The effect of annealing on the junction profile of CoFeB/MgO tunnel junctions

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The tunnelling magnetoresistance of CoFeB/MgO tunnel junctions is exceptionally high, although the electrodes and the barrier are grown at room temperature in the amorphous state. For their functionality annealing steps up to high temperatures are required. We have analyzed in detail the changes in the chemical and magnetization profile upon annealing up to 360°. The multilayers used for this study are similar to those which are used in magnetic tunnel junctions, however with five repeats. In particular, we have used hard non-resonant and soft resonant magnetic x-ray scattering in order to unravel any changes upon annealing. The multilayers exhibit superior structural quality, which hardly changes with annealing. Surprisingly, only little recrystallization of the CoFeB and the MgO layers can be discerned by x-ray diffraction. © *2010 American Institute of Physics*. [doi:10.1063/1.3483956]

I. INTRODUCTION

CoFeB/MgO/CoFeB magnetic tunnel junctions (MTJs) are well known for their very high tunnel magnetoresistance (TMR) values even at room temperature.[1,2] It has been proposed that these high values are due to coherent spin tunneling across the MgO barrier, in contrast to more diffusive tunneling that would provide lower TMR values. Coherent tunneling requires the connection of wave functions on either side of the tunnel barrier,[3,4] which is only possible for epitaxial magnetic layers, such as in single crystalline Fe/MgO/Fe(001).[5,6] However, in CoFeB/MgO/CoFeB the magnetic layers are amorphous after deposition at room temperature because of the high boron content of up to 20%. Amorphous layers guarantee smooth interfaces on an atomic scale, which is also important but not sufficient for high TMR values. It is generally believed that the MgO layer contains (001) textured nanocrystallites which serve as a template for the recrystallization of CoFeB layers upon high temperature annealing and during removal of boron. Therefore, MTJs with amorphous CoFeB electrodes require high temperature annealing steps to steadily improve their TMR values. Although this has indeed been confirmed experimentally, up to now it remains unclear how well recrystallization of the CoFe and MgO layers upon annealing takes place and where the boron content remains. It has been speculated that boron diffuses either into the MgO layer[7] or that it becomes trapped in Ta layers that serve as seed and capping layers in MTJs.[8,9]

Concerning the nanocrystallinity of MgO, Choi *et al.*[10] have shown by transmission electron microscopy that after annealing at 360 °C for two hours an epitaxial relation between CoFeB and the MgO layer can be observed. They suggest a 45°-type epitaxy (001)[110]CoFeB∥(001)×[100]MgO, which is usually observed in these type of heterolayers. This work confirms a crystalline albeit textured and columnar growth after annealing of both MgO and CoFe, layers. Wang *et al.*[11] confirmed by x-ray scattering an improved crystallinity of the MgO(001) layer as the reason for the increasing TMR value measured at high annealing temperatures. At 380 °C, the MgO(002) Bragg reflection intensity increases with annealing time and the full width at half maximum (FWHM) decreases. In this study, the MgO layer with 15 nm was rather thick, much thicker than usually used in MTJs. It is known that the first 1–1.6 nm thick MgO layer is in an amorphous state after annealing. In layers that are such thin it is not clear whether MgO still recrystallizes at high temperatures and whether it can still serve as a template for the crystallization of the neighboring CoFeB layer.

Rumaiz *et al.*[12] have focused their attention on the structural development of the CoFeB layer by investigating the local environment of Co and Fe using EXAFS during annealing up to 400 °C. They conclude that prior to annealing, the CoFeB films are amorphous with boron dissolved interstitially in the CoFe layer. Upon annealing, they observe that the CoFe layer crystallizes to a bcc structure with a concomitant depletion of boron from the layer. A corresponding x-ray structural analysis is not available at the present time. It has been suggested by Read *et al.*[13] that boron may diffuse to the interface, forming a variety of different oxides upon annealing at high temperatures. Thus the interface might serve as a sink for the boron content without changing the properties as tunnel barrier.

