ESRF	**Experiment title:** Russian grant: **XMCD and EXAFS studies of local, electron and magnetic structure of nanocrystal alloys based on the $Nd_2Fe_{14}B$ and $SmCo_5$compounds**	**Experiment number:** HC-2600
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Report:

Introduction:

$Nd_2Fe_{14}B$-based alloys are one of the most popular and commercially used materials for highest performance permanent magnets [1]. Ability to change the properties of these alloys by addition of different alloying elements, as well as the use of various types of heat treatment for controlled changes of microstructure can significantly expand the scope of their practical applications. For example, the Zr addition into $Nd_2Fe_{14}B$ leads to an increase of the coercivity and remnant field of the respective alloys [2]. Similar effect is also observed with grain size decreasing to the nano-scale range at melt spinning.

The present work is devoted to the comparative study of the effect of nano-structuring, as well as the effect of Zr addition to $Nd_2Fe_{14}B$ alloys on their local magnetic properties by means of X-ray Magnetic Circular Dichroism (XMCD) at the $L_{2,3}$ edges of Nd and at the K-edge of Fe.
Preliminary results of this work will be published soon in [3]

1. Experiment

X-ray Magnetic Circular Dichrtoism (XMCD) spectra at the $L_{2,3}$ edges of Nd and at the *K*-edge of Fe have been measured at the ESRF ID-12 beamline [4]. The source was the helical

undulator APPLE-II, which emits either right or left circularly polarized x-rays with a polarization rate in excess of 97%. The X-ray absorption spectra at the $L_{2,3}$-edges of Nd and at the *K*-edge of Fe were recorded using the total fluorescence yield detection mode in backscattering geometry. XMCD spectra were obtained as direct difference of absorption spectra recorded consecutively either by reversing the helicity of the incident beam or by flipping the magnetic field (± 10 Tesla) generated by a superconducting solenoid and applied along the X-ray beam direction. This procedure allowed us to ensure that the experimental XMCD spectra are free from ~~any~~ experimental artefacts. The samples were kept at room temperature. Since the penetration depth of the x-rays is smaller than the sample thickness, the recorded XAS and XMCD spectra had to be corrected for so-called self-absorption effects. The correction is applied by taking into account the chemical composition, thickness of the sample, angle of incidence of the X-ray beam, and the solid angle of the detector [5].

2. Results

XMCD spectra of Zr-doped nano-crystalline sample $Nd_{10,4}Zr_{4,0}Fe_{79,2}B_{6,4}$(Fig. 1, left panel) have a greater amplitude of a signal at the Nd-$L_{3,2}$ absorption edges compared to the undoped $Nd_2Fe_{14}B$ whereas the opposite effect is observed at the *K*-edge of Fe. The highly structured XMCD spectra at the L_3 absorption edge of Nd are due to a variety of electronic transitions involved. The first minimum (prepeak) is due to quadrupolar transitions $2p_{3/2} \rightarrow 4f_{5/2}$, and the second peak (main maximum) – dipolar transitions $2p_{3/2} \rightarrow 5d_{5/2}$ [6]. The oscillating structure at higher energies is a manifestation of multiple scattering of a photoelectron wave from the nearest neighbors, so-called magnetic EXAFS. XMCD signal at the L_2 absorption edge of Nd is characterized by the pronounced main maximum, corresponding to dipolar transitions $2p_{1/2} \rightarrow 5d_{3/2}$, but with much weaker quadrupolar contributions [6]. The shape of the Fe *K*-edge XMCD signal could also be disentangled into quadrupolar and dipolar transitions, but this assignment is usually quite ambiguous.

Thus, the experimental XMCD results could indicate that the introduction of Zr into $Nd_2Fe_{14}B$ phase undoubtedly influences the magnetic state of Nd and Fe sublattices, changing the overall electronic subsystem, in particular, the 5*d* states of Nd and to a lesser extent the 4*p* band of Fe.

According to the TEM data an average grain size was estimated as 5-10 micron in micro-crystalline and as 10-20 nm - in nano-crystalline samples. Comparing XMCD spectra of nano-crystalline and micro-crystalline $Nd_{10,4}Zr_{4,0}Fe_{79,2}B_{6,4}$ samples (Fig. 1, right panel), we can say that the amplitudes of XMCD signals at Nd-L_3 and *K*-Fe absorption edges in the case of nano-crystalline sample are greater, while at Nd-L_2 absorption edge – smaller, compared to micro-crystalline sample.

