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NMR study of the structural properties of electrodeposited Co/Cu multilayers

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Abstract

Structural properties, such as crystal structure and interface roughness, of different series of electrodeposited Co/Cu multilayers were studied with ⁵⁹Co nuclear magnetic resonance. The presence of non-randomly distributed Cu impurities in the Co layers is evident from the NMR spectra.

Recently it has been shown that electrodeposition is a competitive method for growing multilayered films [1,2] and multilayered nanowires [3,4] exhibiting giant magnetoresistance. To obtain detailed information about the structure of the bulk magnetic material and about the interface roughness and topology of electrodeposited multilayers, we studied Co/Cu multilayers with ⁵⁹Co nuclear magnetic resonance (NMR). We will discuss the stabilization of the fcc structure of the Co by fcc Cu, the presence of Cu impurities in the Co layers, and the influence of levelling agents on the microstructure.

The Co/Cu multilayers were electrochemically deposited on Si(100) wafers at room temperature. Prior to electroplating, a base layer of 200 Å Cu or 500 Å Au was sputtered or evaporated onto the Si wafers. Electrodeposition was performed in a single cell from a sulphate-based electrolyte containing Co²⁺ and Cu²⁺ ions. X-ray diffraction (XRD) revealed a (111) texture for the multilayers grown on Au base layers and a (100) texture for the multilayers grown on Cu base layers. More information about the growth procedure, the XRD measurements, and the magnetoresistance of these samples will be reported elsewhere [5].

In Fig. 1 the NMR spectra of thick Co films, grown by electrodeposition in an electrolyte containing both Co²⁺ and Cu²⁺ ions, are plotted. The intensity for hyperfine fields, B_{hf} , larger than 20.75 T arises from Co atoms with only Co neighbours. For the sample grown on a Au base layer, the structure of the Co is a mixture of fcc Co, hcp

Co, and Co in stacking faults. For the Co film grown on the Cu base layer, the structure of the Co is mainly fcc since the fcc phase of the Co is stabilized by the Cu base layer which has an fcc structure and a rather small mismatch with the Co lattice. The intensity for $B_{\text{hf}} < 20.75$ T arises from Co atoms with one or more Cu neighbours. For the sample grown on Cu, this part of the spectrum is also shown enlarged by a factor of seven. The intensities arising from Co atoms with 1, 2, and 3 Cu neighbours can clearly be distinguished in the figure and are numbered. The decrease in hyperfine field when a Co atom is replaced by a Cu atom in the nearest neighbour shell is about 1.2 T (average distance between the numbered lines in Fig. 1). This value is smaller than the 1.6 T reported in the literature [6]. For the film grown on Au, the lines are much broader and the different surroundings can not be separated. For a random distribution of a certain percentage of Cu atoms in a Co layer, the intensity ratios between the different alloy lines and the bulk Co line can be predicted with the binomial distribution law. A comparison of this prediction with the measured intensity ratios shows that the distribution of Cu atoms in the Co layer is not random and that the concentration of Cu impurities in the Co layer is around 2%. This value is comparable with the value of 4% found by X-ray fluorescence [5].

Fig. 2 shows the NMR spectra of (100) (x Co + 40 Å Cu) multilayers grown on Cu base layers. The amount of hcp Co and Co in stacking faults is very small in these multilayers, which is a result of the stabilization of fcc Co by the fcc Cu base and spacer layers. The intensity for $B_{\text{hf}} < 20.75$ T arises from Co atoms at the Co/Cu interfaces and/or from Co atoms in the Co layers with one or more Cu neighbours. The intensity arising from the alloy is

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Fig. 1. NMR spectra of thick Co films electrodeposited on a Cu and on a Au base layer from an electrolyte containing both Co^{2+} and Cu^{2+} ions. The intensity for $B_{\text{hf}} > 20.75$ T arises from Co atoms with only Co neighbours. The intensity for $B_{\text{hf}} < 20.75$ T arises from Co atoms with one or more Cu neighbours. The spectra were measured at 1.6 K and are corrected for enhancement.

proportional to the Co layer thickness and, assuming that the interface roughness is independent of the Co layer thickness, the intensity from the interfaces is constant. The fact that the intensities below 20.75 T coincide for the different samples (except for the sample with 10 Å Co around 17 T) suggests that the contribution from the alloy is much smaller than the contribution from the interfaces. This suggestion is corroborated by the consideration that for multilayers with small Co layer thicknesses a relative large number of Co atoms is located at the interfaces, e.g. for 10 Å Co (5 monolayers) at least 40% of the Co atoms are located at the Co/Cu interfaces. The intensity from these atoms is much larger than the alloy intensity arising from about 2% Cu in the Co layers. From an analysis of the decrease of the bulk Co intensity with decreasing Co layer thicknesses, we find that for Co thicknesses smaller than approximately 6 Å the bulk contribution to the intensity vanishes, which corresponds to an interface roughness of about 3 Å per interface.

The NMR spectra of Co/Cu multilayers electrodeposited on Au base layers (not shown), resulting in a (111) texture, are comparable to the spectra of similar (100)-textured multilayers grown on Cu base layers. Only the width of the bulk lines is larger for the multilayers grown on Au. This is probably due to the larger mismatch between the Au base layer and the multilayer, resulting in more hcp Co and stacking faults. Although for perfectly flat (111) interfaces and for perfectly flat (100) interfaces different interface spectra are anticipated, the interface spectra of the present (111) and (100) multilayers are comparable.

Fig. 2. NMR spectra of (100) (x Co + 40 Å Cu) multilayers normalized to the Co layer thickness. The vertical lines denote the values of bulk fcc Co (21.6 T) and bulk hcp Co (22.6 T). The intensity for hyperfine fields lower than 20.2 T is also shown magnified by a factor of five. The spectra were measured at 1.6 K and are corrected for enhancement.

The addition of frequently used levelling agents to the electrolyte dramatically changes the NMR spectra. For example, for the sample 200 Å Cu + 50 × (16 Å Co + 40 Å Cu) grown with thiourea, the NMR spectrum indicates intermixing at the interfaces over a region of at least several monolayers and with almost no Co atoms contributing to the bulk signal. This picture of very rough, diffuse interfaces is supported by XRD measurements; for the multilayers grown with thiourea no multilayer satellites were observed [5]. Furthermore, the magnetoresistance of these samples was an order of magnitude smaller than for the samples grown without a levelling agent (less than 1% compared to around 10%) [5].

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