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:** Microstructure design of electrodeposited GMR multilayers via annealing	**Experiment number**: MA1148
Beamline: BM20	**Date of experiment**: from: 16.12.2010 to: 20.12.2010	**Date of report**: 28.01.2011
Shifts: 12	**Local contact(s)**: Carsten Baehtz	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Torsten Schucknecht*, Ulrike Ratayski*, Christian Schimpf*, David Rafaja

TU Bergakademie Freiberg, Institute of Materials Science, Gustav-Zeuner-Str. 5,

09599 Freiberg, Germany

Report:

The aim of the experiment was the in situ investigation of the effect of the thermal treatment on the microstructure of electrodeposited (ED) Co/Cu multilayers (MLs) with GMR effect. A series of samples with constant layer thickness was deposited using the single bath method under different current densities. The current density is expected to affect strongly the amount of copper codeposited during the deposition of cobalt and consequently the microstructure of the ED MLs. As in situ methods, symmetrical WAXS and EXAFS were used. WAXS described the microstructure evolution during annealing; EXAFS revealed details on intermixing of the codeposited species.

The isothermal WAXS measurements showed a high thermal stability of the ED MLs up to 200°C. In samples prepared at high current densities, copper segregated after annealing at temperatures exceeding 350°C as seen on the formation of a distinct diffraction line from Cu in Fig. 1a. This was accompanied by an improvement of the multilayer satellites these samples. The samples deposited at low current densities contained hdp Co, which disappeared during the annealing up to 500°C (Fig. 1b) and did not occurred more upon cooling.

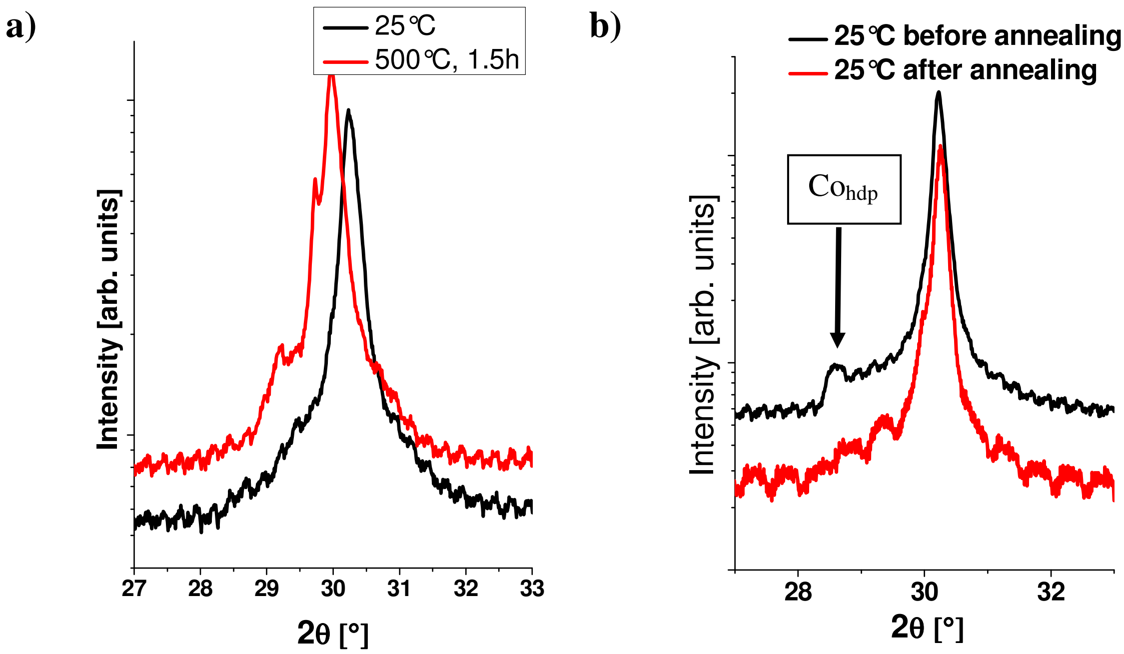

Fig. 1: a) sample deposited using a high current density in the initial state (black) and after 85 minutes at 500°C (red) – the shift of the peaks is due to the thermal expansion b) sample deposited at low current density before (black) and after (red) the heat treatment, the arrow indicates the position of the (100)-Co_{hdp} The periodic, wavelike intensities are an artefact of the line detector.

