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 using the **Electronic Report Submission Application:**

http://193.49.43.2:8080/smis/servlet/UserUtils?start

Reports supporting requests for additional beam time

Reports can now 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:** **Self -assembled Fe and FePt nanoparticles in yttria-stabilized zirconia after iron-platinum implantation**	**Experiment number**: 20-02-677
Beamline:	**Date of experiment**: from: 20.06.2009 to: 23.06.2009	**Date of report**: 12.01.2010
Shifts: **9**	**Local contact(s)**: Dr. N. Jeutter	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

Series of experiments aiming the fabrication and characterization of Fe and FePt magnetic nanoparticles has been performed within the DFG Project PO 1275/2-1 "SEMAN". The current proposal was dedicated for structural investigation of Fe and FePt nanoparticles embedded in yttria stabilized zirconia (YSZ) as a result of Fe and FePt self-assembling, where Fe and Pt ions were doped by means of ion implantation at energies of 110 and 325 keV respectively.

The first part of the application assumed the investigation of secondary phases in YSZ crystals implanted with Fe only. The range of fluences of implanted Fe^+ ions was broadened in respect to initially planned from $1\times10^{14}\ldots1\times10^{16}$ (cm^{-2}) up to $1\times10^{14}\ldots3\times10^{17}$ (cm^{-2}).

The longitudinal $2\theta/\omega$ and transverse ω-scans in double- and triple crystal diffraction mode were performed for all samples in order to reveal the presence of magnetically active Fe-based secondary phases. No additional reflections from pure iron or iron oxides were found for the samples implanted with the fluences of $1\times10^{14}\ldots3\times10^{15}$ (cm^{-2}). High resolution scanning of the reciprocal space close to the vicinity of reciprocal lattice of YSZ substrate did not resolve significant increase of x-ray diffuse scattering with an increase of ion fluence. This result can be caused by the creation of solid YSZ:Fe solution which is possible in the case of low-fluence implantation. Another reason of the reflections absence of Fe (Fe_xO_y) phases is the amorphous structure of the inclusions. The samples implanted with the higher fluences of $1\times10^{16}\ldots3\times10^{17}$ (cm^{-2}) represent diffraction peaks from α-Fe, γ-Fe as well as from oxide-rich (Fe_xO_y) phase.

The x-ray diffraction study of these samples is in good agreement with Mössbauer spectroscopy performed at FZD. Combination of the experimental results obtained at ROBL and in-house research exhibits a variety of possible phases created in YSZ crystals after high-fluence Fe implantation. Moreover, it was found that Fe nanoparticles possess the Fe-core/ Fe_xO_y -shell architecture where the shell thickness is strongly dependent on the size of the nanoparticles. The results of the experiments are submitted to the Journal of Applied Physics. The second set of samples examined during the beamtime consists of YSZ crystals implanted with Fe and Co ions with overlapping ion distribution profiles at the depth of 55 nm. Although the ferromagnetic response of those samples was relatively strong (much stronger then in case of YSZ:Fe), the expected FePt (or any other ferromagnetic) phase has not been detected using x-ray diffraction. Such a confusing result forced us to attest, for a presence of contaminations, also the back, non-implanted side of specimens. It has been found that the backsides of substrates were contaminated with Fe giving the reflections of bcc iron (magnetic phase). Rough estimation of contaminations' volume made us convinced that iron presented at the non-implanted side of the samples is a main source of the observed ferromagnetism. Afterwards it was found that contaminations, most probably, were introduced by metallic clamps employed for the sample holding during the implantation of this particular set of samples. The non-implanted side of YSZ:Fe samples described early was also examined. Contaminations were not found in those samples. In order to avoid such situations in future, the list of recommendations for technical personnel and experimentalists involved to the research has been worked out.