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Application for beam time at ESRF – Experimental Method

Structural characterization of Nd-Fe-B ultrathin films: towards the understanding of the origin of spin reorientation in $Nd_2Fe_{14}B$

Proposal Summary (should state the aims and scientific basis of the proposal) :

The scope of the study proposed to be carried out at ESRF is achieving a precise determination of the correlation between lattice strain, composition and magnetic properties and its variation with temperature in ultrathin films of Nd-Fe-B grown by Molecular Beam Epitaxy (MBE). These films are to be grown up to 10 nm with two buffer layers (either Fe (001) or Ir (001) on MgO (001)) to induce different lattice strain in the tetragonal $Nd_2Fe_{14}B$ structure. The effect of the Nd concentration will also be taken into analysis as an effective way of tuning the morphological, microstructural and magnetic properties of the Nd-Fe-B films. X-ray diffraction (XRD) measurements will provide essential information of accurate phase identification, lattice parameters and in-plane measurements at a determined range of temperatures (100 K-Room temperature). This structural information will be fundamental to complement spectroscopy and magnetic measurements in novel high coercive MBE-grown Nd-Fe-B thin films and to understand the role of lattice strain in the spin reorientation that takes place in $Nd_2Fe_{14}B$ at low temperature (below 135 K), thus affecting dramatically its magnetic performance when decreasing the operational temperature [1, 2].

Scientific background :

Rare-earth transition metal thin films have attracted a lot of attention due to their high magnetic anisotropy that makes them great candidates for several applications including high-density magnetic recording [3-4], microelectromechanical systems and actuators [5]. Rare-earth based thin films also allow the development of novel spintronic devices [6] and they are essential materials for energy-related technologies [7]. Furthermore, the study of certain elements in these rare-earth based systems such as interfaces or grain boundaries can provide a wider knowledge of their coercivity and magnetization reversal mechanisms, or their critical temperature transitions [2, 7-8]. Our study is focused on MBE grown epitaxial films with a tailored composition, that might open the path to a new generation of nanodevices. This research is framed under the H2020 FET-OPEN project UWIPOM2 (Ref. 857654), whose aim is to develop a wireless NdFeB-based microactuator for applications in microsurgery, with additional potential applications in transport and aerospace applications (therefore the importance of achieving an understanding and the possibility of influencing the spin reorientation effect in Nd-Fe-B films).

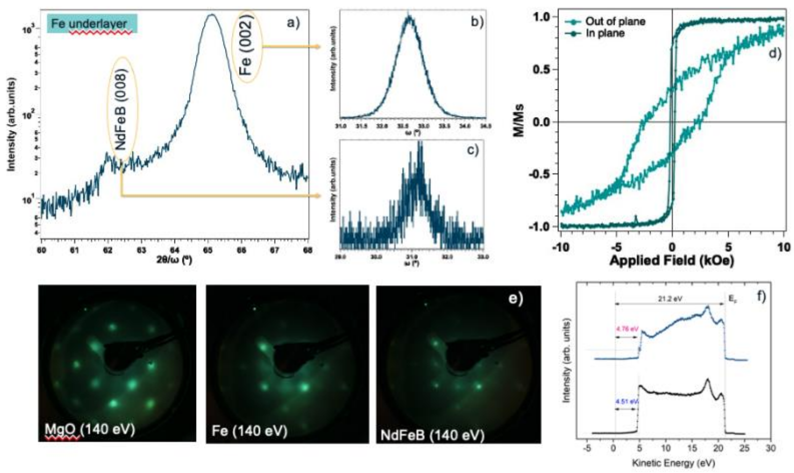

Figure 1: a) XRD pattern of an NdFeB thin film with an Fe underlayer, b) rocking curve measurement of Fe (002), c) rocking curve measurement of NdFeB (008), d) room temperature hysteresis loop e) LEED pattern of MgO substrate, Fe buffer layer and NdFeB film, f) work function measurements of Fe buffer (bottom) and NdFeB film (top).

In the proposed experiment, we intend to perform a deep structural characterization of Nd-Fe-B thin films growth using different buffer layers (Fe, Ir), as well as different concentration of Nd (from $Nd_{15}Fe_{77}B_8$ to $Nd_{18}Fe_{74}B_8$) in order to induce different degrees of strain on the lattice and consequently, modify the magnetic response of the system both at room and low temperatures. The epitaxial character of the films has already been corroborated in our home laboratory (Fig.1a-c) by XRD and low energy electron diffraction (LEED, Fig. 1-e). However, due to the low thickness of the films and the large lattice constant of the $Nd_2Fe_{14}B$ phase, a high fluence source as a synchrotron is mandatory in order to provide a more detailed structural characterization.
The XRD results will be contrasted with magnetic measurements performed by vibrating sample magnetometer (VSM) and magneto optical Kerr effect (MOKE) at low temperature. Preceding magnetic measurements on this system show high magnetic anisotropy in good accordance with the epitaxiallity of the thin films (Fig.1d). Photoemission spectroscopy (with both x-ray (XPS) and ultraviolet (UPS) sources) will allow us to do a thorough investigation of the electronic properties (i.e work function measurements) and will be performed to complete our previous studies (Fig 1f).

Experimental technique(s), required set-up(s), measurement strategy, sample details (quantity...etc):

Samples consist on epitaxial Nd-Fe-B thin films deposited on MgO(001) substrates by MBE co-evaporation using different buffer layers (Fe, Ir). All the process is performed under UHV conditions, without exposing the sample to air. Prior to the evaporation substrates are annealed in UHV at 600ºC during 30 minutes until LEED pattern is observed. Subsequently, for the metallic buffer, a 8 nm Fe/Ir layer is deposited at 250ºC. Afterwards, the Nd-Fe-B film is deposited by co-evaporation at 600ºC. Finally, a 15 nm V capping layer is deposited to protect the system from oxidation. Additionally, for each buffer three different concentrations of Nd are explored by tuning the Nd deposition rate. A total of 6 samples will be characterized.

To obtain a detailed structural characterization XRR scans at low and high angle, XRD rods, reciprocal space maps (RSM) and rocking scans are intended to be performed in NdFeB lattice positions for each sample, in order to characterize interface morphology, strain and crystallographic domain sizes. Taking advantage of the features of the BM25 SXRD+HAXPES experimental station, we intend to perform measurements at different temperatures in a range between RT and 100 K (i.e. including the transition temperature of 135 K). Additionally, high energy x-ray photoelectron spectroscopy (HAXPES) measurements will be performed so as to corroborate the stoichiometry of the film with bulk sensibility and complement previous XPS results.

Beamline(s) and beam time requested with justification :

The SXRD+HAXPES station at the Spanish CRG BM25-SpLine fulfil all the requirements for the successful performance of the proposed experiment. We foresee a total of 3 shifts for the alignment and complete characterization of each sample, including temperature dependence XRD and HAXPES measurements. Therefore, a total of 18 shifts is needed to perform the characterization of the complete set of samples.

Results expected and their significance in the respective field of research :

The proposed experiment aims to obtain a detailed structural characterization of Nd-Fe-B thin films deposited under different conditions and as a function of the temperature. The data obtained at the ESRF will be correlated with previous VSM and MOKE (room and low temperature) characterization in order to understand the role of the structural properties in the magnetic response of the films and optimize growth conditions. The growth of functional epitaxial Nd-Fe-B films with a tailored magnetic response is of great interest for their integration in microdevices for novel applications in microsurgery.

References

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