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Atomic scale structure investigations of epitaxial Fe/Cr multilayers

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ABSTRACT

Fe/Cr multilayers were deposited by molecular beam epitaxy on the MgO(1 0 0) substrate. Structural properties of the samples were analyzed by low energy electron diffraction, high resolution transmission electron microscopy (HRTEM), as well as by X-ray reflectivity, conversion electron Mössbauer spectroscopy (CEMS) and Auger electron spectroscopy. Investigations revealed multilayered system built of well-ordered Fe and Cr thin films with (1 0 0) orientation. A high geometrical perfection of the system, i.e. planar form of interfaces and reproducible thickness of layers, was also proven. Fe/Cr interface roughness was determined to be 2–3 atomic layers. CEMS studies allowed to analyze at atomic scale the structure of buried Fe/Cr interfaces, as well as to distinguish origin of interface roughness. Roughnesses resulting from interface corrugations and from the Fe–Cr interdiffusion at interfaces were observed. Fe/Cr multilayers showed strong antiferromagnetic coupling of Fe layers.

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1. Introduction

Giant magnetoresistance (GMR) has been investigated in a large number of magnetic multilayer systems as they may both serve as an interesting model material and can be used for data storing applications [1–3]. The importance of multilayer quality for their magnetic properties steers a substantial part of interests toward deeply buried interfaces.

Despite the progress achieved in investigations of Fe/Cr multilayers, the effect of interface roughness of such layers on their magnetic behavior is still not fully understood. The reported results show enhancement of GMR with the increase of interfacial roughness due to the strong spin-dependent scattering of electrons [4,5]. In contrast, other groups demonstrate that GMR is reduced by presence of intermixed region [6]. A wide variation of results may arise from insufficient analysis of interface roughness, which usually does not separate the contributions from the corrugations and atom intermixing. Therefore, explaining this inconsistency requires more experiments aimed at detailed microstructure characterization accompanied by magnetic measurements.

A best way to characterize the multilayers phase and chemical composition, as well as their interfaces, is direct observation of their cross-sections by transmission electron microscopy. Due to close values of lattice parameters and atomic numbers of Fe and Cr, the HRTEM observations should be performed using phase contrast and High Angle Annular Dark Field (HAADF) techniques [7]. However, while the techniques mentioned above are powerful enough to reveal a layered structure, they are less efficient in description of an accurate picture of the interfacial roughness in atomic scale. This can be better resolved by other methods, such as X-ray reflectivity (XRR) and conversion electron Mössbauer spectroscopy. Additionally, the CEMS technique allows to distinguish roughness originating from interface corrugations and atom intermixing and to obtain information on the magnetic properties of Fe layers. The aim of this study was to fabricate Fe/Cr multilayers and characterize their microstructure with the HRTEM/HAAD, XRR and CEMS techniques in combination with magnetic measurements.

2. Materials and methods

Fe/Cr multilayers were deposited by molecular beam epitaxy (MBE) on MgO(1 0 0) and capped with Cr layer. Both Fe and Cr layers have the bcc lattice with nearly identical lattice parameters of 0.2866 nm and 0.2879 nm, respectively, which create optimal conditions for epitaxial growth. In the case of the MgO(1 0 0) substrate, a proper epitaxial growth of Fe and Cr can be achieved due to rotation of both lattices (Fe/Cr and MgO) by 45°, resulting in a small lattice misfit of ~3.11% [8]. The substrates were obtained by ex situ cleaving of MgO(1 0 0) single crystal cubes in a chamber filled with N_2 atmosphere. The evaporation process was performed at room temperature with the base pressure during deposition of 10^{-8} Pa using alumina crucibles wrapped around with tungsten coil. During

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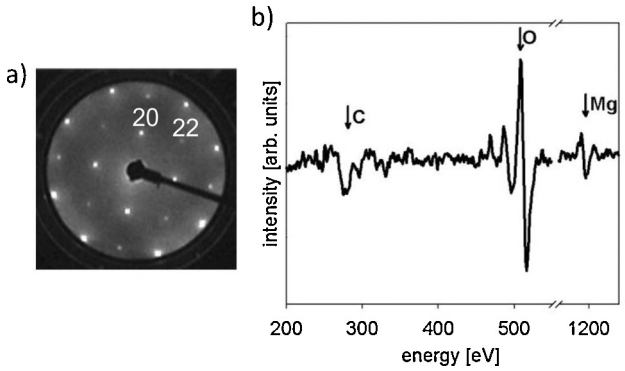

Fig. 1. Low energy electron diffraction pattern at energy equal to 180 eV (a), and Auger electron spectrum with peaks characteristic for C (272 eV), O (503 eV) and Mg (1174 eV) (b), registered on cleaved MgO(1 0 0) surface.

the preparation the evaporation rate and the layer thickness were controlled by a quartz balance. The nominal thicknesses of Fe and Cr layers were equal to 2 nm and 1.2 nm, respectively. The evaporation rate changed from 2.5 Å/min for Fe layers to 3 Å/min for Cr layers. The 5 nm Cr cap layer was evaporated at a rate of 6 Å/min. MgO, Fe and Cr layer surfaces were tested in situ using Low Energy Electron Diffraction (LEED) and Auger electron spectroscopy with a four-grid retarding field analyzer spectrometer. The LEED patterns were registered in a wide energy range (54–200 eV), while AES spectra were collected at three energy ranges: 30–60 eV, 200–600 eV and 1100–1300 eV. The samples were studied ex situ by X-ray reflectivity using Philips X'Pert MRD Pro diffractometer with Cu Kα radiation operated at 40 kV and 30 mA converted to a parallel beam. All XRR spectra were measured in coplanar geometry with specular θ–2θ scans and fitted using X'Pert Reflectivity software. Structure of Fe/Cr multilayers, especially Fe/Cr interfaces was studied by conversion electron Mössbauer spectroscopy. All CEMS measurements were performed at room temperature with 100 mCi Co(Rh) source, using He-10% gas flow counter with γ-ray propagation direction perpendicular to the sample surface. The Mössbauer spectra were fitted using Voigt-based method of Rancourt and Ping [9] as a sum of Gaussian components for the hyperfine magnetic field (B_{hf}), isomer shift and quadrupole splitting distributions. The crystal structure and epitaxial growth were investigated by high resolution transmission electron microscopy in cross-sectional view. Layered structure was observed using scanning mode of transmission electron microscopy (STEM) with a high angle annular dark field detector and energy dispersive spectroscopy (EDS). Hysteresis loops were measured with Kerr effect in longitudinal geometry.

