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Report:

We have performed *in situ* measurements of nuclear forward scattering (NFS) during temperature treatment of NANOPERM-type metallic glasses. Main objective of the project is to contribute to the knowledge on the evolution of magnetic arrangement in the NANOPERM-type metallic alloys during their exposure to enhanced temperature. These alloys are used as precursors for the production of the so-called nanocrystalline alloys by controlling the annealing temperature and time. Disordered nature of structural arrangement in amorphous and nanocrystalline alloys gives rise to advantageous (from a practical application point of view) magnetic properties [1]. Especially nanocrystalline alloys attract a lot of scientific interest because, contrary to their amorphous counterparts, their magnetic parameters do not substantially deteriorate at elevated temperature during the process of their practical exploitation.

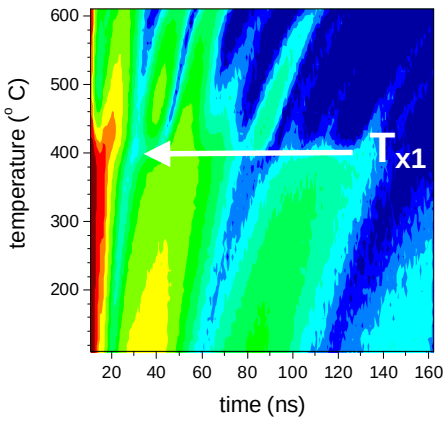

Fig. 1. Contour plot of NFS spectra with the indicated temperature of the onset of crystallization T_{x1} for the $^{57}Fe_{79}Mo_8Cu_1B_{12}$ alloy.

Samples of amorphous $^{57}Fe_{79}Mo_8Cu_1B_{12}$ were prepared by rapid quenching on a rotating wheel. They were enriched to isotope of ^{57}Fe by 50 % to allow for rapid acquisition of the NFS spectra. This alloy is paramagnetic at room temperature providing pure electric quadrupole interactions characteristic for the amorphous residual phase. During the crystallization, only pure bcc-Fe grains are formed with well defined magnetic dipole interactions. Heat treatment was accomplished by continuous increase of temperature with the ramp of 10 K/min up to 600 ^{o}C. The collected spectra are shown in Fig. 1 as a contour plot which shows how the character of hyperfine interactions changes after the onset of crystallization at T_{x1}. In addition to these structural changes, the effects of temperature increase on hyperfine magnetic fields are visible. Evolution of the character of hyperfine

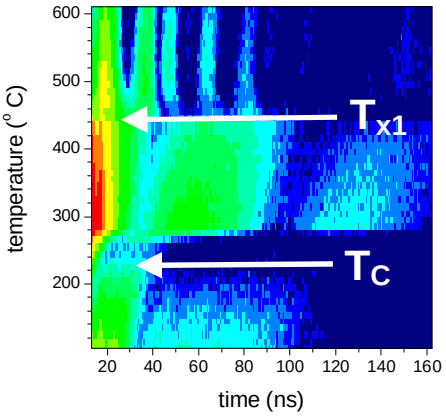

Fig. 2. Contour plot of NFS spectra with the indicated temperature of the onset of crystallization T_{x1} and Curie temperature T_C for the $(^{57}Fe_{0.75}Co_{0.25})_{79}Mo_8Cu_1B_{12}$ alloy.

interactions with temperature belongs to unique results of the current experiment. It is possible to obtain the temperature dependencies for all structural components present in the nanocrystalline alloy.

The fitting model used to evaluate the experimental spectra include component associated with the amorphous residual matrix, nanocrystalline grains as well as interface regions which belong to iron atoms located at the surfaces of nanograins [2]. Both amorphous residual phase and interface regions exhibit structural disorder which is accounted for by distributions of particular spectral parameters including quadrupole splitting, magnetic hyperfine fields, isomer shifts, etc. The use of such approach is time demanding and compete evaluation of huge amount of experimental data is still in progress.

Another type of NANOPERM-type alloy which we have measured is $(^{57}Fe_{0.75}Co_{0.25})_{79}Mo_8Cu_1B_{12}$. This composition features magnetic hyperfine interactions at room temperature. Consequently, during the temperature increase, change in magnetic order is observed, i.e. we can define the Curie point of the amorphous phase (Fig. 2).

Along with dynamical temperature experiments we have performed also isothermal time scans of $^{57}Fe_{90}Zr_7B_3$ alloy. Examples of NFS spectra taken at the temperature of 743 K are shown in Fig. 3.

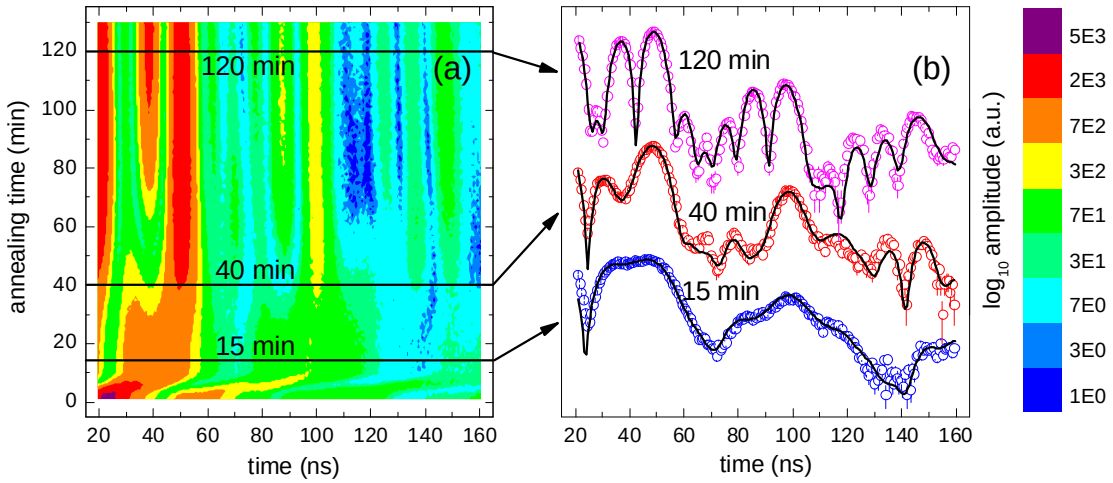

Fig. 3. Contour plot of NFS spectra of $Fe_{90}Zr_7B_3$ annealed at 743 K (a) and selected NFS spectra (open symbols) with fits (solid lines) for 15, 40, and 120 min of annealing (b).

In conclusion, applying nuclear forward scattering of synchrotron radiation *in-situ*, i.e. during the thermal treatment of NANOPERM-type alloys we were able to differentiate not only between Fe atoms in amorphous and crystalline phase but also Fe atoms located inside the nanocrystals and those positioned on their surfaces.

However for detailed understanding of the observed effects, which could be of significant technological importance, more investigations are necessary. For example isothermal measurements at selected temperatures in order to understand the kinetics of crystallization.

References:

[1] Hernando A., J. Phys.: Condens. Matter **11** (1999) 9455.
[2] M. Miglierini and J. M. Greneche, J. Phys.: Condens. Matter **9**, 2321 (1997).