The sharpness of the interfaces and the magnetization profile within the MTJs as a function of annealing temperature has also received much attention in the past. Pym *et al.*[14] have shown by x-ray reflectivity measurements on CoFeB/AlO_x-multilayers that the interfaces between CoFeB

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and AlO_x sharpen considerably upon annealing up to 400 °C. In a similar study on CoFeB/MgO multilayers using both x-ray and neutron reflectivity, Vadalá *et al.*[15] not only compared different growth modes for the heterojunctions but also their structural changes upon annealing. In these multilayers the interface properties depend more on the preparation method used, then on the annealing temperature. Hindmarch *et al.*[16] have used soft x-ray absorption spectroscopy and magnetic circular dichroism for the investigation of the magnetic properties of Co and Fe in CoFeB/MgO heterostructures. They conclude that in the as prepared state some of the Fe atoms become oxidized at the interface to MgO, reducing the magnetization and the polarization close to the interface. Annealing appears to resolve only partially this problem. In a similar study, Anderson *et al.*[17] have analyzed the structural and magnetic changes in CoFeB/MgO tunnel junctions during annealing using soft x-ray resonant magnetic scattering. Although the magnetization profile has not been analyzed, the authors conclude from their studies that the asymmetry ratio between the right and left circular polarized light at the Co and Fe edges increases, indicative for a higher magnetization of the CoFeB layers with increasing annealing temperature.

In the present contribution using non-resonant hard x-ray scattering and resonant soft magnetic x-ray scattering, we focus on the crystallinity of the MgO and CoFeB layers and on the chemical and magnetic profiles within CoFeB/MgO multilayers. In particular, we are interested in the development of the interfaces with increasing annealing temperature. We have chosen a multilayer with five periods, which is a compromise of keeping the optimized growth properties of real MTJ on the one hand, but avoiding the complex layer sequence, on the other hand, in order to make a quantitative x-ray structural analysis feasible. The multilayer periodicity is on the order 5 nm with a MgO tunnel barrier thickness of nominally 2 nm. This MgO layer thickness corresponds to the thickness that is generally used in MTJs.

II. SAMPLE PREPARATION

To study the effect of annealing on the interface structure and the recrystallization of CoFeB and MgO layers, we have investigated CoFeB/MgO multilayers by hard non-resonant x-ray reflectivity and diffraction (XRR and XRD) and by soft x-ray magnetic scattering (XRMS). Multilayers with five periods were sputter deposited on a thermally oxidized Si(001)/SiO substrate and capped with Ru layer to avoid oxidation, using a Canon ANELVA C-7100 magnetron sputtering deposition machine. The multilayers are designated as: Si/SiO(001 nm)/$[$CoFeB(3 nm)/MgO(2 nm)$]_5$/Ru(8 nm), where the numbers in round brackets give the nominal layer thicknesses and the subscript indicates the number of repeats. After sputtering, the wafer was cut in several pieces, one remained as deposited, the others were annealed under high vacuum of 4×10^{-6} mbar for one hour at different temperatures T_A=240, 270, 300, 330, and 360 °C for each sample and in the presence of a magnetic field of 1 T, yielding a total of six samples. Our experiments have shown that up to an annealing temperature of 240 °C no changes can be recognized. Although all samples have been investigated in detail, in the following we show only a few examples, representative for the whole sample set.

