperature achieved on the sample was always well below 900 C (likely around 800C). For this reason, the experiment failed for the most part.

The problems we had to face with the furnace were:

- The maximum temperature was too low for our purposes (about 100C lower than requested in the proposal)

- At the maximum heating condition, the furnace walls were too hot, causing the explosion of the kapton windows.

- The heating filament broke twice.

So, we spent most part of the time trying to fabricate a suitable metallic shield to limit as most as possible the loss of heat by irradiation. Different kind of combinations [shield]/[sample holder]/[chamber cooling system] were tested, but we only could arrange some temporary solution during the last night, when we could finally measure the phase transition on two films (100 nm thick), one embedded in silica and the other one in gold. The fcc and bcc crystalline phases have different Fe K-edge features (see Figure 1). We consider as a preliminary order parameter the value of (a+c)/2-b, where a,b,c are the values of the x-ray absorption coefficient located as in Figure 1. In this way the fcc (bcc) phase has a positive (negative) value of the order parameter. The variation of this parameter as a function of the temperature measured close to the sample is shown in Figure 2 for the case of a 100 nm thick Fe films embedded in silica.

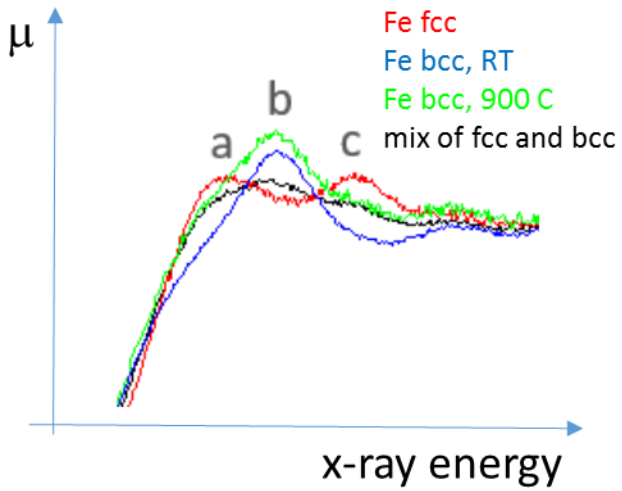

Figure 1. X-ray absorption spectrum (close to the K-edge energy) from a 100 nm-thick Fe film.

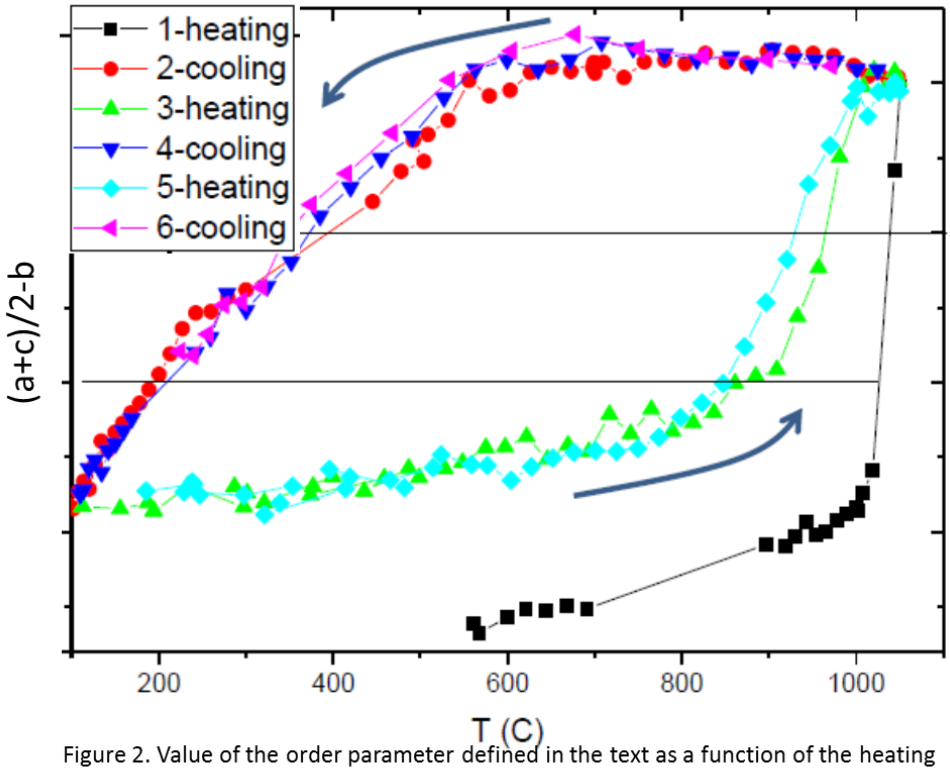

Figure 2. Value of the order parameter defined in the text as a function of the heating temperature, during subsequent heating/cooling cycles in vacuum.

In this sample, the transition is expected to mimic the bulk one, as it actually occurs. This should have been our starting point for the experiment, while it could unfortunately be our last measurement.

For a future experiment, the furnace should be first tested in real conditions, i.e., testing the temperature of the sample and not (only) of the heating filament. From our side, we are in contact with the beamline staff and we sent some suitable samples for a test. We are willing to perform again the experiment in case a full study is preliminary carried out by ESRF staff to provide a furnace within the specifications required.