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# SCANNING AUGER ELECTRON SPECTROSCOPIC INVESTIGATIONS OF MAGNETOSTRICTIVE NANOSCALE MULTILAYERS

EBERHARD NOLD[a], CHRISTEL ADELHELM[a], ALFRED LUDWIG[b], and SHEN DIAN-HONG(沈电洪)[c]
[a] *Forschungszentrum Karlsruhe GmbH,Institut für Materialforschung, 1, Postfach,* 3640, D-76021 *Karlsruhe, FRG*
[b] *Center of Advanced European Studies and Research, Friedensplatz* 16, D-53111 *Bonn, FRG*
[c] *Institute of Physics, Chinese Academy of Sciences, Beiing* 100080, *China*

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The characterisation of thin magnetostrictive multilayers in the nanometerscale range [(4,5nm$Tb_{40}Fe_{60}$/ 9nmFe)×100] demands a surface sensitive analytical technique. A suitable technique is scanning Auger Electron Spectroscopy (AES) to determine the elemental composition, the thickness, the presence of interdiffusion between thin layers, or the presence of contamination at interfaces.Auger sputter depth profiles are obtained by using Xe-Ion bombardment to etch the sample.Sample rotation during sputtering produces shallow craters, minimises roughening and enhances depth resolution. In addition, Auger maps of the craters are used to reveal the separated magnetostrictive $Tb_{40}Fe_{60}$ layers and soft magnetic Fe layers.

**Keywords:** Auger electron spectroscopy ,sputter depth profiles, magnetostrictive multilayer films
**PACC:** 8280, 6865, 8165C

## I. INTRODUCTION

The development of magnetostrictive films which exhibit high magnetostriction combined with soft magnetic properties was driven by the need for suitable microactuator materials.[1−3] Sputter-deposited magnetostrictive thin films are suitable for the realisation of microactuators like, e.g., microfluidic devices, micromotors, laser scanner mirrors etc. In previous investigations it was found that ($Tb_{40}Fe_{60}$/Fe-Co) multilayers represent promising thin film materials with respect to their unique combination of soft magnetic and giant magnetostrictive properties in comparison to single layer films.[3]

The thermal stability of magnetostrictive films is an important feature essential for the function of microactuators in real world environment. Therefore the films were annealed up to 480°C in order to investigate at which temperature the multilayered structure of the films is destroyed. Furthermore, the magnetic and the magnetostrictive properties depend also strongly on the chemical state of the layers, which can be altered by heat treatments.

The characterisation of thin films in the nanometer range with respect to the elemental composition is performed using an analytical technique with low information depth. A suitable analytical technique is Auger Electron Spectroscopy (AES).The low escape depth of the Auger electrons gives this technique its surface sensitivity.[4]

Scanning the electron beam across the sample surface generates Secondary (SE) and Auger (AE) electrons. SE images provide a topographic view by detecting low energy electrons. Auger maps, obtained by measuring the emitted Auger electron intensity reveal the lateral distribution of an element.

Additional information can be obtained by using nobel gas ions to sputter etch the sample. Such sputter etching erodes the sample in a controlled manner and is typically performed with Ar or Xe ions. A sputter depth profile, which plots the Auger signal as a function of sputter time, shows elemental concentrations as a function of sample depth.This technique is particularly effective for thin film analysis and for solving material problems which require identifying: the thickness of a layer, the composition of a thin film, the presence of interdiffusion between layers or the presence of contamination at an interface between layers.

To improve depth profiling, a technique was developed (known as Zalar Rotation™) where the sample is rotated during sputtering.[4] Rotation achieves more uniform etching, which minimises surface roughening, reduces cone formation and enhances interface resolution(Fig.1).

Shallow craters can be produced by scanning a defocused ion beam over a small area during sample rotation.[5] Therefore inclination angles of only a few degrees are obtained. As a result, layers of some

nanometer thickness are "expanded" to up to a micrometer width(Fig.2).

