Report for ESRF Experiment 25-01-1046 - XAS study of electrodeposited Ni-FeMn core-shell nanowires

Introduction

Nowadays, one of the more active research lines in the field of magnetic materials is the search for rare-earth-free permanent magnets. The European Union is strongly promoting this research line in order to reduce the economic dependence on China, main exporter of rare-earth materials in the world. The ideal milestone is the development of a permanent magnet based on Fe, the most common and cheap magnetic element. In this sense, the coupling between ferromagnetic and antiferromagnetic iron based materials at the scale of nanometers can be used to produce magnetically hard material taking advantage of the expected strong interfacial anisotropy [1,2]. Electrodeposition is a very suitable technique to grow structures with high-aspect ratio[3], which is essential in these applications because it allows taking advantage of the shape anisotropy as an additional contribution to the total anisotropy in the magnet [4]. In this context, the electrodeposition of an iron-based antiferromagnet is a challenge.

In a previous work, We successfully electrodeposited FexMn1-x alloys showing the possibility to electrodeposited antiferromagnetic FeMn alloys [5]. Now, we have electrodeposited FeMn nanowires trying to control the magnetic behaviour of these FeMn nanowires; It determines its applicability in the synthesis of nanostructures with interfacial exchange bias coupling in order to obtain rare-earth-free permanent magnets. In a second step, we have electrodeposited core-shell nanowires with a nickel core and a FeMn shell. To ensure that the magnetic hardening observed in these nanowires is coming from interfacial exchange bias coupling it is compulsory to used synchrotron radiation to study (i) if the shell is a FeMn alloy and (ii) if there is no oxides in the structure.

Experimental

Ni-FexMn1-x core-shell nanowires have been grown by electrochemical deposition. Ni core has been growth using sulphate-based electrolytes using polycarbonate membranes as a template. In a second step, FeMn shell was electrodeposited using chloride-based electrolytes. Electrodeposition was carried out at 60º C for Ni growth and at room temperature for FeMn growth, under stirring, in a three-electrode configuration, using Pt gauze as a counter electrode and an Ag/AgCl reference electrode.

The morphollogy of the Ni-FeMn core shell nanowires have been studied using different techniques (scanning electron microscopy, energy disspersive spectroscopy and x-ray diffraction). X-ray absorption experiments were performed on the SpLine beamline at the European Synchrotron Radiation Facility (ESRF). We carried out X-ray absorption near edge structure (XANES) spectroscopy experiments at the Fe K-edge (7.11 keV) and Mn K-edge (6.539 keV) in fluorescence mode at room temperature. Magnetic characterizacion has been carried out in SQUID magnetometers.

Results

We have electrodeposited FeMn nanowires and studied their structural and magnetic properties as a function of the Fe/Mn rate. The structural measurements are necessary to get more insight into the structure. In particular, it is necessary to know the local environment of Mn and and Fe atoms in the films to elucidate whether they are an alloy. We have performed XAS measurements on different set of nanowires with coposition Mn70Fe30, Mn80Fe20 and Mn45Fe55. We have also measured pure Fe and Mn foils as well as a Mn50Fe50 foil as a reference. Fig. 1 shows the results obtained in the XAS measurements in the ESRF beamtime. In fig. 1.a., we show the XAFS signals at Fe K-edge of Fe and FeMn foils and Fe-Oxides references. The comparison between the nanowires and the references provides us with information about the structure on the wires. Figure 1.b., show the XAFS signals at Fe K-edge of FeMn nanowires. As can be seen in this figure, the XAFS signals at Fe K-edge of FeMn nanowires, show the characteristic spectra of iron oxide and mixtures of oxidized iron and metal iron in other. This high content of oxides makes difficult to determine the chemical state and the final properties of the samples. New growth experiment at Ar or N atmosphere will be necessary to avoid the oxide formation.

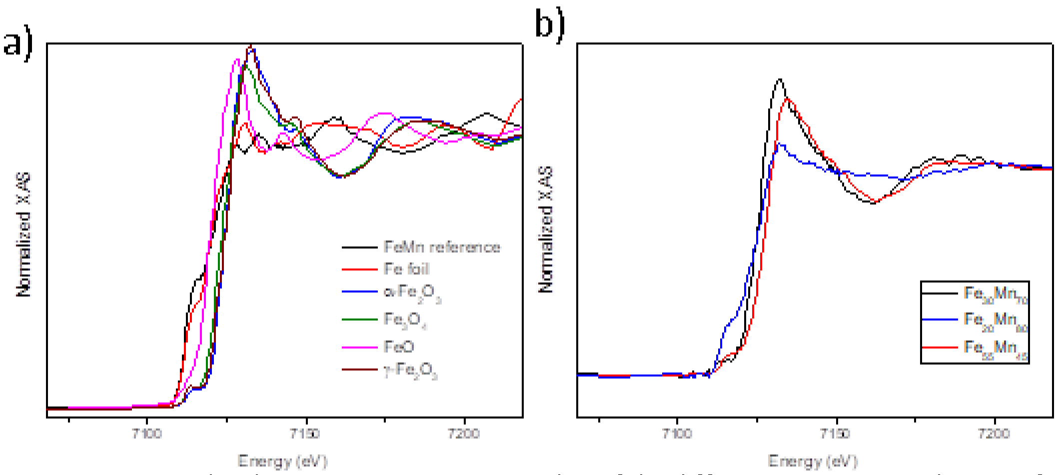

Figure 1. Normalized XANES spectra at Fe K-edge of the different Fe, FeMn and FeO$_x$ reference samples. (b) Normalized XANES spectra at the Fe K-edge of Fe$_x$Mn$_{1-x}$ nanowires measured at SpLine (ESRF).

References

References

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