EUROPEAN SYNCHROTRON RADIATION FACILITY

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ESRF

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office via the User Portal:

https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Reports supporting requests for additional beam time

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** Texture inducement in technologically important sputtered thick films of NdFeB	**Experiment number**: **HS - 4375**
Beamline: BM23	**Date of experiment**: from: 09/04/2011 to: 11/04/2011	**Date of report**: 08/06/2013
Shifts: 9	**Local contact(s)**: Sakura Pascarelli	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Yuepeng Zhang*, Institut Néel, CNRS

Nora Dempsey*, Institut Néel, CNRS

Dominique Givord*, Institut Néel, CNRS

Report:

Extended X-ray Absorption Fine Structure (EXAFS) measurements were carried out to probe the local structure of NdFeB films deposited at different temperatures, with the aim to understanding texture development in these technologically important materials. Owing to the closeness of the absorption energy of the Fe K and Nd L1 edges, we studied x-ray absorption spectra near the K edge of Nd (43-45 keV) using BM23. Probing of Nd rather than Fe also facilitates data analysis (in the Nd2Fe14B structure there are 6 non-equivalent Fe sites but only 2 non-equivalent Nd sites).

Stacks of thick films (total thickness = 220 μm) of near stoichiometric composition were prepared and a powder sample of near stoichiometric $Nd_2Fe_{14}B$ was used as a reference sample. Two films were studied, one sputter deposited at room temperature and the other deposited on a subtrate heated to 450°C. EXAFS measurements were carried out in the temperature range 10-300 K. Data treatment and modeling was carried out using the ATHENA and ARTEMIS programs from the IFEFFIT simulation package. The crystallographic data for $Nd_2Fe_{14}B$, used for fitting the EXAFS data of the reference $Nd_2Fe_{14}B$ sample, was taken from Ref.1. Within experimental accuracy, the films appeared to be amorphous at all temperatures. However, a certain amount of short range order was identified in the film deposited at 450 °C (Table 1). The formation of such ordered regions during deposition at a temperature below the crytsallisation temperature of the $Nd_2Fe_{14}B$ phase, may serve as nucleation sites during the 750 °C post-deposition crystallisation of amorphous films deposited at 450 °C. This confirms our tentative explanation in Ref. 2 that the decrease in grain size with increasing deposition temperature was due to the formation of multiple nuclei during deposition on heated substrates. For technical reasons, it was not possible to carry out measurements at different angles to check for anisotropy in the absorption spectra, which could have explained the texture developed during crystallisation.

	$Nd_2Fe_{14}B$ crystals		NdFeB films T_{dep}=450 °C		NdFeB films T_{dep}=RT	
Near neighbors	number	r_{Nd-x} (Å)	number	r_{Nd-x} (Å)	number	r_{Nd-x} (Å)
Fe1	9	3.082	9	3.077	8.4	3.067
Fe2	7	3.275	6	3.248		
Nd	2.5	3.760	-	-	-	-

Table 1: Number and distance of Nd near neighbours, as extracted from the Fourier transform of EXAFS signal at the Nd K edge of the $Nd_2Fe_{14}B$ reference sample and Nd-Fe-B films deposited at room temperature or 450 °C. .

To complement this analysis, high resolution TEM imaging and 3D-Atom Probe analysis have since been carried out on these samples (collaboration NIMS, Tsukuba). Neither nanocrystals nor clusters were found in the film deposited at elevated temperature, confirming the short range nature of the order identified by EXAFS.

A paper reporting on the EXAFS,TEM and atom probe analysis of NdFeB thick films is in preparation.

Reference:

1. J. F. Herbst, J. J. Croat, F. E. Pinkerton, W. B. Yelon, Phys. Rev. B 29, 4176 (1984).
2. N. M. Dempsey, A. Walther, F. May, D. Givord, K. Khlopkov, O. Gutfleisch. Appl Phys Lett 90, 092509 (2007).