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Modification of Fe/Nb Multilayers under Xe-ion Irradiation*

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Abstract: The behavior of the metallic multilayers of Si/[Fe(10 nm)/Nb(4 nm)/Fe(4 nm)/Nb(4 nm)]$_2$/[Fe(4 nm)/Nb(4 nm)]$_4$ under 2 MeV Xe-ion irradiation has been investigated by depth profile analysis of Auger electron spectroscopy, X-ray diffraction and vibrating sample magnetometer. The obtained experimental results show that the inter-mixing between Fe and Nb layers occurs in the 1.0×10^{14} ions/cm^2 irradiated multilayer sample which results in the formation of Nb-based and Fe-based FeNb solid solution. For the samples irradiated to fluence larger than 1.0×10^{14} ions/cm^2, amorphisation is observed, and moreover, the layered structure of the multilayer samples is broken up completely for the samples under 1.0×10^{16} or 2.0×10^{16} ions/cm^2 irradiation. Vibrating sample magnetometer measurement also reveals that the magnetization of the samples changes with the evolution of the structure of multilayers. Possible mechanism of the modification in Fe/Nb multilayers induced by Xe-ion irradiation is briefly discussed.

Key words: ion irradiation; Fe/Nb multilayers; depth profile analysis of AES; XRD; VSM

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1 Introduction

Metallic multilayers with modulation wavelength in the nanometer range show special properties[1－8]. Extensive experimental and theoretical works indicated that the interfacial quality plays a very important role in determining the properties of the multilayers[9－16]. For instance, perpendicular magnetic anisotropy caused by surface-anisotropy is very sensitive to the morphology at the interfaces[17]. Giant magnetoresistance in magnetic multilayers is related to the scattering of conduction electrons at or near the interfaces in the antiferromagnetic phase[18, 19]. So the investigation of interface is of importance to understand the origins of multilayer properties.

In order to clarify the role of interface in properties of multilayers, techniques to modify the interfacial structure are requested. Ion irradiation, as a nonequilibrium processing technique, could deposit energy to a high density in matter, which could induce enhancement of atomic diffusion as well as phase formation and phase segregation[20－28]. Therefore, ion irradiation could be used to study the relationship between the interfacial structure and multilayer properties.

In the present work, Fe/Nb multilayers were irradiated at room temperature(RT) with 2 MeV

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Xe-ion to different fluences. From the investigations of depth profile of Fe/Nb atoms, crystallite structures and magnetic properties of the multilayers, the modification of Fe/Nb multilayers induced by ion irradiation was studied.

2 Experiment

Si/[Fe(10 nm)/Nb(4 nm)/Fe(4 nm)/Nb(4 nm)]$_2$/[Fe(4 nm)/Nb(4 nm)]$_4$ (the subscript refers to layer numbers) multilayers were prepared by alternating depositions of pure iron (99.99% Fe) and niobium (99.95% Nb) on cleaned Si(100) substrates by magnetron sputtering at RT. The base vacuum was less than 2.4×10^{-4} Pa, and the vacuum during deposition was kept at about 0.68 Pa. The thickness of the film was monitored in-situ by a film thickness monitor during the deposition.

The prepared multilayers were irradiated at RT with 2 MeV Xe-ions to 1.0×10^{13}, 1.0×10^{14}, 1.0×10^{15}, 1.0×10^{16} or 2.0×10^{16} ions/cm^2, respectively. Then the samples were characterized using depth profile analysis of Auger electron spectroscopy (AES), X-ray diffraction (XRD) and vibrating sample magnetometer (VSM). The AES measurements were performed on an Auger Microprobe PHI700 with the electrons of 3 keV impinging at 30° to the specimen surface. The samples were sputtered with argon ions. The XRD analyses were carried out on a Philips Expert Pro spectrometer under a glance angle of about 1 degree, and Cu K_α line was selected as the incident light. The hysteresis loops were measured using a Lakeshore 730 VSM with the external field applied in the sample plane. Both crystallite structure change and interfacial atom diffusion of the multilayers corresponding to irradiation fluence were observed.