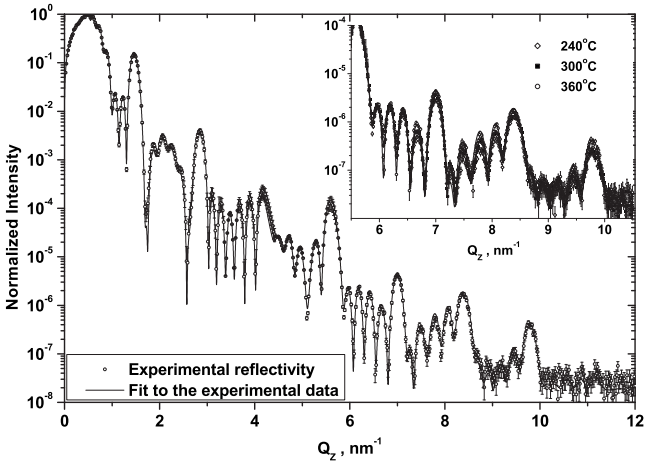

FIG. 1. X-ray reflectivity of a sample Si/SiO(001 nm)/$[$CoFeB(3 nm)/MgO(2 nm)$]$5/Ru(8 nm) annealed at the highest temperature T_A=360 °C. The solid line is a least square fit to the model. The inset shows the development of the x-ray reflectivity for different annealing temperatures. Only the high Q_z part is shown as this is more sensitive to small changes in the interface.

III. SMALL ANGLE HARD X-RAY REFLECTIVITY

For the structural analysis, we used the synchrotron source DELTA at the University Dortmund, and the Hasylab at DESY in Hamburg, both situated in Germany. Non-resonant x-ray longitudinal scans in the small angle regime for the investigation of the multilayer structure reflectivity (XRR) as well as high angle scattering for the investigation of the CoFeB and MgO sublattice recrystallization were carried out (XRD). All measurements were performed at room temperature at the wavelengths λ=0.1541 and 0.1127 nm. In Fig. 1, we show XRR results [intensity versus the scattering vector $Q_z=(4\pi/\lambda)\sin\theta$, θ=glancing angle to the substrate] together with the best fit to the experimental data for the multilayer annealed at T_A=360 °C.

We observe multilayer Bragg peaks up to the seventh order, revealing very flat interfaces and low roughness. The hump between the first and the second order Bragg peak is due to the presence of the Ru cap layer. Furthermore, between any two Bragg peaks thin film thickness oscillations (Kiessig fringes) can be recognized. From a qualitative analysis of the experimental data we find that the effect of annealing is not highly pronounced in the low angle XRR part, but is slightly more visible at high scattering vectors Q_z as shown in the inset to Fig. 1, where the intensity of the Bragg peaks is seen to increase and the width to decrease with increasing annealing temperature. Least square fit to the model provided the experimental thicknesses t_{CoFeB} and t_{MgO} for the CoFeB and MgO layers, respectively, for each measured sample and their respective mean square roughness parameters σ. All fit parameters are listed in Table I. The roughness parameters are on the level of 0.1–0.3 nm, for both layers, indicative of very smooth interfaces. There are some but not dramatic changes in layer thickness and in the

TABLE I. Structural parameters of CoFeB/MgO multilayers with five periods as derived from hard and soft x-ray fits to the reflectivity measurements. t is the layer thickness and σ the root mean square interface roughness parameter.

	MgO		CoFeB	
Annealing temperature	t (nm)	σ (nm)	t (nm)	σ (nm)
240 (soft)	1.640 ± 0.18	0.10 ± 0.05	2.820 ± 0.16	0.23 ± 0.12
240 (hard)	1.682 ± 0.01	0.23 ± 0.01	2.770 ± 0.03	0.26 ± 0.01
360 (soft)	1.490 ± 0.16	0.30 ± 0.06	2.990 ± 0.11	0.27 ± 0.07
360 (hard)	1.662 ± 0.01	0.22 ± 0.01	2.785 ± 0.01	0.24 ± 0.01

layer density. Furthermore, the very high quality of the fit to the data points could only be achieved by the assumption of a very small gradient in layer density from bottom to top, while the bilayer thickness over the same range remains essentially constant (details of the fit and parameters will be reported elsewhere).

