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ION-BEAM-ASSISTED DEPOSITION OF Ni/C MULTILAYER X-RAY MIRRORS

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The reflectivity and the energy resolution of multilayer X-ray mirrors depend on the interface roughness created during deposition of the layers. We have investigated the influence of argon ion impact during and after deposition by evaporation of nickel on the interface roughness of an Ni/C multilayer. Roughness changes have been monitored by X-ray reflection. Etching of one and a half periods of nickel by argon ions of energy 200 eV has turned out to improve the reflectivity of one interface by a factor of 3.

1. INTRODUCTION

Multilayer systems have been shown to be applicable in the reflection of soft X-rays. The principle is based on the interference of radiation reflected by interfaces of materials with high (tungsten, nickel, rhodium and rhenium) and low (silicon and carbon) refractive indices[1,2]. Material combinations which do not interdiffuse have to be chosen. The reflection at each interface depends not only on the difference in refractive index but also on interface roughness[1,2]. The roughness increases mainly during deposition of the metal component[3]. There are indications that the impact of energetic particles on the substrate during deposition reduces the roughness of the surface of a growing metal layer. This explains the better reflectivity of multilayers deposited by sputtering compared with those deposited by evaporation. In this paper an investigation of the influence of argon ion bombardment on the interface roughness of the electron-beam-evaporated Ni/C system is reported. Ion beam modification during as well as after deposition is investigated.

2. EXPERIMENTAL AND ANALYSES TECHNIQUES

Deposition and sputtering have been performed in an ultrahigh vacuum system with a base pressure of 10^{-8} Pa. Layers of carbon and nickel were deposited using two electron beam evaporators. Changes in layer thickness during growth and ion milling have been monitored by a soft X-ray reflection system (N Kα, $\lambda = 3.16$ nm, $\theta = 35°$)[4]. A quartz crystal monitor could be used during deposition to compare the layer thickness with that obtained from the interference maxima in the reflected soft

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X-ray signal. A Kaufman source was used to produce Ar^+ ions with energies below 1 keV at an angle of incidence of 45°. The source was differentially pumped, in order to keep the working pressure below 10^{-5} Pa. The ion energy was kept below 1 keV, in order to prevent intermixing of the deeper interfaces as much as possible (on the basis of a simulation with TRIM[5]).

Since a low surface roughness is needed to obtain a maximum X-ray reflectivity, Si(111) crystals were used as substrates. The substrates were kept at room temperature.

Three types of experiments were performed.

(a) Nickel was deposited on an Si(111) surface, followed by etching with 750 eV argon ions.

(b) Nickel was deposited on an Ni/C multilayer, followed by etching with 750 eV as well as 200 eV argon ions.

(c) Nickel was deposited on an Ni/C multilayer during 200 eV argon ion bombardment.

The reflectivity as a function of film thickness was measured and fitted to a computational scheme which solves the Fresnel equations and uses the Debye–Waller factor to describe interface roughnesses[4]. The complex indices of refraction were calculated from the density and the atomic scattering factors f_1 and f_2 [6].

3. RESULTS AND DISCUSSION

In Fig. 1 the X-ray reflectivity during the growth (full line) and sputtering (dotted line) of a thick nickel layer on top of an Si(111) substrate is shown. The maximum thickness of the nickel layer on top was 12.5 nm. The distance between the interference maxima (2.7 nm), extracted from the Bragg relation, agreed with the