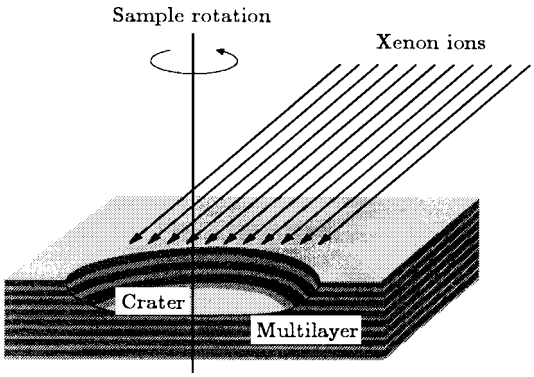



**Fig.1.** Schematic cross section of a Xe-sputter etched crater in a multilayer system. The sample is rotated during sputtering under inclined ion incidence.

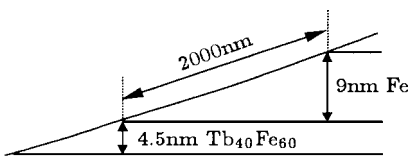



**Fig.2.** Schematic sketch showing the expansion of nanometer thick layers to microscale range on a shallow crater wall.

These shallow craters offer the additional possibility to investigate nanoscale layers by AES scanning equipment without very high lateral resolution. Therefore, Auger maps reveal visually thickness, composition, diffusion and contamination at interfaces of the nanometerscale layers in the crater. The crater created by this method in a Mo/Fe multilayer system[5] could be analysed by applying scanning force microscope.

## II. EXPERIMENTAL

The (4.5nm $Tb_{40}Fe_{60}$/9nm Fe)×100 multilayer film was magnetron sputtered onto a Si (100) substrate using a modified Leybold Z 550 device with alloy targets in a rotary turn table technique. The base pressure was $5\times10^{-5}$Pa and the Ar pressure 0.4Pa. Annealing of the multilayers was performed at 270°C or 480°C in a high vacuum furnace.

The total thickness was determined by a surface profiler. The single layer thicknesses were adjusted by the sputtering time using a known deposition rate. High resolution TEM micrographs verified the measured and calculated thickness. AES measurements were performed using a PHI 600 Scanning Auger Microprobe (SAM) system with a base pressure $< 1\times10^{-9}$mbar in the analysis chamber. For AES measurements the primary beam energy was set to $E_p$=5kV or 10kV, respectively. The primary beam current was held at $I_p$=500nA for profiles, $I_p$=100nA for element maps and $I_p$=0.1nA for SE images. The angle of incidence of the primary electron beam was 30° with respect to the sample surface. In order to minimise the influence of the electron radiation on the sample, the profiles were recorded in the defocused mode with a beam size of approximately 10$\mu$m. Secondary electron images (beam size <20nm) and Auger element images (beam size ≈400nm) were mapped in the scanning mode with a focused primary electron beam.

The energy resolution of the cylindrical mirror analyser was set to 0.6% for the kinetic energy of the Auger electrons. The following Auger transitions for Auger depth profiles and Auger element images were used: Tb=150eV, Fe=705eV, O=510eV and C=275eV.

Quantification was performed empirically by the use of similar standard layers with known composition. To etch the samples during rotation a differentially pumped duoplasmatron microbeam ion gun was used. During depth profiling craters were created by bombarding the sample with Xe ions under an angle of incidence of 45° with respect to the sample surface. The ion beam current approached 8nA with an ion energy of 3kV.

The ion beam was defocused to a beam size of approximately 200$\mu$m. As a result shallow craters were produced by scanning the relatively large defocused ion beam over a relatively small rectangular area of 500$\mu$m×500$\mu$m during sample rotation (1rpm). The inclination of the wall of the created crater was ≈ 1° with respect to the sample surface, corresponding to an expansion of >2000 (9nm Fe layer was expanded up to 20$\mu$m). A calibration of the sputter etch rates was not required because the thickness of the layers was known.

