

## Growth of aluminum oxide thin films on Cobalt : an AES and AFM study

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H. Oughaddou<sup>1,\*</sup>, S. Vizzini<sup>1</sup>, B. Aufray<sup>1</sup>, B. Ealet<sup>1</sup>, L. Ravel<sup>1</sup>, J-M. Gay<sup>1</sup>, J.P. Bibérian<sup>1</sup>  
and F. A. d'Avitaya<sup>1</sup>

<sup>1</sup>CRMCN-CNRS, Campus de Luminy, Case 913,13288 Marseille Cedex 09, France

### Abstract

Auger Electron Spectroscopy (AES) and Atomic-Force-Microscopy (AFM) were used to study the formation of a very thin Aluminum oxide on cobalt and the surface roughness. The results show that the superficial oxide (AlO<sub>x</sub>) which is obtained after oxidation at room temperature (RT) of one aluminum monolayer (ML), is not alumina (Al<sub>2</sub>O<sub>3</sub>). It presents a composition profile close to .../Co/Co/Al/O. This superficial oxide is very stable with temperature since there is no dissolution process of AlO<sub>x</sub> oxide at least up to 500°C. The superficial oxide formation do not change the initial surface roughness of the cobalt.

## 1 INTRODUCTION

Aluminum oxide have been extensively studied in the last years mainly for its technological applications as catalysis [1] and as insulating barrier in magnetic tunnel junctions [2].

In all cases, and specially in the magnetic tunnel junctions, the control of the growth of ultrathin films oxide on metallic surfaces is of crucial importance. Many studies are done to find procedures and experimental conditions to grow well defined and homogenous oxide layers. [3-6].

In this paper, we report a preliminary study of the first steps of the formation of very thin film of aluminum oxide on cobalt substrate. The choice of this system i.e. aluminium oxyde on a ferromagnetic substrate like cobalt, has been mainly motivated by the use of these material in the magnetic memories based on the Magnetic Tunnel Junctions properties.

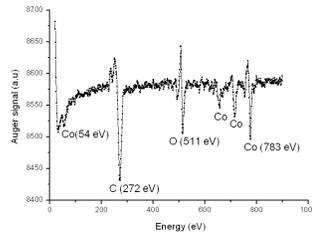
## 2 RESULTS AND DISCUSSION

The cobalt substrate was prepared by ST-Microelectronics compagny (Rouset, France) using an industrial procedure. These procedure consists to evaporate a cobalt films (15 nm) on an SiO<sub>2</sub> thin film oxide by Physical Vapor Deposition (PVD) without soft-sputter-etching (SSE). The SiO<sub>2</sub> oxide was grown on Si wafer in Sub-Atmospherical Chemical Vapor Deposition (SA-CVD) at 550°C, then polished by the Chemical-Mecanical-Polishing technic (CMP). Figure-1 shows an Atomic Force Microscopy image of the surface cobalt film, recorded at air in contact mode, just after cobalt deposition. We observe that the surface roughness is relatively small ( $RMS = 8.9 \text{ \AA}$ ).



**Fig. 1.** Initial AFM image ( $5\mu\text{m} \times 5\mu\text{m}$ ) of cobalt surface recorded in contact mode (RMS=9.8 Å).

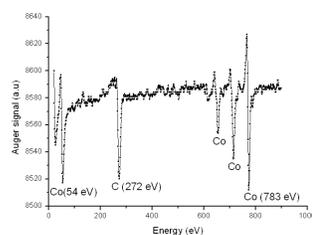
After this first characterization the sample is then introduced in a UHV chamber which contained, among other, an Auger Electron Spectrometer (AES) and an aluminium home-made evaporation source. AES spectra are always recorded in a derivative mode. The Auger spectrum of Figure-2 corresponds to the sample surface just after introduction in the UHV chamber. As expected, this spectrum is characteristic of a Co surface contaminated with carbon and oxygen. A soft ion sputtering with Ar at 600 eV for few minutes, allows us to remove oxygen from the surface but not carbon (Figure-3) which is probably due to the preparation mode.



