Experimental report of the beamtime 25-01-1022

Title. Investigation of the structural properties of electrodeposited Fe-Ga-O thin films by XAS

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Beamline. BM25-A

Experiment performed during: 16/March/2017@08H00 - 18/March/2017@08H00

Summary

This proposal aimed the investigation of electrodeposited Ga-Fe-O thin films by means of XAS to establish the possibility of using this growth technique to synthetize this material system.

Experimental procedure

Standard XAFS experiments were carried out in Ga-Fe-O thin films grown in different electrolytes and using different overpotentials. XAFS spectra were acquired in Fe and Ga K-edges in fluorescence yield mode and in transmission for the powder and foil references.

We satisfactory got good quality spectra merging from at least 4 single spectra. The EXAFS spectra were acquired upon photoelectron wavenumber values of 15 $Å^{-1}$ for the Fe and Ga K-edges. The global good quality of the spectra from all the elements makes possible to obtain valuable information from all of them.

Experimental Results

We have firstly analyzed XAS spectra of Ga being observed evidences of oxidation (Fig. 1). From the XANES region we can infer the oxidation of Ga from the shift of the absorption edge and from the intensity increase of the main absorption peak. Further evidences of the partial oxidation of Ga can be found when performed the Fourier Transform (FT) of EXAFS (Figure 2). The FT shows a strong metallic component (peak at 2 Å). However, there appear components related to oxides at 1.5 Å (Ga-O) that we have marked with an arrow and another component related to Ga-Ga at 3.75 Å, also marked with an arrow (figure 2). Therefore, Ga keeps its metallic state being only partially oxidized.

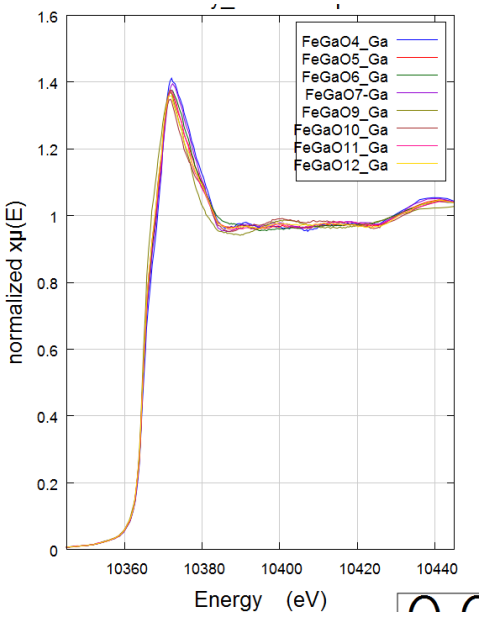

Figure 1. XANES spectra in the Ga edge of different Ga-Fe-O layers.

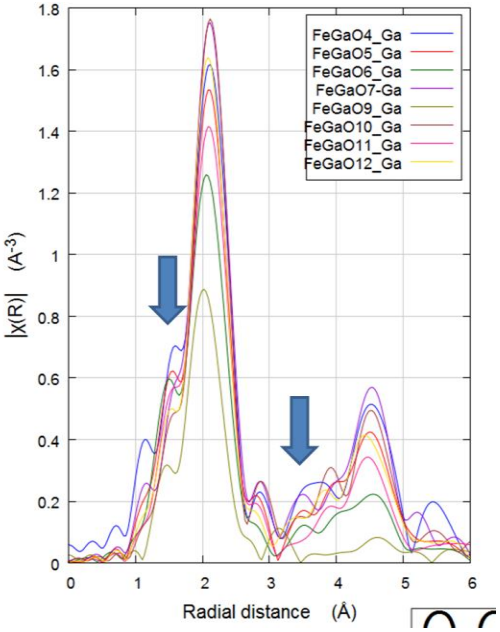

Figure 2. Fourier transform of EXAFS measurements of the Ga K-edge.

When analyzing the Fe absorption edge, we observe there is not a shift of any of the absorption edges with respect to pure Fe (Figure 3) that clearly indicates that Fe is its metallic state. We only observe a distortion of the absorption spectra that can be related to the polycrystallinity of the samples.

