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TEM Characterization of a Magnetic Tunnel Junction

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Abstract. TEM characterization of a CoFeB/MgO/CoFeB magnetic tunnel junction (MTJ) has been performed. A TEM cross section was prepared using focused ion beam (FIB) milling techniques. However, the high energy Ga⁺ beam causes sample damage and induces the redeposition of the sputtered materials on the section surface. Complementary investigation of the crystal structure of the active trilayer in the MTJ was performed by depositing films directly onto a TEM holey carbon film. The TEM imaging, selected area electron diffraction (SAED) and energy dispersive X-ray (EDX) analysis were employed to study the nanostructure. The MgO layer is found to be incompletely crystalline with randomly oriented MgO crystallites. The CoFeB layer is amorphous and is homogenously deposited.

1. Introduction

Nanostructures and interfaces are crucial for spin dependent tunnelling (SDT) or tunnelling magnetoresistance (TMR) in magnetic tunnel junctions (MTJ); a multilayer structure consisting mainly of two ferromagnetic layers separated by an insulating layer. A MTJ with a large TMR value would be a significant candidate for magnetic random access memory (MRAM) applications and the next generation magnetic sensors [1-2]. Lattice matching between the (001) MgO tunnel barrier and the (001) bcc ferromagnetic electrodes (Fe, Co or CoFe) has been predicted to give a very high TMR percent [3-5]. A TMR of 500% at room temperature and 1010% at 5 K has been reported in the CoFeB/MgO/CoFeB pseudo-spinvalve tunnel junction [6]. Apart from a crystalline MgO layer oriented with the {001} planes parallel to the junction, the crystallization of the CoFeB is considered to be another factor responsible for a high TMR value. The crystallization of this layer depends not only on the initial crystallinity of the neighbouring MgO but also on its own composition and thickness [6]. Therefore, in the multilayer MTJ structural nano-characterization is essential.

Imaging the atomic lattice with TEM is a powerful technique to study crystalline nanostructures in materials. However, to get a good lattice image, the sample must be very thin, approximately less than 50 nm. A high quality TEM sample can be achieved from a conventional sample preparation route; grinding, dimpling and milling, which is a time consuming process. Recently focused ion beam (FIB) milling has been used for rapid cross-sectioning and TEM sample preparation [7]. In this paper, we present the TEM characterization of MTJs with a MgO insulating barrier deposited by a combination of dc and rf magnetron sputtering. A TEM cross-section of the as-deposited MTJ was prepared by FIB and was subsequently used to investigate the crystal structure and interfaces of the active layer of the MTJ.
2. Experiment

The films were deposited on a Si/SiO₂ substrate by rf magnetron sputtering with a base pressure in the range of 2x10⁻⁸ Torr. The MTJ device contains the following layer order: Ta[3-4]/Cu[20]/Ta[3-4]/NiFe[1]/IrMn[7-8]/CoFeB[4-5]/MgO[7.5-10]/CoFeB[4-5]/Ta[3-4]/Cu[20]/Ta[3-4], the numbers in parenthesis are the layer thicknesses in nanometres estimated by X-ray reflectivity measurement. The MgO layer was formed using a MgO target at a pressure of 6 mTorr and the other layers were deposited at a pressure of 2.7 mTorr in Ar atmosphere. The details of the film fabrication have been described in [8]. A thick MgO layer was deposited in this test structure to observe its crystal structure and as a first step to developing the technique to characterize the thin MgO layer (a few nanometres) present in typical MTJ devices.

TEM cross-section preparation using Dual beam FIB with in-situ lift-out

A TEM cross section was prepared by milling an electron-transparent membrane using a dual beam FIB (FEI novalab 600). The 30keV Ga⁺ ion beam is used to cut a section through the bulk structure of the MTJ which is pre-coated with a 5 nm Pt conductive layer using conventional sputtering. At the start of the cutting process, a 1 μm thick Pt strip was deposited in the FIB in order to protect sample surface from ion beam damage. The cross-section of 4x12x0.4 μm in depth, length and thickness, respectively was then lifted out using a manipulator and attached to a copper TEM half grid by local Pt welding. Finally, the membrane was further thinned using a lower beam current (0.1-0.3 nA) until the final thickness is as thin as possible without any apparent damage. The final thickness of this cross-section is approximately 90 nm. The cross-section was then taken to be analyzed with a Philips CM200-FEG field emission gun TEM.