Selected samples were subjected to EXAFS measurements. The intention of these measurements was to describe the local chemical environment of Co and to identify the intermixing or decomposition effects. All samples were measured at the Co-Kβ edge (7700 eV). The background absorption $\mu_0(E)$ has been removed using the *Athena* program; the Fourier transform $\chi(E)$of the absorption fine structure was loaded into the *Artemis* program. Both programs are included in the IFeffit package. The analysis of the data was done using the 'model for doped materials' explained by B. Ravel at the 2001 EXAFS workshop [1]. This model calculates the intermixing parameter x. Several other parameters like the relative displacements ΔR of the first neighbours of the absorbing Co atom were used for refinement as well. The resulting fits are shown in Fig. 2. In the sample deposited at high current density (j = 84 mA/cm²), Co is surrounded mainly by Co ($x = 0.14$). Due to a larger atomic radius of Cu as compared to Co, Cu atoms are shifted in outward direction with respect to the original cell. An opposite shift was observed for Co. The annealing leads to an intermixing of Co and Cu until a random distribution around the Co absorber is achieved ($x = 0.57$). The intermixing is expected to occur as substitutional occupation of the Wyckoff positions of the fcc cell by Co and Cu, which can be the cause of the vanishing displacement

(ΔR) of Co and Cu for the annealed sample. Lower current deposition density (j = 16 mA/cm²) leads to enhanced intermixing of Co and Cu ($x = 0.32$). The opposite displacement of Co and Cu persists.

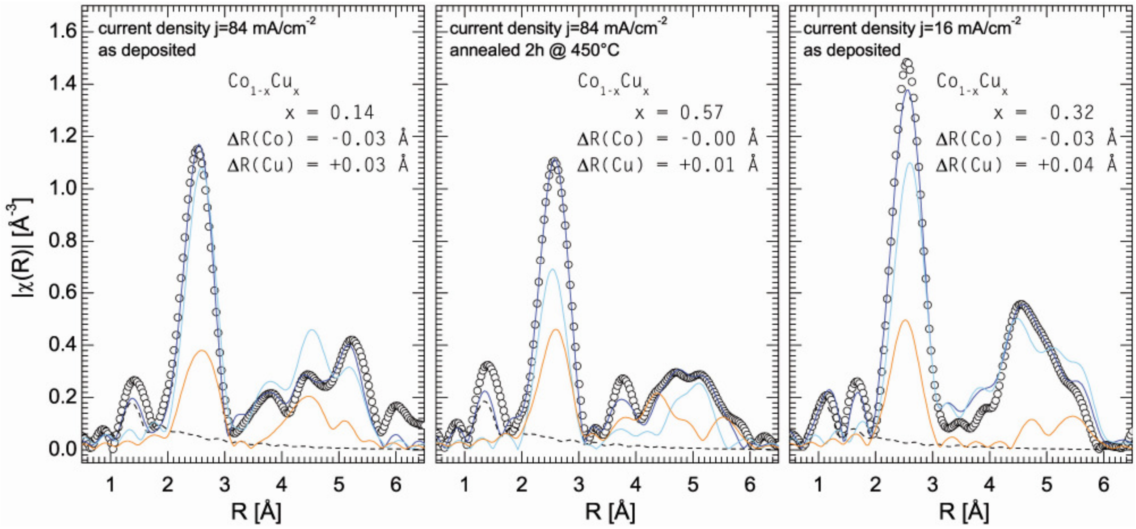

Fig. 2: Results of the fitting of EXAFS spectra measured for Co-Cu multilayers deposited at high deposition current density, the same sample after annealing and a sample deposited at low deposition current density. Results are given in the text. The dots represent experimental data, blue line the resulting fit, cyan line the Co contribution, orange line the Cu contribution and dashed line the background

[1] http://leonardo.phys.washington.edu/~ravel/talks/course/notes.pdf