## III. RESULTS AND DISCUSSION

### A. Auger depth profiles

Auger depth profiles of a multilayer system with the uppermost 4.5nm $Tb_{40}Fe_{60}$ layers and 9nm Fe layers are presented in Fig.3(a) room temperature (b) after 270°C annealing temperature (c) after 480°C annealing temperature. The samples were etched by Xe

ions during sample rotation.The scaled atomic concentration of O KLL, Tb MNN and Fe LMM have been plotted as a function of sputter time. The sputter rate calculated is ≈4nm/min. It can be seen from Fig.3(a) that the time required for sputtering oxygen is very short, which indicates a normal adsorption layer on the surface. The quantitative profiles of Fe and Tb show a clear separation of the single layers with sharp interfaces. The composition of the single layers are close to stoichiometric $Tb_{40}Fe_{60}$ and Fe, respectively. The small increase of the interface width at the end of the profiles is influenced by Xe ion bombardment

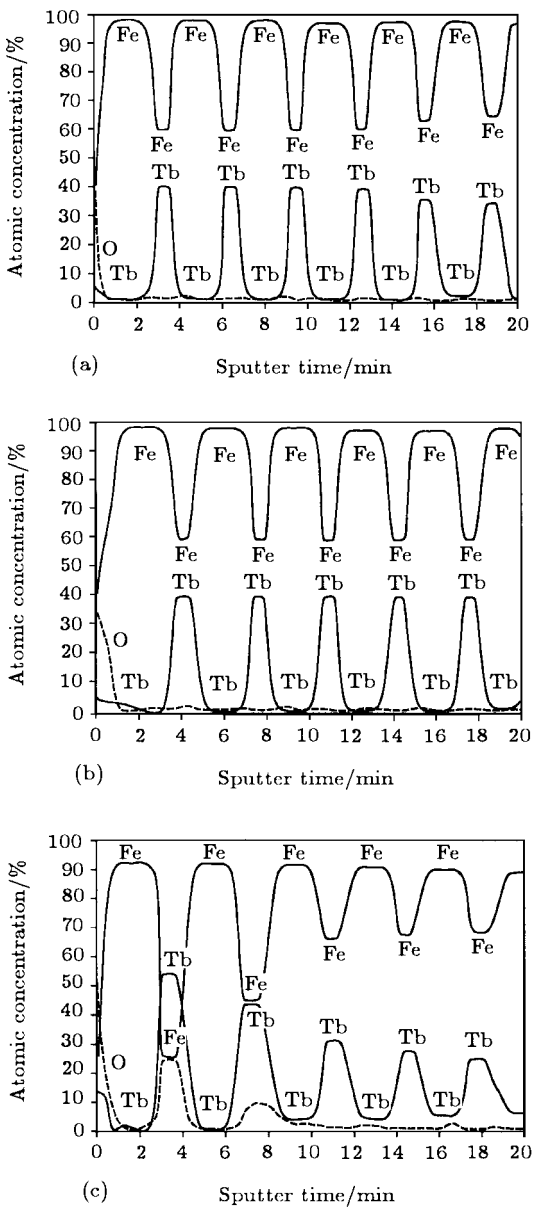



**Fig.3.** Quantitative Auger depth profiles (Xe bombardment) of a magnetostrictive 4.5nm $Tb_{40}Fe_{60}$/9nm Fe multilayer, obtained during sample rotation (a) room temperature (b) after annealing at 270°C (c) after annealing at 480°C.

and not by interdiffusion between the Fe layers and the TbFe layers. The Auger depth profiles of the sample annealed at 270°C show similar results (Fig.3(b)) as the room temperature sample. Only the O profile reveals a thicker oxygen layer of about 2nm at the surface which results from the uptake of O during annealing. The Auger depth profiles of the sample annealed at 480°C (Fig.3(c)) are significant different to the profiles of the samples annealed at 270°C and at room temperature, respectively. Two characteristic regions could be distinguished in the profiles. The first one comprises the uppermost four layers with an oxygen content in the TbFe layers. It can be seen that the oxygen coming from the residual gas diffuses through the top Fe layer into the first TbFe layer below and through the second Fe layer to the next TbFe layer while oxidising only the TbFe layers.The oxygen amount within the uppermost TbFe layer is higher than in the TbFe layer below and depends on the diffusion velocity of oxygen driven by the oxygen affinity to the rare earth element Tb in the TbFe layers. The oxidised TbFe layers act as a diffusion barrier. The second region below the uppermost four layers reveals interdiffusion between TbFe and Fe layers. Mainly Fe diffuses within the TbFe layers and changes the stoichometry dramatically up to about $Tb_{20}Fe_{80}$, accompanied by a Tb diffusion within the Fe layers. In this region, broader interfaces can be observed between the intermixed Fe layers and TbFe layers. Undoubtedly, the oxygen amount in this region is too low to form oxidised TbFe diffusion barriers which would prevent interactions. Hence, it follows that the TbFe/Fe multilayers are stable at least up to annealing temperatures of about 300°C, whereas annealing at 480°C leads to a degradation of the multilayer.