3 Results and Discussion

The structural evolution of multilayers is investigated as function of irradiation fluence. AES depth profiles of the as-deposited sample, along with the multilayers irradiated at RT with 2 MeV Xe-ion are shown in Fig. 1(a)—Fig. 1(f). The abscissa and ordinate represent the sputter time and concentrations of Si, Fe, and Nb atoms respectively. Considering the surface sputtering during irradiation, the AES results are revised to make sure that the maximum ratio between Fe-concentration and Nb-concentration at the surface Fe layer are located at the same abscissa. Fig. 1(a) shows the measured depth profile of as-deposited multilayers. It is found that the interfaces of the as-deposited multilayers are not sharp, and the rather wide interfaces between the Fe and Nb layers could be observed, which may be caused by inter-diffusion of Fe and Nb atoms during deposition, whereas the layered structure also could be observed as expected. Fig. 1(b) shows that upon irradiation with Xe-ion to 1.0×10^{13} ions/cm^2, no evident change of the AES depth profiles is observed. However, as shown in Fig. 1(c), after irradiated to 1.0×10^{14} ions/cm^2, the concentration of Fe(Nb) in Nb(Fe) layers increases, which indicates that the inter-mixing between Fe and Nb layers starts to occur when fluence up to 1.0×10^{14} ions/cm^2. The concentration change of Fe(Nb) in Nb(Fe) layers is more evident in Fig. 1(d), it is certain that irradiation results in inter-mixing between Fe and Nb layers after irradiated to 1.0×10^{15} ions/cm^2. Up to fluence of 1.0×10^{16} or 2.0×10^{16} ions/cm^2, it is obviously that the inter-mixing between Fe and Nb layer results in the lost of the concentration peaks of Fe and Nb layers.

Fig. 2 shows the XRD patterns recorded on the as-deposited as well as irradiated samples. The peaks situated at the region from 50 to 60 arise from Si substrate, so it has been omitted from the figure. For the as-deposited sample, The Fe(100), Fe(200), Fe(211) and Nb peaks are visible, but the peaks are broadened. This may be from the small grain size. After the 1.0×10^{13} ions/cm^2 irradiation, the position of all the peaks has no evident

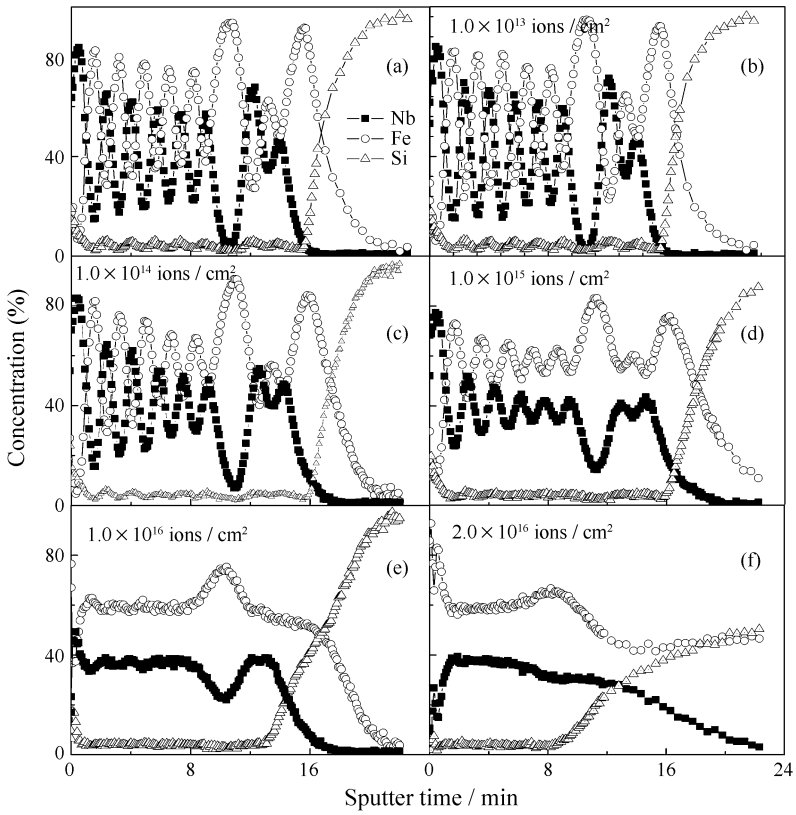

Fig. 1　AES depth profiles of Si/[Fe(10 nm)/Nb(4 nm)/Fe(4 nm)/Nb(4 nm)]$_2$/[Fe(4 nm)/Nb(4 nm)]$_4$.

