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Development and Application of Small d-spacing Multilayer Mirrors by Ion polishing

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Abstract

Ni/Ti and Ni/Mn multilayer neutron mirrors which were deposited with ion beam sputtering deposition and ion polishing have been investigated. The dependencies of ion polishing time, ion acceleration energy and incidene angle on the interface roughness were studied for the optimization of beam parameters. The dependency of multilayer period spacing of d on the interface roughness was also studied to determine the minimum d-spacing at which ion-polishing works well. TEM observation was conducted on these multilayers for the observation of morphology and inter-diffusion between layers. It was clearly observed that the interface roughness of multilayer was reduced by ion polishing.

1. Introduction

Multilayer neutron mirrors[1] with high reflectivity could be achieved if the interface roughness of the multilayer can be kept small compared to the bilayer spacing. One of the most important problems in producing the small d-spacing multilayers is the reduction of the interface roughness which becomes larger with the number of bilayers deposited. We have applied and succeeded in argon ion polishing in combination with ion beam sputtering deposition for the fabrication of Ni/Ti multilayers[2]. In this study, the effectiveness of argon ion polishing from viewpoints of the ion polishing time, ion acceleration energy and incidence angle on the interface roughness were investigated for Ni/Ti and Ni/Mn multilayers. The minimum d-spacing at which ion-polishing works well was studied by fabricating some multilayer mirrors with very small d-spacing.

2. Development of ion polishing technique in combination with ion beam sputtering deposition

2.1 Deposition system and sample preparation

Ni/Ti and Ni/Mn multilayers were deposited and ion-polished using an ion beam sputtering system as shown in Fig.1[3]. The system is equipped with dual bucket sources those are used to generate Ar^+ ions. One is used for sputtering deposition of multilayers. The other is used for ion polishing. The conditions of sputtering deposition were fixed for all multilayer mirrors, on the other hand, these of the ion polishing were scanned. The ion polishing was applied immediately after the deposition of each layer to smoothen the layer. Multilayers were deposited on Si(111) substrates of 75mmϕ with surface roughness of 2Årms ~ 4Årms. The base pressure during the operation was 1×10^{-7} mbar.

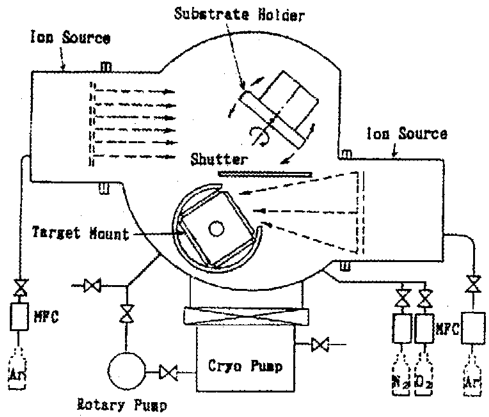


Figure 1 Schematic diagram of the ion beam sputtering deposition system.

2.2 Optimization of the ion beam parameters

In order to get the dependencies of ion polishing time, we have coated and characterized Ni/Ti multilayers with d-spacing of d = 120 Å and the number of bilayers N = 10 pairs and also Ni/Mn with d = 100 Å and N = 10 pairs. Either Ni layers or Ti (or Mn) layers were ion-polished for various Ar^+ ion polishing times. The ion acceleration energy and incidence angle were fixed at 100eV and 10° during this coating.

The d-spacing and interfacial roughness of multilayers were evaluated with X-ray grazing angle reflectivity measurements, which were performed in a θ-2θ mode using Cu K_α radiation (λ=1.54Å). The measured reflectivity was fitted with the reflectivity which was calculated using the Fresnel's formulae and the interface roughness considered in terms of the Debye-Waller factor σ, assuming the optical parameters of the material.

The evaluated interface roughness σ of the multilayers are shown in Fig.2, which illustrate that the ion polishing (in all cases) is very effective for the reduction of σ. In cases of ion-polishing nickel layers of Ni/Ti multilayer, the interface roughness of 6.5 Å r.m.s. decreases to a minimum value of 3.5 Å r.m.s. at a polishing time of 69 sec. The value of σ is evaluated to be 4.5Å at a polishing time of 120 sec and constant over every polishing time in cases of ion polishing titanium layers. On the other hand, Ni/Mn multilayer shows the almost same tendency. In cases of ion-polishing nickel layers, the interface roughness of 5.3 Å r.m.s. decreases to a minimum value of 4.1 Å r.m.s. at a polishing time of 69 sec. The value of σ is evaluated to be 3.9 Å r.m.s. at a polishing time of 60 sec and constant over every polishing time in cases of ion-polishing manganese layers.