3. Result and discussion

Bright Field TEM images of the prepared FIB cross-section of the MTJ show the relatively clear and uniform interfaces of the electrode and Ta layers but the active trilayer’s interfaces cannot be clearly observed (Figure 1).

Figure 1. Bright field TEM micrograph of the FIB cross-section, the inset is a high magnification image of the MgO barrier region showing some lattice fringes.

Figure 2. Scale thicknesses schematic diagram of the expected cross-section of the device based on measurements from X-ray reflectivity.

The layer thicknesses are broadly consistent with those expected from the X-ray reflectivity measurement (schematic shown in Figure 2). The Cu and Ta seed layers can be clearly recognized because of their contrast and thicknesses. The regions with the very thin layers; CoFeB/MgO/CoFeB
in the middle of junction and the pinning layer IrMn and NiFe, can be identified but each individual layer can not be distinguished. High magnification images of the MgO layer show intermittent lattice fringes. In addition, there is a large amount of material deposited as surface nanoparticles which is evidently seen on the entire surface of the section. EDX analysis suggests that these particles could be Pt and/or Cu which are the only materials found over the whole section. Their lattice spacings are more likely to correspond with those of Cu. This section is too thick to get the detailed crystal structure of the active junction from the high magnification TEM image. The active layers are only a few nanometers wide making it difficult to do elemental analysis of the position of an individual layer (because of the large interaction volume). Therefore, we also decided to perform a plan view characterization of the active tunnel junction layer after it had been deposited directly onto a holey carbon TEM support grid.

An active trilayer of CoFeB[4-5]/MgO[15-20]/CoFeB[4-5] was directly deposited onto a holey carbon TEM support grid using the same sputtering system. In addition, a single layer of CoFeB[30] was also prepared onto a separate TEM support grid in order to observe its crystalline structure and its relationship to the CoFeB/MgO/CoFeB trilayer.

Imaging, EDX and EFTEM (the latter two not shown here) suggest that each layer of CoFeB and MgO is continuous across the support film. The as-deposited single CoFeB layer is amorphous (confirmed by its diffuse ring SAED pattern in Figure 3 inset). The MgO layer (Figure 4) consists of isolated crystallites with a mean grain size of 5 nm. SAED patterns confirm that the crystalline regions are the MgO phase lying in random orientations (figure 5b) similar to the case of smoke deposited MgO cubic crystals (Figure 5a). High magnification imaging of individual crystallites reveals that not all of them are lying with their [001] normal to the growth direction (e.g. the crystallite lying [01\bar{1}] seen in Figure 6).

Additionally, we have also found that the high energy electron beam causes a change in the crystal structure of the MgO, causing both crystallization and crystal growth. For this reason, it is
worth studying the effects of the post-annealing process on the crystallization of the MTJ layers, which is thought to improve their TMR magnitude.

Figure 5. SAED Pattern from a 180 nm diameter region of a) the MgO smoke cubes b) the CoFeB/MgO/CoFeB film on a TEM support grid. c) The relative intensity profile of a ring patterns a) and b), and d) the X-ray stick pattern of randomly oriented polycrystalline MgO. The smoke ring pattern (a) can be indexed to MgO crystallites in random orientation. All the expected MgO reflections are visible in the trilayer (b) and to a first approximation the MgO is in random orientation (cf. c) and d)).

Figure 6. High magnification TEM image of an individual crystallite in the active trilayer lying with a zone axis parallel to the electron beam. The orientation of the lattice planes can be readily seen in the inset FFT and are indexed to a MgO crystal lying with its $[0\bar{1}\bar{1}]$ zone axis.

4. Conclusion

A TEM cross section of a MTJ prepared by FIB has been contaminated by the redeposition of Pt and Cu making it difficult to characterize. However, a simple planar sample characterization has been successfully performed on the active trilayer deposited on a TEM support grid. The films of CoFeB and MgO are uniformly deposited across the support film. The MgO is partially crystalline with a random orientation and not in a $\{100\}$ texture. These planar views of the active layer show a promising approach to the characterization and development of understanding of the crystallization of the active layers in an MTJ. Such knowledge will be used to improve the fabrication process and device performance.

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