IV. HIGH ANGLE HARD X-RAY DIFFRACTION

XRR is not sensitive to the crystalline structure on an atomic scale. Therefore, we have investigated the development of the crystallinity with annealing temperature by means of high angle XRD. The results are shown in Fig. 2. The peak at $Q_z = 45\ \text{nm}^{-1}$ is the Si(004) from the substrate. We observe at $Q_z = 28.5\ \text{nm}^{-1}$ a small and broad peak due to MgO(002). Another peak is visible at $Q_z = 38.6\ \text{nm}^{-1}$, which can be attributed to the bcc-CoFeB(002) peak, assuming that the boron content does not change the lattice parameter. From the FWHM, we derive an out-of-plane structural coherence length of about 10 nm, which hardly changes with increasing annealing temperature. The inset shows an enlarged portion of the scan in the area of interest and on a linear scale. The intensity of the CoFeB(002) and MgO(002) peaks increase upon annealing, indicative for crystal growth and/or crystal alignment. However, for a complete and textured recrystallization of the layers, we would have expected

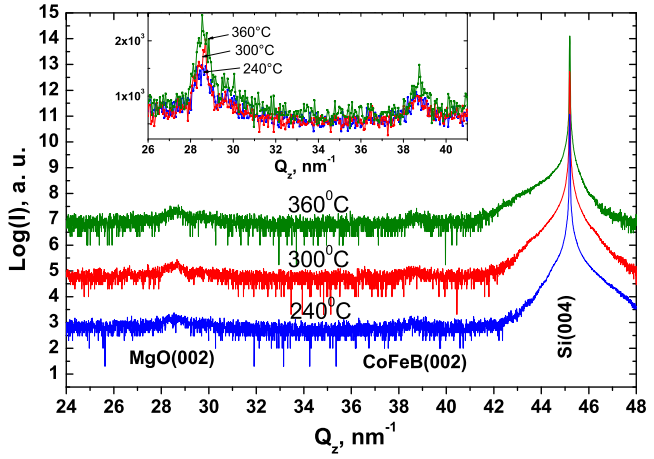

FIG. 2. (Color online) High angle x-ray scattering of the sample series Si/SiO(100 nm)/[CoFeB(3 nm)/MgO(2 nm)]$_5$/Ru(8 nm) annealed at different indicated temperatures. The scans are off-set by a constant factor for clarity. The inset shows the Q-range of the MgO(002) and CoFeB(002) peak on a linear scale for the same three annealing temperatures.

to observe Bragg peaks from the individual CoFeB and MgO layers surrounded by Laue oscillations corresponding to their respective layer thicknesses, as observed for epitaxial Fe layers on MgO by Tusche *et al.*[18] and for Fe/MgO multilayers by Raanaei *et al.*[19] The layers may be crystalline on a very small scale, but they lack longer range and textured crystallinity even after annealing up to 360 °C. In fact further x-ray studies have shown that the Bragg peaks observed are due to randomly oriented polycrystalline MgO. This is surprising and may be due to the residual boron content in the sample, which suppresses the long range recrystallization process. In a recent study by Vadalá *et al.*[15] of CoFeB/MgO multilayers using different deposition and annealing methods a more pronounced MgO(002) peak was observed. Also You *et al.*[20] report about some recrystallization in CoFeB/MgO(001) bilayers after annealing at 360 °C for 1 h, albeit for a much thicker MgO layer of 20 nm instead of 1.6 nm in the present case.

From the present XRD study, we cannot make conclusions about any further oxide formation either of the metals or of boron at the interface. In the diffraction pattern oxides show up only if they are crystalline. Furthermore, any additional oxide layer is anticipated to be very thin. In Q_z scans normal to the interfaces Bragg peaks from oxide layers should be very broad and difficult to distinguish from the background. Only in surface scattering mode with a glancing angle of the incident x-rays close to the total reflection angle one might be able to find indications of oxide formation. Furthermore, it is very difficult to keep track of the boron content, if boron is dissolved on interstitial sites. In this case, boron should cause some lattice expansion of the CoFe lattice. But this is a contradiction in itself. Either boron is present and CoFe is in an amorphous state, where boron cannot be detected by XRD methods, or CoFe is crystalline, in which case boron should have escaped the crystal lattice leaving an unstrained lattice.