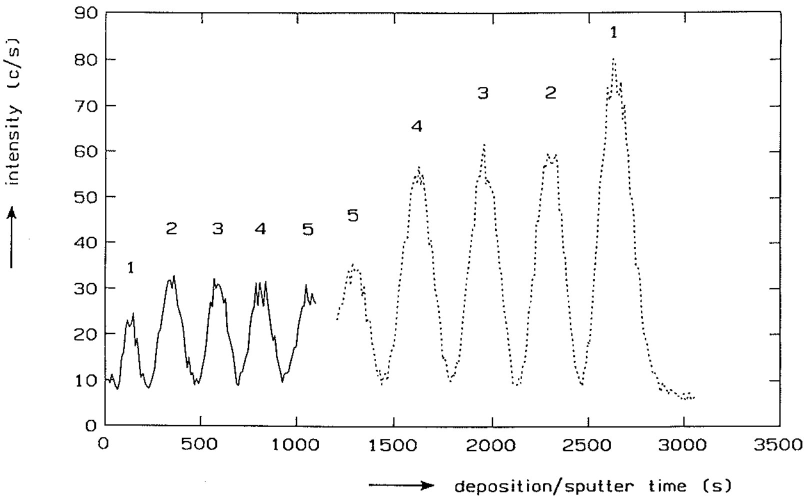

Fig. 1. The reflectivity of N Kα X-rays ($\lambda = 3.16$ nm, $\theta = 35°$) as a function of the nickel layer thickness, during growth (———) and during destructive sputtering using Ar^+ of energy 750 eV (····). The maximal nickel thickness was 12.5 nm.

thickness as measured with the quartz crystal within its accuracy (3%), assuming bulk density for nickel. In order to simulate the reflectivity curve during growth, an increase in the surface roughness had to be assumed. Taking an initial roughness of the silicon surface of 0.4 nm, we obtained a final roughness of 0.8 nm. Sputtering was then performed using Ar^+ ions of energy 750 eV (ion current, 1.5×10^{-5} A cm^{-2}). The increasing amplitude of the oscillations in the reflected X-ray signal indicates either a sputter-enhanced smoothing of the top surface or an increase in the nickel density. A density increase of at least 30% is needed to explain the measured reflectivity curve. This, however, is rather unlikely to occur[7]. We therefore suggest that the increase in the oscillations of the curve which represents ion milling should be explained by a reduction in the surface roughness to less than 0.2 nm.

The full line of Fig. 2 shows the X-ray reflectivity during growth of an Ni/C multilayer consisting of four periods of 2.7 nm (I–IV) with a thick nickel layer (12.5 nm) on top (oscillations 1–5). In the first part of the curve the growth of the multilayer is represented by an increasing X-ray reflection during nickel deposition, followed by a decreasing reflection during carbon deposition. The enhanced average reflection for a system of four periods is caused by interference of the radiation reflected from the four interfaces. The other five oscillations belong to the deposition of the nickel top layer of thickness 12.5 nm. Simulation revealed an increasing surface roughness during deposition of the thick nickel top layer, by 0.4 nm. The dotted line represents the X-ray reflectivity of the stack during sputtering of the film using Ar^+ ions of 750 eV. The reflectivity curve obtained during sputtering of the first 12.5 nm of nickel can be simulated when a decrease in the surface roughness of more than 0.4 nm is assumed. The reflection pattern obtained during sputtering of the multilayer below the thick nickel film on top would suggest an increasing surface roughness or might be explained by interface roughening or mixing. Calculations

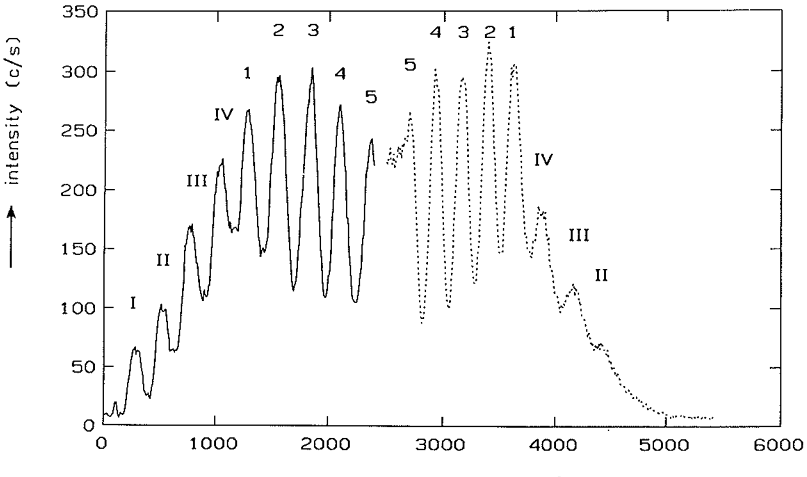

Fig. 2. The reflectivity of N Kα X-rays ($\lambda = 3.16$ nm, $\theta = 35°$) for an Ni/C multilayer of four periods, having a periodicity of 2.7 nm and a thick nickel layer (12.5 nm) on top: ———, during growth of the system; ····, the reflectivity during sputtering of the system using Ar^+ (750 eV).

with the computer code TRIM86 for Ar^+ ions of energy 750 eV on a multilayer consisting of 1.3 nm of nickel, 1.3 nm of carbon and 17.4 nm of nickel, at perpendicular incidence, were carried out. The results of these calculations confirmed that intermixing of the first interface should be expected and that the influence on the second interface is considerably less. The reflectivity behaviour during ion milling of the multilayer in Fig. 2 is therefore probably due to the intermixing at the first interface, resulting in a decreasing amplitude of the interference oscillations.