These results confirm the effect of crystal grain's size on the local magnetic properties. It should be noted that XMCD spectra change differently depending on the effect of nano-structuring or Zr doping. These results are discussed in the paper [3].

References

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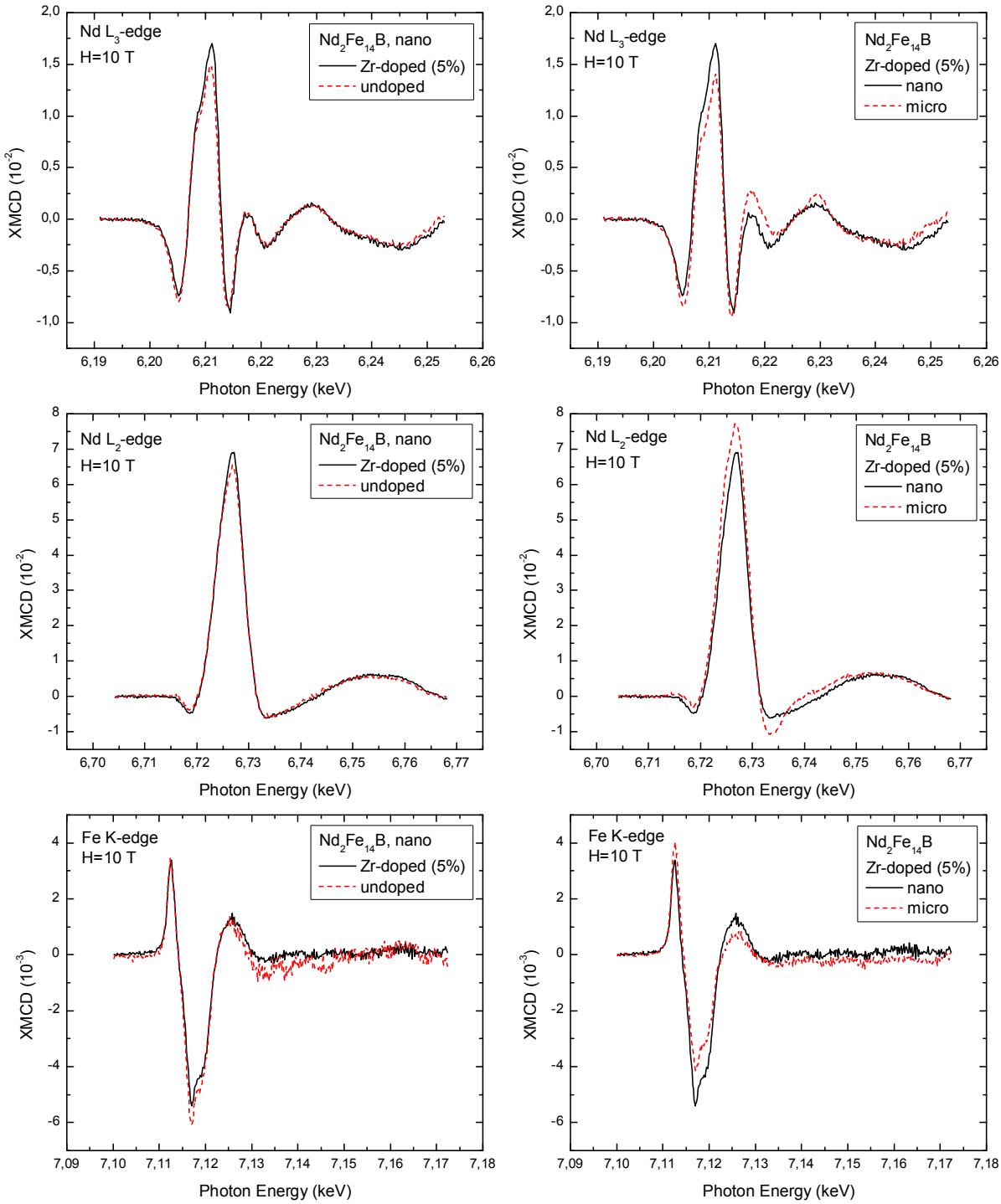

Fig. 1 Comparative XMCD spectra of doped $Nd_{10,4}Zr_{4,0}Fe_{79,2}B_{6,4}$ and pure $Nd_2Fe_{14}B$ nano-crystalline samples (left panel), and comparative XMCD spectra of nano- and micro-crystalline samples of $Nd_{10,4}Zr_{4,0}Fe_{79,2}B_{6,4}$ (right panel), taken at Nd-$L_{3,2}$ and K-Fe absorption edges in a magnetic field of 10 T