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Influence of ion assisted deposition on interface broadening in Fe/Al multilayers investigated by medium energy ion scattering

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ABSTRACT

Trilayers of Al/Fe/Al and Al/Fe multilayers produced by magnetron sputtering both with and without ion assistance have been depth profiled using Auger electron spectroscopy and medium energy ion scattering. Important differences are observed in the layer structure, with ion assisted deposition giving the narrowest Al/Fe interfaces and so maintaining the most clearly defined layer structure. Both types of sputtering result in some oxygen contamination, particularly at the surface that modeling shows to be associated with the Al layers.

1. Introduction

Multilayers of Fe and Al have been the subject of several investigations motivated both by the interest in their soft magnetic properties, including low coercivity and high saturation magnetisation [1] and by their use as an example of solid-state reaction [2]. Multilayers with bilayer periods in the range of 3–60 nm have been deposited by electron beam evaporation [2–4], RF sputter deposition [1,5], DC sputter deposition [6] and pulsed laser deposition [7]. Aside from the magnetic properties, the main focus has been on the degree of intermetallic compound formation at room temperature and following a post-deposition anneal. The consensus is that even at room temperature a broad interface forms by reaction between Fe and Al that is of the order of 1–2 nm wide [3,7]. Upon annealing to sufficiently high temperature, the film converts to a solid-state compound [3,4]. The motivation for the study reported in this paper is to discover if the use of ion assisted deposition, which results in the production of denser, smoother layers, can reduce the width of the interface between the Fe and Al layers.

Magnetron sputter deposition is one of the most common methods for the deposition of metallic multilayers [8]. Two important features of sputter deposition are the higher energy of arriving species compared with evaporation and the back reflection of Ar neutrals from the target onto the growing film [9]. These neutral atoms are produced by neutralisation of energetic ions in the cathode dark space and upon back reflection from more massive target atoms impinge upon the growing film. The combined effect of these features tends to produce denser, smoother films. When sputter depositing a light element such as Al, however, there will be little back-reflected flux of Ar neutrals as the mass of Ar exceeds that of Al. This may be replaced by using a variant of ion assisted deposition. Rather than using a separate ion source, an unbalanced magnetron can provide the flux necessary to smoothen and densify the film. In an unbalanced magnetron, the flux from the outer ring of magnets does not equal that from the inner ring causing flux to leak away to the substrate. This flux draws plasma to the growing film surface where it may be accelerated by applying a negative bias to the substrate. This impinging ion flux has a strong effect on the film microstructure and could suppress the development of roughness in multilayers and may also suppress interdiffusion by creating denser films.

A previous investigation of the influence of bias sputtering on Fe/Al multilayers used grazing incidence reflectivity primarily to determine the average roughness of the interfaces [10]. It was found that the use of bias sputtering reduced the roughness of the interfaces and produced samples that gave much clearer Bragg

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peaks from the superlattice period. A comparison of the curves from bias and non-bias sputtered multilayers would indicate a much better bilayer structure in the bias sputtered case, but the reciprocal space nature of X-ray analysis makes the formation of a clear cut picture of the structures a non-trivial exercise. A simple depth profile through the films would give a clearer picture, but the narrowness of the layers makes this difficult by traditional means. Depth profiling by Auger electron spectroscopy (AES) and ion erosion is often limited in depth resolution to a few nm [11] by the electron escape depth and crater roughness. Whilst secondary ion mass spectrometry can do better than this, it is difficult to quantify.

Medium energy ion scattering spectroscopy (MEIS) [12,13] is a variant of Rutherford backscattering spectroscopy (RBS) [14,15] that gives greater surface sensitivity and greater depth resolution whilst retaining the quantifiable nature. It utilises typically He^+ or H^+ ions with energy of the order of 100 keV rather than the MeV energies found in RBS. MEIS can give elemental analysis of surfaces and thin films with sub-nanometre resolution [13,16–20].

In this paper we report a study on the use of MEIS to analyse Al/Fe multilayers and trilayers that have been produced by sputtering from an unbalanced magnetron both with and without substrate bias. We find that structures deposited without plasma assistance show a broad interface between the Fe and Al layers, causing the layer-by-layer structure to largely break down at the top of a multilayer stack. The use of bias sputtering is shown to reduce the interface width and to help maintain the multilayer integrity. In addition, the utility of MEIS for the surface analysis of these nanometre scale structures (see also Ref. [21]) is further demonstrated.

2. Experimental

Two types of samples were deposited: Al/Fe/Al trilayers and Fe/Al multilayers. In each case, they were deposited onto Si substrates with a native oxide both without ion assistance by grounding the substrate and with ion assistance by biasing it at −200 V. The deposition was carried out utilising DC planar magnetron sputtering in an Ar^+ atmosphere (99.995% pure source gas) in a modified Nordiko system that has been described elsewhere [22,23]. The samples were transferred outside of vacuum to separate systems for AES and MEIS analyses as detailed below.