In order to apply ion beam modification to an Ni/C system with a period of 2.7 nm, without intermixing of deeper interfaces, TRIM86 calculations were performed for Ar^+ ions of different energies. From these calculations it was concluded that, for 200 eV argon ions, the maximum in the ion range profile is situated 0.5 nm below the surface, with a tail ending up at a depth of 1.3 nm.

As described before, a set of multilayers with four periods of 2.7 nm was produced, of which the thickness of the fifth nickel (top) layer varied from half a period (1.35 nm) to 2.5 periods (6.75 nm). Afterwards, the thicknesses of all nickel top layers were reduced to half a period by ion milling with an energy of 200 eV. As in Fig. 2, the process was monitored by soft X-ray (N Kα) reflection during growth and sputtering. Figure 3 shows the final reflection of N Kα radiation after the ion milling process, normalized to the reflection of a system with the top layer of half a period "as deposited". A maximum gain in the reflection by a factor of 3 can be observed for the system in which an excess layer of 1.5 periods was removed by ion milling. Removal of two extra periods only results in a gain by a factor of 2.

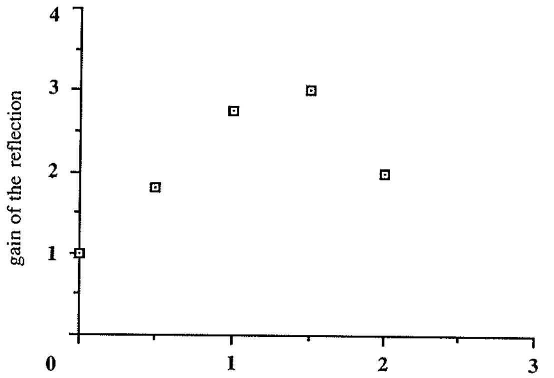

Fig. 3. Gain in the peak intensity of reflected N Kα radiation by multilayers with five periods, after modification of the top nickel layer by argon ions of energy 200 eV, with respect to the same multilayer system without any ion beam treatment. Modification of the top layer is carried out by removing the extra periods by ion milling, until half a period is left.

Finally, the effect of ion impact during deposition was investigated. A basic four-period multilayer was produced. In order to be able to compare with the results obtained as described before, ion-beam-assisted deposition was performed during the growth of the fifth nickel layer. To keep the mixing of the first interface as low as possible, ion bombardment was started after preliminary deposition of 0.6 nm of nickel. The result of this process was a reflectivity gain by a factor of 2, with respect to a non-modified multilayer.

The decreasing roughness of the surface of the top layer can be explained as follows. In the energy range used in our experiments, particles are removed from the surface by recoil effects. It is clear that the particles with the lowest binding energy, with the fewest nearest neighbours, will be removed first. Therefore particles on top of islands will be removed first, reducing roughness. Depending on the initial roughness before ion milling, it can be understood that the resulting roughness after ion milling depends on the number of layers removed. It can also be expected that a limit in the roughness reduction is obtained after removing a layer of a certain thickness (1.5 periods for nickel). However, we do not yet understand why removing more than 1.5 periods by ion milling results in a lesser reduction in the roughness of the nickel surface.

It is also not yet clear why ion-beam-assisted deposition does not result in the lowest surface roughness. However, in that case, according to TRIM calculations, interface mixing effects cannot be excluded and will be investigated for different ion energies.

4. CONCLUSIONS

We conclude that argon ion beams can be successfully applied in the reduction of the interface roughness of Ni/C multilayer systems. A low enough ion energy has to be chosen to prevent intermixing in the deeper interfaces. Treatment with ions after deposition results in a higher reduction in the roughness than ion-beam-assisted deposition.

It is worth investigating the total gain in reflectivity of a multilayer system of which all interfaces are treated by ions.

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