EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

Experiment Report Form

	Experiment title: Time-resolved local atomic fluctuations in Fe-based bulk metallic glasses with Invar effect	**Experiment number**: HC 4434
Beamline: BM23	**Date of experiment**: from: 05/05/2021 to: 10/05/2021	**Date of report**: 09/08/2021
Shifts: 15	**Local contact(s)**: Olivier Mathon	*Received at ESRF:*

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(** indicates remote users. Due to Covid 19-related regulations, only one single user was allowed on campus)

Report:

It is known that ferromagnetic Fe-based bulk metallic glasses (BMGs) universally show an anomalously low coefficient of thermal expansion below their Curie temperature. This effect is known as the Invar effect and rarely seen in crystalline materials, with Fe-Ni around 36 at% Ni being a notable exception. The Invar effect in BMGs is inherently linked to the amorphous atomic arrangement because the Invar effect is not present anymore after the BMGs have crystallized. The volume transformations of the Invar effect are fully reversible. Whether the changes are accompanied by local structural/atomic rearrangement is completely unclear. The key point is to investigate whether local atomic rearrangement is taking place at the Curie temperature and if so, characterize it and relate it to the macroscopically observed Invar effect.

During this beamtime we performed extended X-ray absorption fine structure (EXAFS) experiments on two quaternary BMGs, $(Fe_{71.2}B_{24}Y_{4.8})_{96}Nb_4$ and $(Fe_{73.2}B_{22}Y_{4.8})_{95}Mo_5$, and crystalline $Fe_{64}Ni_{36}$ at the Fe, Ni, Y, Nb and Mo K-edges. The BMG samples were prepared as a fine powder mixed with boron-nitride and pressed into a pellet. The FeNi sample was measured as a thin foil. EXAFS scans were recorded continuously while subjecting the samples a temperature sequence of 20°C → 600°C → 20°C by using a homemade - ESRF-device in an Argon atmosphere. The samples were heated at around 1 K/min with an acquisition time of around 60 seconds per spectrum. For every EXAFS scan, a foil of the corresponding absorption edge was measured as reference simultaneous with the sample.

Figure 1 shows the EXAFS absorption signal of $Fe_{64}Ni_{36}$ at various temperatures, room temperature (20°C), Curie temperature (230°C) and far above the Curie temperature (595°C). Throughout the whole temperature range, the low frequency oscillations of the absorption signal, $\chi(k)$, do not move. However, several finer features get lost as the sample is heated and the Curie temperature is passed.
The pair-distibution-like distance distribution is presented in Figure 2. From about 200°C to 300°C the nearest neighbour distribution becomes much less pronounced relative to the background.

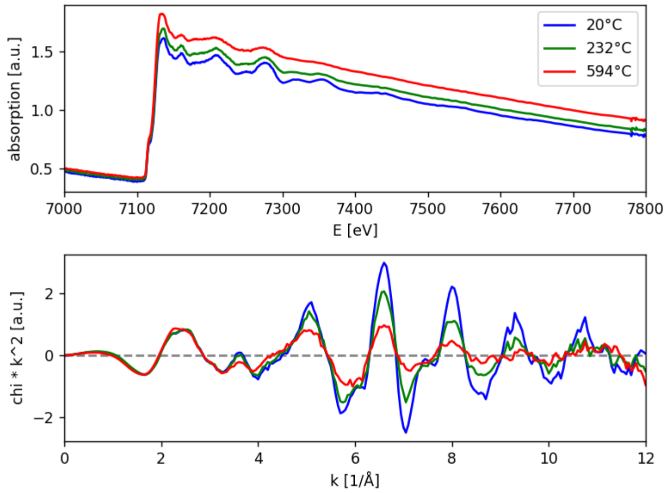

Figure 1: EXAFS scans on Fe-Ni foil at Fe K-edge (7112 eV).

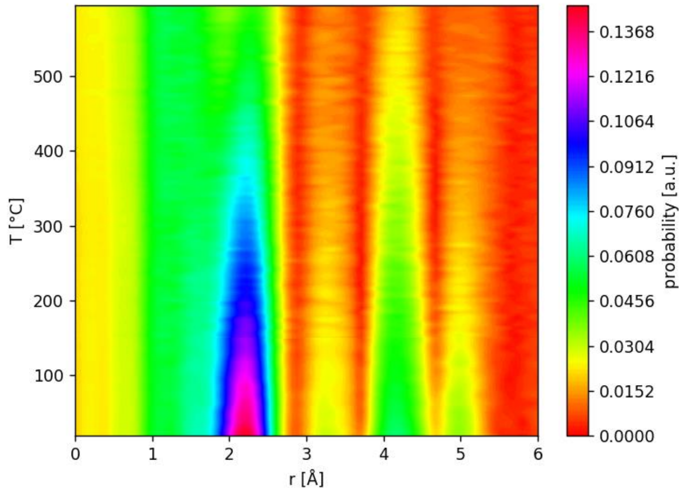

Figure 2: Heatmap of $\chi(r)$ of Fe-Ni foil at Fe K-edge (7112 eV).

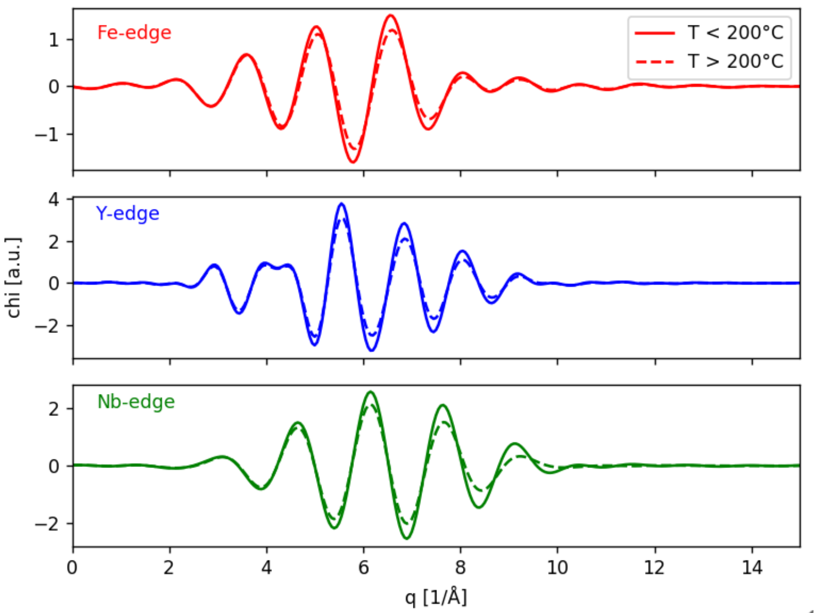

Figure 3: EXAFS signal $\chi(q)$ of FeBYNb metallic glass.

The BMG samples were each studied at 3 different absorption edges. The EXAF signal $\chi(q)$ of the FeBYNb alloy is shown in Figure 3. Solid lines show the signal below Curie temperature and dashed lines the signal above Curie temperature. Having measured each alloy at three absorption edges allows to build a model of the amorphous structure by Reverse Monte Carlo (RMC) modelling. This model can then serve as a basis for fitting the EXAFS equation to uncover the element-specific contribution to the Invar effect. The RMC modelling of the BMGs are under analysis and a scientific publication is currently in preparation.