Manipulation of ordered layered structures by interface-assisted ion-beam mixing in immiscible Ag-Co and Ag-Ni systems

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Artificial ordered layered structures were grown in the nanosized Ag-Co and Ag-Ni multilayered films upon ion irradiation and were both identified to consist of two overlapping fcc lattices, corresponding to new magnetic states of ferromagnetic metals Co and Ni. Surprisingly, in the newly formed structures, the average magnetic moment per Co atom was found to be $2.84\mu_B$, much larger than ever reported before, while the average magnetic moment per Ni atom decreased to 1/3 of its equilibrium value. The driving force for growing the ordered layered structures was from a dynamic atomic collision triggered by ion irradiation together with interfacial free energy, which was properly designed to elevate the initial state of the multilayered films up to nearby the respective highly energetic nonequilibrium levels in the immiscible Ag-Co and Ag-Ni systems.

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I. INTRODUCTION

In developing new materials of high performance, a powerful way is to fabricate novel artificial microstructures or atomic configurations frequently by some nonequilibrium and even far-from-equilibrium processes, which could provide excess energy to help reaching a highly energetic state, corresponding to a nonequilibrium solid phase, in a material system of interest.¹ For instance, a scheme of ion-beam mixing (IBM) of multiple metal layers was introduced in early 1980s and has been employed to produce a great number of nonequilibrium solid phases in the binary metal systems, such as amorphous, metastable crystalline, and quasicrystalline phases.^{2,3} In the multilayered films, there are a certain number of interfaces, which possess interfacial free energy. Generally, the interfacial free energy was in positive proportion to the fraction of the interfacial atoms versus the total atoms in the films and was a controllable factor by adjusting the number of interfaces, while the total thickness of the films was fixed as constant. In recent years, the experimental scheme of IBM has been advanced: i.e., the individual layer thickness of the multilayered films was intentionally designed to be down to a couple of nanometers. Under such circumstances, the interfacial free energy could contribute significantly in elevating the initial state of the multilayered films up to a highly energetic nonequilibrium state. Meanwhile, the atomic migration length required for intermixing and alloying between the constituent metal layers was reduced considerably and actually down to a couple of nanometers. Consequently, IBM of nanosized multilayered films has resulted in forming some new nonequilibrium solid phases or microstructures in some equilibrium immiscible binary-metal systems.⁴

In this study, the Ag-Co and Ag-Ni systems were chosen to investigate the possibility of manipulating some new nonequilibrium microstructures by ion-beam mixing with an important assistance of the interfacial free energy as a controllable parameter. These two systems are essentially equilibrium immiscible and characterized by large positive heats of formation $(H_f^{\text{Ag-Co}} = +28 \text{ kJ/mol} \text{ and } H_f^{\text{Ag-Ni}} =$ +23 kJ/mol), calculated by the model of de Boer et al. at an equiatomic stoichiometry⁵ together with large size mismatches being 16% and 15%, respectively. In addition, the systems have attracted much attention, because the nanostructures of the successive Ag and Co layers could possibly featuring serve as spin valve systems giant magnetoresistance.⁶ Besides, as Co and Ni are both ferromagnetic metals, the possible magnetic property change could also provide some useful information in identifying the nonequilibrium microstructures to be formed. We report, in this paper, the growth of artificial Ag-Co and Ag-Ni ordered layered structures in the nanosized Ag-Co and Ag-Ni multilayered films upon IBM assisted by the interfacial free energy stored in the multilayered films as well as the magnetic property changes related to the growth of the nonequilibrium structures.

II. EXPERIMENTAL PROCEDURE

Based on the interfacial free energy calculation,⁷ three sets of Ag-Co (with 6, 12, and 18 layers, respectively) and Ag-Ni (with 12, 16, and 20 layers, respectively) multilayered samples were designed and prepared by depositing alternately pure Ag and Co/Ni onto NaCl single crystals in an ultrahigh-vacuum (UHV) e-gun evaporation system with a background vacuum level on the order of 10^{-11} Torr. During deposition, the vacuum level was better than 1.6 $\times 10^{-8}$ Torr. The depositing rate was controlled at 0.5 Å/s, and during deposition, no special cooling was provided to the sample holders. The total thickness of all the multilayered samples was about 40 nm, which was required to match the projected range plus projected range straggling of 200keV xenon ions employed in IBM experiment. The desired overall compositions of the Ag-Co/Ni multilayered films, i.e., Ag₈₀Ni₂₀ and Ag₅₀Co₅₀, were obtained by adjusting the relative thicknesses of the individual Ag and Co/Ni layers. After deposition, the real compositions of the multilayered films were confirmed to be Ag₇₅Ni₂₅ and Ag₅₀Co₅₀, respectively, by energy-dispersive spectrum (EDS) analysis with a

