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Institute of Physics, Maria Curie-Skłodowska Univsersity

John SULLIVAN\*, Zdzisław WROŃSKI, Sayah O. SAIED\*, Juliusz SIELANKO

# The XPS Measurements of High Dose Low-Energy Nitrogen Implanted into Aluminum

Rentgenowska spektroskopia fotoelektronowa niskoenergetycznej implantacji jonów azotu do aluminium

#### INTRODUCTION

The high dose low-energy ion bombardment is used extensively in surface analysis for removing of the contamination layer and depth profiling in techniques such as Auger electron or X-ray photoelectron spectroscopy, or as a primary beam in ion scattering spectroscopy and secondary ion mass spectrometry. Other applications include ion cleaning and conditioning, sputter deposition, semiconductor doping and lithographic pattern creation.

It is well known that such ion bombardment used in ion etching applications in depth-profiling analysis will introduce distortion in the measured profile [1, 2, 3]. In this paper the concentration profile of 2 keV nitrogen ions implanted at a high dose into aluminum, measured by means of X-ray photoelectron spectroscopy, are compared with the results of the Monte-Carlo computer simulation. The influence of the collisional atomic mixing and ionbombardment-induced segregation on the distortion of the measured depth profile of implanted ions is discussed.

#### EXPERIMENT

The low-energy implantation as well as the surface analysis was carried out in VG ESCALAB 2000D apparatus. The aluminum samples (99.99%

<sup>\*</sup> Aston University, Birmingham, UK

pure) were mechanically polished and initially cleaned in ultrasonic alcohol system. Because of the strong chemical affinity of the oxygen-aluminum set, the samples were treated in ESCALAB apparatus using a 2 keV Ar ion beam. Depth profiling was achieved by bombardment with Ar ions. The implantation of nitrogen was performed using a 2 keV N<sub>2</sub><sup>+</sup> ion beam with the current density equal 6.67  $\mu$ A/cm. X-ray photoelectron spectroscopy (XPS) was used for the surface analysis purpose. XPS spectra were recorded between successive periods of ion bombardment and concentration profiles were calculated from the peak intensities using Scofield cross sections.

#### MONTE-CARLO COMPUTER SIMULATION

The Monte-Carlo computer code used in the paper was described before [4, 5]. The XPS depth profiling computer simulation is related to the observations of the nitrogen concentration changes in the near surface layers of the implanted sample, continuously irradiated with the primary 2 keV Ar ions. To obtain the relatively good statistics of the Monte-Carlo simulations of the saturation effect ( $N_2^+$  — implantation) as well as of ion bombardment depth profiling, the "histories" of about  $10^5-10^6$  primary ions were followed in the calculations.

#### **RESULTS AND DISCUSSION**

The comparison of the results of XPS measurements of the near surface nitrogen concentration growth during the  $N_2^+$  implantation with the Monte-Carlo computer simulation is shown in Figure 1. In the calculations, the mean concentration of nitrogen is calculated for the region from the surface up to the 32 Angstrom (which is close to the mean effective escape depth for the photoelectrons).

From the data obtained during the run of the computer simulation code the calculated concentration depth distribution of implanted nitrogen is available after every  $3.3 \times 10^{15}$  cm<sup>-2</sup> dose step of nitrogen implantation. In the Figure 2a the last calculated profile determined at the end of implantation time (~5000 sec., see Figure 1) is presented. Additionally in the Figure 2a the calculated from SUSPRE program profile of implanted N<sub>2</sub> is shown. The last one was calculated for the solubility of nitrogen equal 32%.

The calculated M-C concentration depth distribution of implanted nitrogen formed at the end of implantation was taken as the starting profile to the XPS-depth profiling computer simulation.



Fig. 1. The atomic concentration of nitrogen in Al as a function of implantation time. Comparison of the XPS-measurements with the Monte-Carlo calculations

Ryc. 1. Koncentracja atomowa azotu w Al jako funkcja czasu implantacji. Porównanie danych eksperymentalnych (XPS) z obliczeniami symulacji komputerowych Monte-Carlo



Fig. 2. a) The calculated nitrogen depth concentration profile in the Al sample. b) The nitrogen concentration profile after implantation of N<sub>2</sub> to the Al sample as a function of Ar ion etching time. Comparison of the MC-calculations with the XPS-measurements Ryc. 2. a) Rezultaty obliczeń glębokościowego rozkładu azotu w próbce Al. b) Rozkład koncentracji zaimplantowanego do Al azotu w funkcji czasu trawienia wiązką jonów Ar. Porównanie obliczeń Monte-Carlo z danymi z pomiaru metodą XPS

The results of nitrogen concentration depth profiling measurements, and comparison with the calculations, are presented in Figure 2b. The profiling was performed using 2 keV Ar ion beam for the 'layer-by-layer' micro sectioning. For such energy of  $Ar^+$  ions, their penetration depth is comparable with that of implanted nitrogen. In such a case a great influence of atomic mixing and preferential sputtering can be expected. The atomic mixing, which is connected with the build-up of collisional cascade, is a fundamental contribution to the broadening of depth profile. The preferential sputtering is responsible for the change of the composition of the near surface layers of a multicomponent system due to a variety of physical and chemical processes and may be responsible for significantly lowering the measured concentration in comparison with that real in the sample before the starting of the Ar bombardment etching process. The disagreement between the experiment and the calculated profile (concentration vs ion etching time) in the tail of profile can come from the diffusion — or radiation enhanced diffusion — of nitrogen in aluminum that is not taken into account in computer simulations.

#### ACKNOWLEDGMENT

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#### STRESZCZENIE

Niskoenergetyczne bombardowanie jonami jest szeroko stosowane w analizie powierzchni ciała stałego do zdzierania kolejnych warstw w celu określenia profili głębokościowych przy użyciu takich metod, jak spektroskopia elektronów Augera (AES) lub rentgenowska spektroskopia fotoelektronowa (XPS). Niskoenergetyczne bombardowanie jest używane także do szybkiego czyszczenia powierzchni, nakładania warstw metodą rozpylania i w wielu innych aplikacjach.

Jest oczywiste, że bombardowanie jonowe, nawet niskoenergetyczne, powoduje znaczne zaburzenia mierzonych profili głębokościowych w analizach AES i XPS. W niniejszej pracy porównano profile głębokościowe azotu zaimplantowanego z niskimi energiami (2 keV) do aluminium, zmierzone metodą XPS z odpowiednimi profilami otrzymanymi przy komputerowej symulacji metodą Monte-Carlo. Wykazano, że bombardowanie jonami argonu, użyte do usuwania kolejnych warstw, silnie deformuje profile głębokościowe zaimplantowanego azotu poprzez proces mixingu i wybiórczego rozpylania.