### B. Auger element images and secondary electron images

The Auger element images and the SE images measured in the shallow craters show the 4.5nm TbFe layers and 9nm Fe layers expanded to microscale ranges of about 10$\mu$m for the TbFe layers and 20$\mu$m for the Fe layers, respectively.Auger element images of Fe, Tb and O as well as the secondary electron image of the sample at room temperature are presented in Fig.4. It can be seen that in the crater the TbFe layers are clearly separated from the Fe layers. Oxygen is detected outside the crater and reveals a normal oxide layer on the surface. These results are in good agreement with the Auger depth profiling analysis of the

sample.

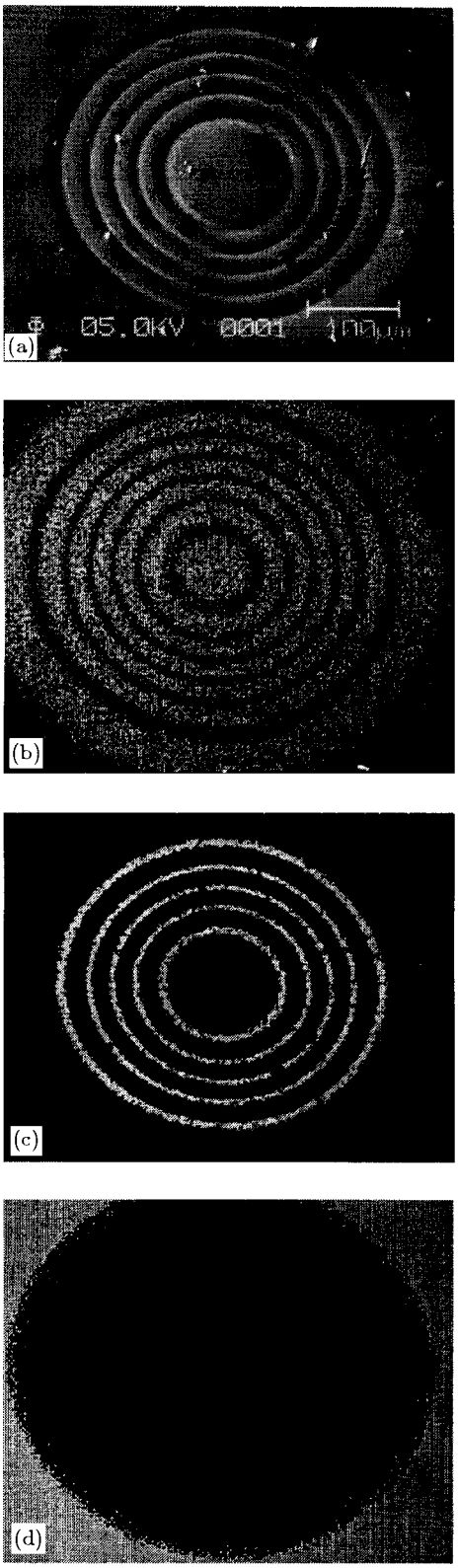



**Fig.4.** Images of a sputter crater produced by inclined Xe bombardment and sample rotation showing the uppermost 10 layers of 100 layers of a 4.5nm $Tb_{40}Fe_{60}$/9nm Fe multilayer system at room temperature (a) secondary electron image, (b) Fe Auger image (c) Tb Auger image and (d) O Auger image