The trilayers and multilayers were deposited sequentially onto Si(111) wafers of 0.525 mm thickness that had been cleaved to squares of size of 2 cm × 2 cm. Al and Fe targets of 5 cm diameter were used, the Al target being 99.95% pure and 2.0 mm thick and the Fe target being 99.95% pure and only 0.25 mm thick (to reduce magnetic shorting of the magnetron). The Al target was mounted into an unbalanced magnetron so that some of the argon plasma could leak away and impinge upon the substrate during growth. The Fe target was mounted into a balanced magnetron as Fe is sufficiently heavy to back-reflect Ar neutral species onto the growing film. The base pressure of the deposition system was 1×10^{-7} mbar and a sputtering gas pressure of 2.6×10^{-3} mbar was used with a target power of 70 W. This gave approximate deposition rates of 0.33 nm s^{-1} for Al and 0.15 nm s^{-1} for Fe, which is comparable with those used in other reports [2,4]. The target to substrate distance was 10 cm with the two magnetrons angled towards the centre of the substrate. The Al and Fe layers were alternately sputtered onto the substrate using time-controlled, pneumatically actuated shutters on the magnetron sources. The deposition rates had previously been calibrated using X-ray reflectivity measurements and nominal structures that were deposited were Si(111)/$SiO_{x(native)}$/Al(4.0 nm)/Fe(3.7 nm)/Al(4.0 nm) for the trilayers and Si(111)/$SiO_{x(native)}$/[Fe(3.7 nm)/Al(4.0 nm)]$_{19}$/Fe(1.0 nm) for the multilayers.

The multilayer films were transferred under normal atmosphere conditions for analysis by AES using a JEOL Jamp 7100 system. The samples were analysed using a primary electron energy of 3 keV, a current of 0.7 μA and a spot-size of 100 μm diameter. The transitions used for analysis were the Fe 703 eV (LMM), Al 68 eV (LVV) and Si 92 eV (LVV). Depth profiling was carried out using Ar^+ bombardment.

The trilayer and multilayer samples were transferred under normal atmosphere conditions to the UK National MEIS Facility at Daresbury Laboratory [24]. This facility has an ultra-high vacuum end station with a load-lock for sample introduction. He^+ ions with a nominal energy of 100 keV were directed at the target at an incidence angle of 45° to the surface normal and were detected at a scattering angle of 90°.

3. Results and discussion

From the AES analysis, it was found that the main contaminants were a surface C peak, some surface O and O distributed throughout the multilayer at a level of about 10% atomic fraction. The thickness of the surface oxide was about 8 nm (which would encompass much of the first bilayer). The presence of the bulk oxide is not unexpected for the deposition of Al by sputtering in high vacuum, and has been reported by other authors [1]. It is difficult to deposit pure Al films by this method due to its reactivity, but since the main interest in Fe/Al films is in the magnetic properties of the Fe layers [25] some contamination of the Al layers is tolerable.

The Auger depth profile of the two multilayer samples is shown in Fig. 1. The near surface region (small etch time) is shown with an expanded scale as in this region there is reasonable depth resolution. With increasing etch time the resolution degrades due to statistical effects in the sputtering. From the graph it can be seen that the surface is oxidised to a greater extent as would be expected. The first Fe layer can be identified in the multilayer grown with bias, but not in the one grown without bias.

The results of the MEIS analyses of the trilayer and multilayer samples are shown in Figs. 2 and 3 respectively. When viewing these spectra it is important to bear in mind the mechanism of MEIS depth profiling. The scattering event is a binary collision

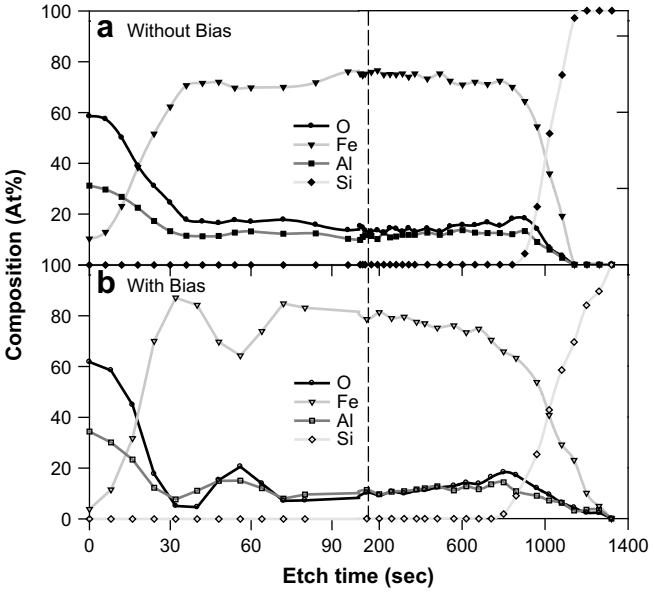

Fig. 1. Auger electron spectroscopy depth profile of multilayer samples grown: (a) without a substrate bias and (b) with a substrate bias. Note the break point in the etch time axis show an expanded depth scale near the surface.