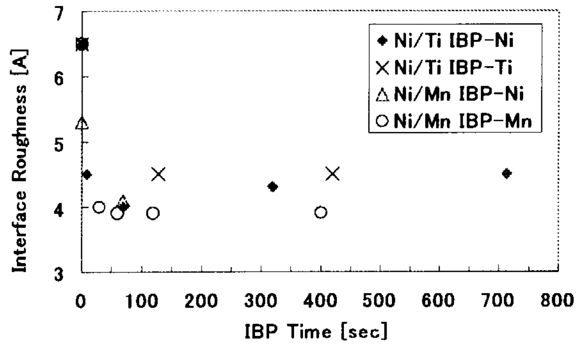


Fig.1 Evaluated interface roughness of Ni/Ti and Ni/Mn multilayers which were ion-polished as a function of ion-polishing time.

The dependencies of Ar^+ ion acceleration energy and incidence angle have been investigated. Ni/Ti and Ni/Mn multilayers (d = 100 Å, N = 10 bilayers) with either ion-polished nickel layers or ion-polished titanium (or manganese) layers were coated for various ion acceleration energies (100 eV, 300 eV and 600 eV) and incident angle (10° and 45°). In the study of ion acceleration energy dependence, the ion polishing time was determined to fix the quantity of sputtered atoms in consideration of the sputtering rate which increases in proportion to the acceleration energy. In case of the study of incident angle dependence, the ion polishing time was determined to fix the ion flux with which the multilayer surface was irradiated.

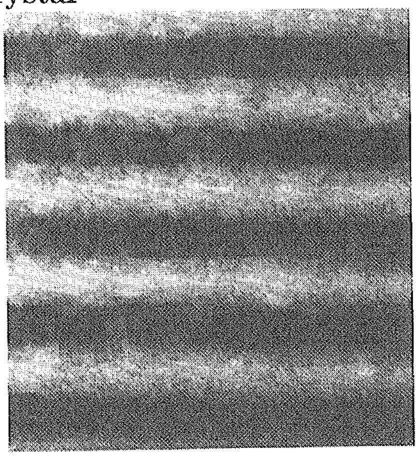
It was observed that σ of all the samples ion-polished, becomes smaller than those of the sample without ion-polishing. The results did not show clear ion-energy dependence both in cases of Ni/Ti and Ni/Mn multilayers.

In case of the incident angle of 45°, σ of the sample with Ni layers ion-polished at 100eV may be smaller than that of the sample ion-polished at 10°. On the other hand, σ of the sample with Ti layers ion-polished at 45° and 100eV shows the same value with σ of the sample ion-polished at 10°.

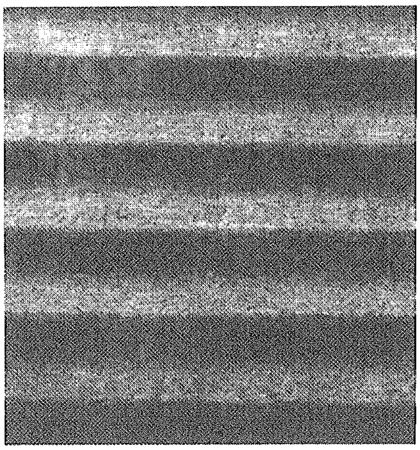
2.3 Cross-section observation by transmission electron microscope

The deposited multilayer structure was investigated by cross-sectional observations of the multilayer using a high resolution transmission electron microscopy (TEM, HITACHI Ltd. H-9000NAR), which was operated at 300 kV. The maximum resolution was 1.8 Å. Cross-sectional TEM samples were thinned mechanically, followed by an Ar^+ ion milling to perforation.

Photograph 1-(1) shows a TEM image (magnification : × 2,000,000) of non-polished Ni/Ti multilayer consisting of d = 100 Å and N = 20 bilayers. The dark layers correspond to nickel layers and the gray layers correspond to titanium layers. The large grained nickel is observed, which results in increase of the interface roughness. Some crystallinities as well as the Ni(111) texture of nickel layer are observed. The contrast variation in the nickel layers is due to different small angular tilts (of the order of 1 degree) of the Ni(111) crystallites away from the direction normal to the substrate surface. The titanium layers show micro-crystal



(1) non ion-polished case



(2) ion-polished case

Photograph 1 TEM images (magnification : × 2,000,000) of the structure of (1) non ion-polished and (2) ion-polished Ni/Ti multilayers with d = 100 Å and N = 20 bilayers.

structures with some amorphous region. One can observe the inter-diffusion region with thickness of about 20 Å at the both side of each Ni layer. The silicon substrate is covered by a native oxygen layer, upon which 20 bilayers of nickel and titanium were deposited.