**Fig. 2.** Initial AES spectrum showing the cobalt surface before the ion sputtering.

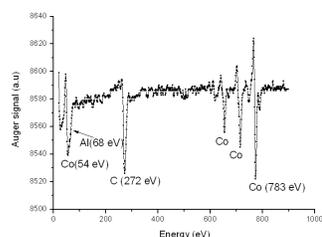
On this surface, one aluminum monolayer (ML) is then deposited (in the low  $10^{-10}\text{Torr}$  pressure range) with an evaporation rate of about 0.25 ML/min (calibrated elsewhere using a quartz balance and AES technic [7]). Figure-4 shows AES spectrum of the cobalt substrate after aluminum deposition. In comparison with Figure-3, one observes at about 68 eV a weak shoulder close to the Co Auger peak which corresponds to aluminum in a metallic state. This Al peak is not resolved due to the close proximity to the Co Auger peak ( $\text{Co}_{54\text{eV}}$ ,  $\text{Al}_{68\text{eV}}$ ). However one observes that intensities of Co peaks are attenuated ( $\text{Co}_{54\text{eV}}$ , as well as  $\text{Co}_{783\text{eV}}$ .) but, surprisingly, not the carbon one. This suggests that carbon stays on the topmost surface layer during the Al deposition.

The oxidation process was performed at RT in the same chamber. After isolation of the aluminium source, the chamber is filled by molecular oxygen at a pressure equal to  $2 \times 10^{-6}\text{Torr}$

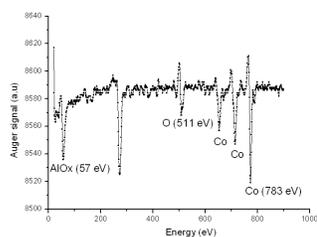


**Fig. 3.** Initial AES spectrum showing the surface state after a soft ion sputtering.

. Figure-5 shows the AES spectrum recorded at the end of the oxydation process which is very rapid since this spectrum is recorded few minutes after the oxygen introduction and do not change with time exposition. In comparison with the previous spectrum one observes that (i) the carbon Auger peak is still the same while all Co Auger peaks are attenuated, (ii) the Al Auger peak was shifted to the lower energy ( $57eV$ ) (iii) the characteristic oxygen Auger peaks due to the oxydation process.



**Fig. 4.** AES spectrum recorded after the deposition of 1 aluminum monolayer on cobalt substrate.



**Fig. 5.** AES spectrum corresponding to the complete oxidation at RT of 1 aluminum monolayer.

As expected, the Al peak has shifted to a lower energy from 68 to about  $57 eV$ . This value is quite far from that obtained from alumina  $\alpha - Al_2O_3$  ( $51 eV$ ) [8] which means that the

chemical nature of the oxide obtained is probably not alumina. Note also that all the amount of aluminum has been oxidized since the Al peak at 68 eV completely disappeared.

From the successive attenuations of the  $\text{Co}_{54\text{eV}}$  Auger peak intensities of about 28% after the deposition of 1 Al ML then of about 27% more after the oxydation process, we can propose that the composition profile close to the surface is .../Co/Co/**Al** after the deposition of aluminium and .../Co/Co/**Al/O** after the oxydation process. This conclusion is very close to the ones proposed by *Manabu Kiguchi et al.* [9], concerning the growth of MgO on Ag(001) and to the ones proposed concerning the growth of AlOx on Ag(111) using the same experimental procedure [7]. Finally the carbon seems not affect the oxydation process since it stays on the topmost surface layer. This show again that carbon could act as for thicker deposit.

In order to check the stability of the superficial oxide (AlOx) carry out at RT, we have annealed the sample up to 500°C under UHV conditions for few minutes. This thermal treatment do not affect the superficial oxyde since we do not observe evolution/modification of the different Auger peaks in relative intensities as well as energy position. This behavior clearly shows the surprising large thermal stability of this superficial oxide carry out by this way since there is no dissolution or evaporation of aluminum and/or oxygen atoms at elevated temperature. The oxidation process stabilizes aluminum atoms at the surface forming a stable AlOx compound. It should be now very interesting to modify the order of the procedure (e.g. to start by an oxydation at elevated temperature of the Co surface before the deposition of aluminum and check the possible formation at the surface of the AlCoO spinell .

At the end and in order to mesure the influence of the oxydation process on the surface roughness the sample was transfered out of the chamber for a new *ex situ* AFM analysis. Figure-?? shows an AFM image in contact mode of the surface of the sample after the oxydation process and the annealing at 500°C. The surface roughness ( $RMS = 10.2 \text{ \AA}$ ) is very close to initial cobalt substrate ( $RMS = 9.8 \text{ \AA}$ ).

### 3 CONCLUSION

In conclusion, these results show that the superficial oxide (AlOx) obtained after oxidation at RT of one aluminum ML deposited on a cobalt substrate, is not alumina. The superficial oxyde formed in this way presents a composition profile close to .../Co/Co/Al/O. It is stable with temperature since there is no dissolution process at least up to 500°C. The superficial oxide formation do not affect the initial surface roughness.

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- [\*] Author to whom correspondence should be addressed, FAX: +33 4 91 82 92 04. Electronic address: Hamid@crmen.univ-mrs.fr
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