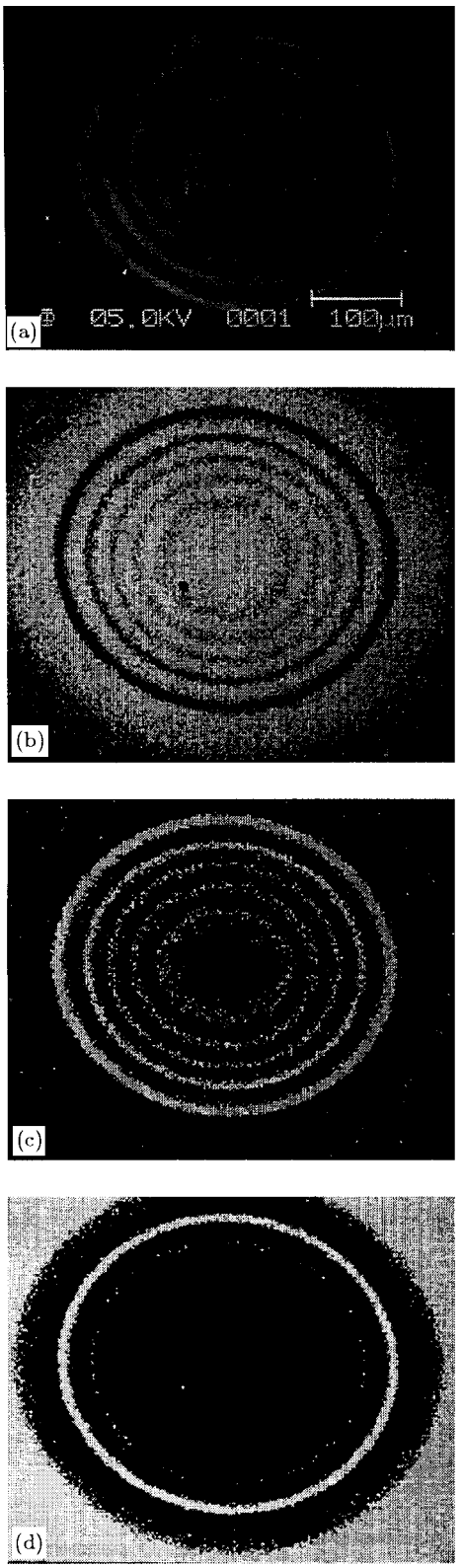



**Fig.5.** Images of a sputter crater produced by inclined Xe bombardment and sample rotation showing the uppermost 10 layers of 100 layers of a 4.5nm $Tb_{40}Fe_{60}$/9nm Fe multilayer system annealed at 480°C, (a) secondary electron image, (b) Fe Auger image, (c) Tb Auger image and (d) O Auger image

The Auger element images of Tb, Fe, O and also the secondary electron image of the sample annealed at 480°C are shown in Fig.5. The two characteristic regions can be distinguished also in the images on the crater wall. The upper region shows the separated layers without diffusion,which is prevented by the oxidised TbFe layers, while the lower region shows TbFe layer and Fe layer intermixing by diffusion. The Auger element images in these craters reveal the layer thickness, the presence of interdiffusion between the layers and the presence of contamination at interfaces and layers.

## IV. CONCLUSIONS

Scanning Auger Electron Spectroscopy is a useful method for the characterisation of multilayer systems. Sample rotation during ion sputtering improves depth resolution by minimising surface roughening. In the shallow craters produced by inclined ion bombardment during sample rotation the nanometerscale layers are expanded laterally to the microscale range. Auger investigations in shallow craters offer additional analytical possibilities to demonstrate the chemical compostion in nanometer thick layers.

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# References


[1] Quandt E, Ludwig A, Betz J, Mackay K and Givord D 1997 *J. Appl. Phys.* **81** 5420

[2] Quandt E, Ludwig A, Lord D G and Faunce C A 1998 *J. Appl. Phys.* **83** 7267

[3] Ludwig A and Quandt E 2000 *J. Appl.Phys.* **87** 4691

[4] Childs K D, Carlson B A, LaVanier L A, Moulder J F, Paul D F, Stickle W F, Watson D G *Handbook of Auger Electron Spectroscopy* (C. L. Hedberg, Physical Electronics,Inc., Minnesota, 1995 ) 1-405

[5] Shen D H, Berlinger A, Nold E, Lai W and Luo G M 1999 *Appl. Phys.* A **68** 425