Photograph 1-(2) shows a TEM image (magnification : × 2,000,000) of the structure of an ion-polished Ni/Ti multilayer with d = 100 Å and N = 20 bilayers. The interfaces are very sharp and inter-diffusion between the nickel and the titanium layers as well as roughness may be decreased in comparison with the non-polished sample. The amount of the Ni(111) crystal texture increased, it may be said that argon ion polishing contributes to the texture orientation as pointed out by Y. Nagai[(4)]. The titanium layers show quasi-amorphous structure. The inter-diffusion region is observed with thickness of about 10Å at the lower side of each Ni layer.

3. Very small d-spacing multilayers

Very small d-spacing multilayer mirrors were coated at the above mentioned optimal ion polishing condition. We coated Ni/Ti and Ni/Mn multilayers with d = 20 Å, 26 Å, 30 Å and N = 50, 300 pairs. All nickel and titanium layers were ion-polished. Figure 3 shows the evaluated interface roughness σ of Ni/Ti and Ni/Mn multilayers with and without ion-polishing as a function of d-spacing. Results show that the interface roughness decreases at all of the cases of d-spacing less than 27 Å. It decreases to 3.5 Å r.m.s in case of d = 26.6 Å and N=50 paires, and it is kept to be 4.3 Å r.m.s. in cases of d = 26 Å and 30 Å even if the number of layers reaches 300 pairs. On the other hand, the interface roughness decreases to 8.2 Å r.m.s in case of d = 20 Å. It may be concluded that the intermixing of interface takes place by argon ion bombardment when the layer thickness is 20 Å and the critical boundary of layer thickness at which intermixing takes place may exist between the layer thickness of 20 Å and 26 Å.

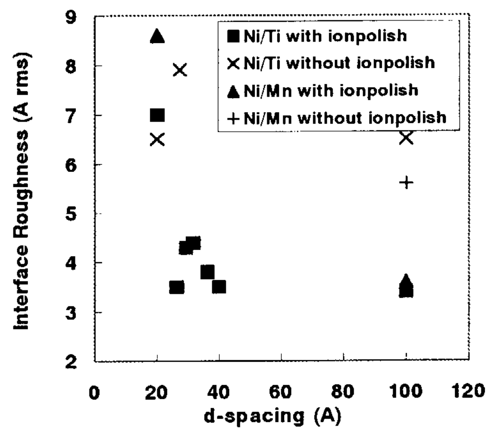


Figure 3 Evaluated interface roughness σ of Ni/Ti and Ni/Mn multilayers with and without ion-polishing as a function of d-spacing.

4. A neutron bender for epi-thermal neutrons

In this section, we have discussed a neutron bender would be utilized for the deflection of epi-thermal neutrons. At spallation sources, t-zero choppers have been used to block the fast neutrons and to have the thermal neutrons of interest. But the use of epi-thermal neutrons is limited up to about 1eV because the chopper materials are so heavy and the beam is fully open at that energy. On the other hand, neutron benders are also used at sources to eliminate the fast neutrons and pass the thermal and cold neutrons. If a supermirror with the very high critical angle is developed, the wavelength of available neutrons deflected by the bender would be higher.

The neutron beam transmitted through the curved guide with multiple reflections is characterized by the characteristic wavelength λ^* which is defined as follows :

$$\lambda^* = \sqrt{\frac{2a}{\rho}}\sqrt{\frac{\pi}{Nb_{coh}}} \bullet \frac{1}{m}$$

where a is the width of the neutron guide channel and ρ the radius of curvature, N the atomic number density, b_{coh} the average coherent scattering length and m multiplication factor of a supermirror to a nickel mirror. In case of the use of a soller type bender with a beam width of 1 cm, a = 1 mm and a total length of 4.3m, neutrons up to 0.09 Å may be deflected at the radius of curvature of 2291 m by using m=6 supermirrors. Then a shielding with a width of 11 m is required to separate a direct beam 1cm apart.

Development of M=6 supermirror have been tried using m=4 supermirror and a set of 20 plates of monochromators with variable d-spacings which cover the wide q-range. One of the most important problems for producing such supermirrors is the absorption by each substrate, then substrates of those monochromators are thinned using optical polishing and chemical etching.

6. Conclusion

We have characterized some ion polished Ni/Ti and Ni/Mn multilayers those were deposited with ion beam sputtering. It was observed that the reflectivity of multilayers and the interface roughness are obviously improved by using ion polishing. For the best condition of the ion polishing, the interfacial roughness decreased to 4 Å r.m.s. even if the number of layers reach 300 pairs. This result indicates that we may have a very small d-spacing multilayer mirror which have an enough reflectivity for practical use.

References

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