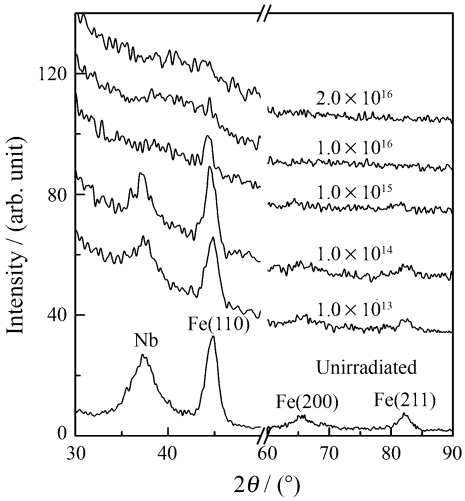

Fig. 2 XRD patterns of Si/[Fe(10 nm)/Nb(4 nm)/Fe(4 nm)/Nb(4 nm)]$_2$/[Fe(4 nm)/Nb(4 nm)]$_4$.

shift comparing with that of the as-deposited multilayers. After irradiation with 1.0 × 10^{14} ions/cm^2, appearance of a new peak at 2θ～35.97°, accompanying with the Nb and Fe(100) peaks shifted to lower angles comparing with that of the as-deposited multilayers, which indicate the position of the peaks may correspond to the Nb-based and Fe-based FeNb solid solution respectively. After irradiated with 1.0×10^{15} or 1.0×10^{16} ions/cm^2, besides a broad hump arising from amorphous FeNb alloy only the crystalline peak corresponding to the Fe-based FeNb solid solution could be found. In the samples irraliated with 2.0×10^{16} ions/cm^2, only a clear broad hump indicating formation of amorphous FeNb alloy could be found. All these results reveal that ion irradiation induces the intermixing between the Fe and Nb layers and formation of Nb-based and Fe-based FeNb solid solution and amorphous FeNb alloy.

Ion irradiation induces the atomic transport through the interfaces and the phase transformation in multilayers, which directly affects the magnetic property of multilayers. Fig. 3 shows the hysteresis loops measured in the direction of parallel to the film plane of the samples at RT. Only the reduction of the saturation magnetization could be observed after irradiated up to 1.0×10^{13} and

1.0×10^{14} ions/cm^2, for larger fluences of 1.0×10^{15} or 1.0×10^{16} ions/cm^2, both saturation magnetization and the coercivity decrease obviously. At fluence of 2.0×10^{16} ions/cm^2, the magnetization displays a paramagnetic behavior with a monotone increase as a function of the applied magnetic field; exhibits no hysteresis and no sign of saturation. The modification of the multilayers induced by ion irradiation can be divided into two steps: atomic collision cascade step and consequent relaxation step. Atomic cascade, a process of far-from equilibrium state, enhances Fe and Nb interdiffusion and induces atomic transport through interface, determines the atoms ratio of different materials at the interfacial disorder region. During the relaxation process, controlled by the thermodynamic and kinetic factors, the atomic rearrangements take place at the interfacial disorder region. Because the process is extremely short, the disorder region relaxation and phase transfer are not complete and thus a possible intermediate or metastable state could be obtained. At low fluence, the mixing is limited and the crystalline structure of the samples is not destroyed completely, there are Fe-based and Nb-based disorder domains at the nearside of the Fe layer and Nb layer. During relaxation process, it is easy to form Fe-based and Nb-based FeNb solid solution at the disorder region; So after the irradiation of 1×10^{13} ions/cm^2, mixing between the Fe and Nb layers and the formation of FeNb alloys are not observed on the AES and XRD patterns, but it may be that limited FeNb alloys form at the interface of the multilayers, which induces the decrease of the saturation magnetization value. For the samples irradiated at 1.0×10^{14} ions/cm^2, the formation of the Nb-based and Fe-based FeNb solid solution lead to a decrease in the saturation magnetization. For high fluence irradiation, the spatial superposition of cascades induces the long-range migration and the Fe and Nb layers mix completely. The completely disordered samples relax in a short process and the amorphous FeNb alloy may form. After irradiated at 1.0×10^{15} or 1.0×10^{16} ions/cm^2, the formation of amorphous and crystalline FeNb solid solution lead to the decrease of the saturation magnetization and coercivity. At highest fluence, only amorphous FeNb alloy forms as the layered structure are broken up and the hysteresis loop demonstrates paramagnetic character.