3. Results and discussion

Fig. 1a shows a typical LEED pattern for the MgO(1 0 0) substrate with a low intensity background, and sharp spots in the whole electron energy range. This indicates a low level of point defects (contaminations) and a presence of large flat terraces that do not give broadening of the spots. The AES spectrum in Fig. 1b shows three lines originating from carbon (KLL transition – 272 eV), oxygen (KLL transition – 503 eV) and magnesium (KLL transition – 1174 eV) with intensity ratio of I_C/I_{Mg_2} and I_C/I_O equal to 1 and 0.2, respectively. Perfect background-free LEED pattern and a relatively small C line intensity qualify these substrates for the MBE deposition process.

Crystallographic symmetry of deposited layers and the quality of the surfaces were measured in situ by LEED, as shown in Fig. 2. The presented patterns were registered on the Fe layer, the Fe/Cr bilayer $(Fe/Cr)_6$ multilayers and on the Cr cap layer. The LEED patterns were collected in a wide energy range, but in Fig. 2 the pictures measured at approximately 180 eV are shown. All pictures indicate four-fold symmetry that could be assigned to the bcc structure of a (1 0 0) zone axis in the bcc lattice. The spot broadening and high intensity of the background, especially on the first Fe layer, indicate that growth of Fe on MgO does not proceed layer by layer, leading to island-like morphology of the Fe layer. This likely results from the substantially larger surface-free energy of Fe(1 0 0) (4.0 J/m^2) as compared to MgO(1 0 0) (1.2 J/m^2) [10]. Despite the island-like growth at the beginning, the diffraction patterns measured at all preparation stages confirmed-well ordered structure and epitaxial growth, even on the cap layer. LEED patterns measured on the MgO(1 0 0) substrate (Fig. 1a) and on the Fe(1 0 0) layer (Fig. 2a) clearly indicate rotation of (20) spots of Fe by 45° in relation to (20) spots of MgO, as well as corresponding positions of (11) spots of Fe and (20) spots of MgO.

In Fig. 3, cross-sectional HRTEM images with Fast Fourier Transform (FFT) patterns recorded on Fe/Cr multilayers (a), multilayers/MgO interface (b) and the MgO substrate (c) are shown. A continuous character of the contrast across Fe/Cr multilayers is due to the epitaxial, pseudomorphological growth, and a very small lattice mismatch between Fe and Cr. FFT pattern of Fe/Cr multilayers shows four-fold symmetry of (0 1 0) bcc surface, while Fourier transformation of MgO image indicates (1 1 0) fcc surface orientation (Fig. 3c). Both surfaces were obtained by a vertical cut of as-deposited sample and therefore are perpendicular to surfaces observed by LEED. FTT registered on multilayers/MgO interface (Fig. 3b) shows a superposition of the spots coming from Fe/Cr multilayers (darker spots) and the MgO substrate (lighter spots). The (1 1 0) spots of multilayer are rotated by ~10° in relation to (1 1 1) spots of MgO while (0 2 0) spots of Fe/Cr are aligned along (2 0 0) spots of MgO. Therefore, Fig. 3b reveals an epitaxial relation of the Fe/Cr multilayer on MgO(1 0 0) as followed: (2 0 0) Cr II (2 0 0) MgO and [0 0 1] Cr II [0 1 1] MgO. This dependence confirms expected rotation of Fe/Cr multilayers in relation to the MgO substrate.

The continuity of layers, especially important for magnetic properties of Fe/Cr multilayers [11], was investigated with AES spectra. Typical LMM decay used in AES analysis is observed for Fe and Cr at energy range between 491 eV and 705 eV. To increase surface sensitivity of AES measurements, instead of LMM transitions, for which mean electron free path is above 1 nm, MMM transition, also called

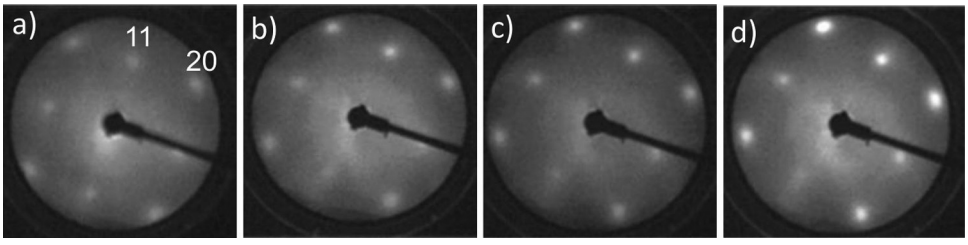

Fig. 2. Low energy electron diffraction pattern measured at energy about 180 eV on a first Fe layer (a), on a first Fe/Cr bilayer (b), then on $(Fe/Cr)_6$ multilayers (c) and on Cr cap layer (d).

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