V. SOFT X-RAY RESONANT MAGNETIC SCATTERING

XRMS measurements were performed at the undulator beamline UE56 of the synchrotron source BESSY II, using the ALICE chamber for soft resonant magnetic scattering.[21] Scans were taken tuning the photon energy before, at, and after the Fe and Co L_2 and L_3 resonance edges, respectively. The XRMS scans were simulated with the REFTOOL program,[22] in which the reflectivity intensities are calculated following the universal approach to magneto-optics proposed by Zak *et al.*,[23] where multilayers with arbitrary magnetization direction M are considered. The refractive index is commonly split into real and imaginary parts according to

$$n_{\pm} = 1 - \delta_{\pm} + i\beta_{\pm} = 1 - (\delta \pm \delta_m) + i(\beta \pm \beta_m),$$

where δ and β are the dispersive and absorptive contributions, respectively, and δ_m and β_m are the corresponding magnetic contributions to the refractive index, and + and − stand for right and left circularly polarized light.

Figures 3 shows the measured and the calculated charge scattering intensities $(I_+ + I_-)/2$ in the upper panels and the measured and the calculated asymmetry ratios $(I_+ - I_-)/(I_+$

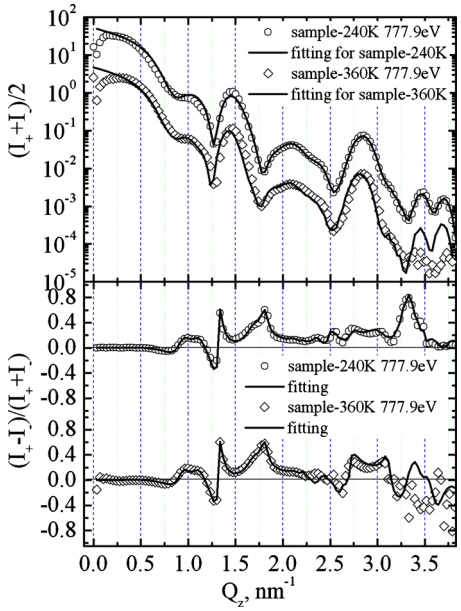

FIG. 3. (Color online) The measured and the calculated charge scattering intensities $(I_+ + I_-)/2$ (upper panel) and the asymmetry $(I_+ - I_-)/(I_+ + I_-)$ (lower panel) of the CoFeB/MgO multilayer sample measured at a photon energy of 777.9 eV and annealed (circles) at 240 °C and (diamonds) at 360 °C. The scans in the upper panel are off-set by constant factor for clarity.

$+I_-$) in the lower panels for multilayers annealed at 240 °C and 360 °C, respectively, and for a photon energy of 777.9 eV, which is at the L_3 resonance edge of Co. We first discuss the charge scattering. From the fitting (solid line) of the reflectivity curves, the experimental thickness and roughness parameters for the CoFeB/MgO multilayers for both samples are derived. Those parameters are listed in Table I and are in good agreement with the hard x-ray results, although the quality of the fit is not quite as high. Based on the fitting results, the roughness of the CoFeB appears to slightly increase with increasing annealing temperature.

The magnetic contribution to the resonant scattering can be obtained from the asymmetry ratio $(I_+ - I_-)/(I_+ + I_-)$. The overall shape of the asymmetry as a function of scattering angle does not change dramatically between the annealing temperatures of 240 and 360 °C, only the values at the Bragg peaks and at the valleys are different, most prominent at $Q_z = 3.25$ nm^{-1}. These changes are due to the CoFeB layers, which are close to the cap layer and to the substrate. Similar measurements at the Fe L_3-edge were also done (not shown here) confirming the results from the Co L-edge. The fitting of the asymmetry provides the magnetization profile of the multilayer via the depth dependent imaginary part of the refractive index. The magnetization profile of Co in CoFeB/MgO multilayer annealed at 240 and 360 °C are shown in Fig. 4. The magnetization amplitudes close to the cap layer and to the substrate are reduced compared to those inside. However, the reduction becomes less pronounced and the overall amplitude increases slightly during annealing at 360 °C. It should also be mentioned that the 240 °C sample is representative for the situation after deposition at room temperature, as we have not observed any changes between these temperatures.