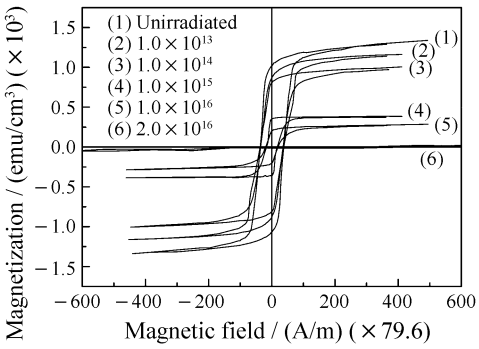

Fig. 3 Hysteresis loops of Si/[Fe(10 nm)/Nb(4 nm)/Fe(4 nm)/Nb(4 nm)]$_2$/[Fe(4 nm)/Nb(4 nm)]$_4$.

4 Summary

Modification of Si/[Fe(10 nm)/Nb(4 nm)/Fe(4 nm)/Nb(4 nm)]$_2$/[Fe(4 nm)/Nb(4 nm)]$_4$ multilayers under 2 MeV Xe-ion irradiation at RT has been revealed by use of AES, XRD and VSM measurements. It is clear that the mixing between the Fe and Nb layer induces the formation of the Nb-based and Fe-based FeNb solid solution at a fluence of 1.0×10^{14} ions/cm^2. Amorphisation is observed when the samples are irradiated to fluence larger than 1.0×10^{15} ions/cm^2. The Fe and Nb layers are completely mixed after the Xe-ion irradiation fluence up to 2.0×10^{16} ions/cm^2. Furthermore, the VSM results suggest that the formation of the Nb-based and Fe-based FeNb solid solution leads to the decrease of the saturation magnetization and hysteresis loop demonstrates paramagnetic character when the amorphous FeNb alloy forms after irradiated up to 1.0×10^{16} ions/cm^2.

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Fe/Nb 多层膜中离子辐照效应研究*

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摘　要: 采用磁控溅射技术在 Si 衬底上沉积 Si/[Fe(10 nm)/Nb(4 nm)/Fe(4 nm)/Nb(4 nm)]$_2$/[Fe(4 nm)/Nb(4 nm)]$_4$ 多层膜。用 2 MeV 的 Xe 离子在室温下辐照多层膜。采用俄歇深度剖析、X 射线衍射和振动样品磁强计分析辐照引起的多层膜元素分布、结构及磁性变化。AES 深度剖析谱显示当辐照注量达到 1.0×10^{14} ions/cm^2 时, 多层膜界面两侧元素开始混合; 当辐照注量达到 2.0×10^{16} ions/cm^2 时, 多层膜层状结构消失, Fe 层与 Nb 层几乎完全混合。XRD 谱显示, 当辐照注量达到 1.0×10^{14} ions/cm^2 时, Nb 的衍射峰和 Fe 的各衍射峰的峰位相对于标准卡片向小角方向偏移, 这说明辐照引起 Nb 基和 Fe 基 FeNb 固溶体相的形成; 当辐照注量大于 1.0×10^{15} ions/cm^2 时, 辐照引起非晶相的出现。VSM 测试显示, 多层膜的磁性随着结构的变化而变化。在此实验基础上, 对离子辐照引起界面混合现象的机理进行了探讨。

关 键 词: 离子辐照; Fe/Nb 多层膜; AES 深度剖面分析; XRD; VSM

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