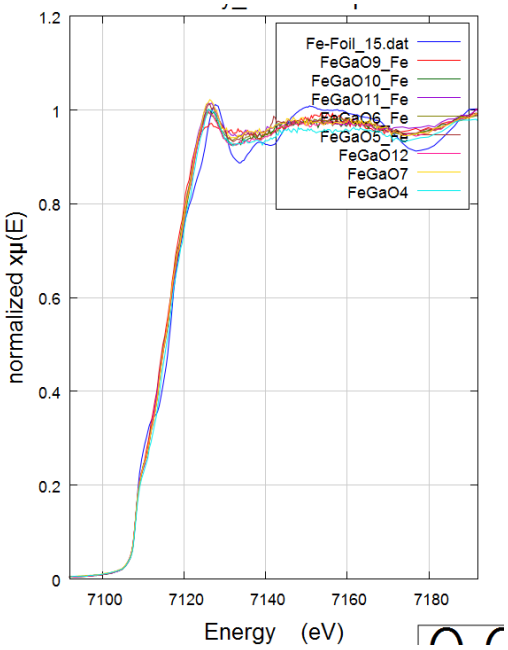

Figure 3. XANES spectra measured in the Fe K-edge of different Ga-Fe-O layers.

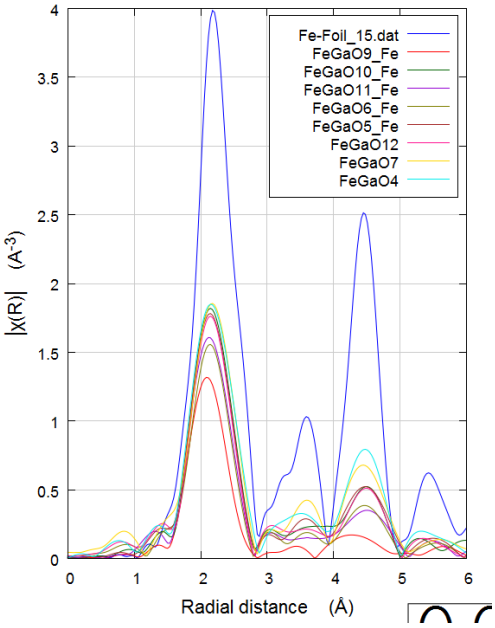

Figure 4. Fourier transform of EXAFS measurements of the Fe K-edge.

The metallic behavior of Fe is further confirmed by the FT of the EXAFS measured in the Fe K-edge (Figure 4). All the components appear in the same values as for the metallic reference. There is only one component around 1 Å that could be related to the oxidation of Fe. However, its intensity is so small that is hardly distinguished from the background.

Discussion

Standard x-ray diffractometry measurements performed in these layers show diffraction peaks that could be related to GFO as well as to Ga oxides. After XAS measurements, we can confirm the presence of Ga oxides but not of GFO. Therefore, electrodeposited layers studied in this proposal consist of Fe-Ga metallic alloys together with Ga oxides.

Conclusions and perspectives

The XAFS analysis of the ternary alloys shows: i) the feasibility of this technique for studying these systems, ii) we have observed indications of partial oxidation in Ga, iii) Fe keeps its metallic state being only observed very little, almost negligible, indications of its oxidation.

Considering these results we can conclude that the layers studied in this proposal consist preferentially of Fe-Ga alloys together with Ga oxides. As already reported in a previous work [1], Ga prevents the oxidation of Fe keeping it in its metallic state being therefore not possible to produce GFO with these growth conditions.

We plan to prepare new samples changing the electrolyte composition, pH and overpotentials because we have not found clear indication of the formation of GFO by XAS when using the growth conditions used in this work. These growth conditions were prepared considering a previous work on metallic Fe-Ga alloys [2] and after this XAS experiment, we have ideas about how to optimize the electrolyte.

Problems

There was not any problem during the scheduled beam time.

References

[1] P. Alvarez-Alvarez, A. Prados, A. Muñoz-Noval, and R. Ranchal. J. Alloys Compnd. **713** (2017) 229.

[2] R. Ranchal and D. Maestre, J. Phys. D: Appl. Phys. **47** (2014) 355004.