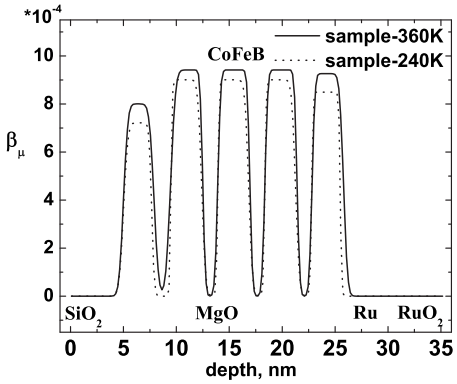

FIG. 4. Depth-dependent magnetic imaginary part of the refractive index of the CoFeB/MgO multilayer sample annealed at 240 and 360 °C.

VI. DISCUSSION AND CONCLUSION

In the present study, we have used non-resonant hard x-rays and circularly polarized resonant soft x-rays for the investigation of the chemical and magnetic profile of CoFeB/MgO multilayers annealed between 240 and 360 °C. This is the temperature range where upon annealing a dramatic increase in TMR values are usually observed. The multilayers are similar to CoFeB/MgO/CoFeB trilayers used in MTJs. In particular, the CoFeB and MgO layer thicknesses are comparable. However, the main difference to real MTJs are Ta top and bottom layers, which are used as diffusion barriers and getters. We found that the layer structure of the multilayer has very high quality immediately after growth and does not change much upon annealing, aside from a slight increase in the interface roughness after annealing at 360 °C. At high diffraction angles, we observe some onset of recrystallization of the CoFe and MgO layers, which is, however, not dramatic. The observed Bragg peaks indicate a polycrystalline nature of the MgO layer. This may be due to the fact that the MgO layers were actually slightly thinner than nominally used in MTJs. Below a thickness of 1.5 nm, MgO in proximity to an amorphous CoFeB layer is suspected to be also in an amorphous state. Lacking the nanocrystallinity, MgO may no longer serve as a seed for its own crystal growth nor as a seed for the crystallization of the CoFeB layer.

The magnetization profile as derived from the asymmetry of the x-ray magnetic scattering shows some reduction at the top and bottom layer and is higher inside of the multilayer. Such a behavior is often observed in complex multilayers, as the top and bottom layers have different contacts to the substrate and the capping layers, whereas the interior is representative for the intrinsic properties.[24–27] In fact, they may experience a slightly different interdiffusion, oxidation, and roughness affecting the magnetization profile. Upon annealing the amplitude of the magnetization profile increases, which may be the result of some diffusion of boron out of the CoFeB layers at higher temperatures or due to the reduction in metal oxides at the interfaces to MgO back to the metallic state, as suggested by Hindmarch *et al.*[16] Again the annealing effect is more pronounced in the outer layers as compared to the inner ones.

We believe that in our CoFeB/MgO multilayers the bo-

ron content in the magnetic layer still plays an important role even after annealing at the highest temperature, effectively hindering a more pronounced recrystallization. We speculate that in real MTJs, Ta seed and cap layers serve at the same time as getter for the boron content,[2] which were absent in the present samples. A second potential problem is the thickness of the MgO layers, which when too thin may not develop the required nanocrystalline seeds. Perhaps the MgO layers and subsequently the CoFeB layers would recrystallize after prolonged tempering at 360 °C or at even higher annealing temperatures before interdiffusion destroys the multilayer.

VII. SUMMARY

In summary, we have studied the structural and magnetic properties of CoFeB/MgO multilayers, which have nominally the same layer thicknesses as in real MTJs. The multilayers were annealed in steps up to 360 °C for one hour in a field of 1 T. Reflectivity measurements with hard and soft x-rays reveal very sharp chemical and magnetic interfaces. These interfaces improve only little upon annealing up to the highest temperature. Surprisingly, the crystallinity of the CoFeB and MgO layers remains poor even after annealing at the highest temperature. Several reasons are discussed which might impede a more pronounced recrystallization, including the boron content in the magnetic layers and the missing nanocrystalline seeds in the MgO layer.

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