measuring error around 3%-4%. These results confirmed that the thicknesses of the Ag and Co/Ni layers in the deposited films were fairly close to the designed values. It should be noted that the thickness of the individual layer of the Ag-Co/Ni multilayered samples was about 4 nm, and therefore the atomic migration length to cross the interfaces for intermixing and alloying upon IBM was only a couple of nanometers. Some of the as-deposited films were then subjected to 200-keV xenon ion irradiation in an implanter with a base vacuum level on the order of 10^{-6} Torr. It has been reported by Cheng et al.⁸ that in the binary-metal systems with positive heat of formation, the lower the IBM temperature, the higher the mixing efficiency, because a high temperature would result in thermal excitation leading to separation between the immiscible constituent metals. Our IBM experiments were therefore conducted with 200-keV xenon ions at room temperature to irradiation doses from 5 to 90 $\times 10^{14} \text{ Xe}^+/\text{cm}^2$ for the Ag-Co system and at 77 K to irradiation doses from 5 to 9×10^{15} Xe⁺/cm² for the Ag-Ni system. The xenon ion current density was confined to be about 0.5 μ A cm⁻² for avoiding an otherwise overheating effect during xenon ion irradiation. The temperature increase during IMB was measured to be within 50° and the effect of temperature rise on the nonequilibrium structure formation was therefore minor. Meanwhile, some of the as-deposited films were removed from the NaCl substrates by de-ionized water and placed onto the Mo grids for solid-state reaction (SSR) experiments, i.e., thermal annealing of the films conducted in a hot stage attached to the transmission electron microscopy (TEM) with a base vacuum level on the order of 10^{-7} Torr. The thermal annealing procedure began at 150 °C with an increasing step of 50 °C, and the staying time at each annealing temperature was at least 30 min. Transmission electron microscopy examination and selected area diffraction (SAD) analysis were employed to characterize the microstructural change emerged in the multilayered films. The magnetic properties of the films were measured by an alternating gradient magnetometer (AGM), which has a resolution of 2×10^{-6} emu in a magnetic field of up to 5 kOe. The measuring error of AGM was about 2%. To obtain a precise value of the Co/Ni content in the films, the PLASMA-SPEC-I inductive coupled plasma atomic emission spectrum (ICP) was employed and its measuring error was about 6%. After the magnetic measurements, the films were first completely dissolved in 5 ml hydrochloric acid, and then from the acid solution the total content of Co/Ni was determined by ICP spectra. The average magnetic moments per Co/Ni atom were then calculated based on the above obtained data, and the error of the average magnetic moment per Co/Ni atom was therefore around 8%. It will be shown later that the magnetic property changes associated to the nonequilibrium structure formation would be much greater than the measuring and calculated errors.

III. RESULTS AND DISCUSSION

A. Growth of the ordered layered structures

We first examine the initial states of the as-deposited Ag-Ni/Co multilayers. Take the Ag-Ni multilayers consisting of



FIG. 1. Cross-section TEM micrograph of the original Ag-Ni multilayers with 20 layers.

20 layers as an example. Figure 1 shows a micrograph taken from a cross-section sample, and it shows clearly a layer-bylayer structure in the initial state. Figure 2 is a SAD pattern, showing the sharp diffraction lines from the polycrystalline fcc Ag and Ni, from which the lattice constants were determined to be a_{Ag} =4.07 Å and a_{Ni} =3.58 Å, respectively. Similar results were also obtained for the Ag-Co multilayered films. These results indicated that the initial states of the films were of discrete layered structures with both Ag and Co/Ni polycrystalline phases, respectively.

After IBM, Fig. 3(a) shows a SAD pattern for the Ag-Ni multilayered films (20 layers) irradiated at 77 K to a dose of 5×10^{15} Xe⁺/cm², and Fig. 3(b) shows a SAD pattern for the Ag-Co multilayered films (18 layers) irradiated at room temperature to a dose of 5×10^{14} Xe⁺/cm². One sees that both SAD patterns are composed of two sets of diffraction spots, corresponding to two overlapped lattices of both fcc structures. For the Ag-Co case, the inside spots were from a large fcc lattice with a lattice constant of a = 3.76 Å, while the outside spots were from a small fcc lattice with a = 3.27 Å. It is of interest to note that the lattice constant of



FIG. 2. SAD pattern of the as-deposited Ag-Ni multilayers with 20 layers.



(a)



FIG. 3. SAD patterns of (a) the Ag-Co multilayers with 18 layers after IBM at room temperature to a dose of $5 \times 10^{14} \text{ Xe}^{+}/\text{cm}^{2}$ and (b) the Ag-Ni multilayers with 20 layers after IBM at 77 K to a dose of $5 \times 10^{15} \text{ Xe}^{+}/\text{cm}^{2}$.

the small fcc Ag-Co lattice was smaller than those of pure fcc Ag and Co and that the size of the large fcc Ag-Co lattice was in the middle of the pure Ag and Co lattices. For the Ag-Ni case, the inside spots were from a large fcc lattice with a=4.29 Å, and the outside spots were from a small fcc lattice with a=3.80 Å. Note that the lattice constant of the large fcc Ag-Ni lattice was greater than those of pure fcc Ag and Ni and that the size of the small fcc Ag-Ni lattice was in the middle of the pure Ag and Ni. In short, the two overlapped fcc lattices were grown in the Ag-Co and Ag-Ni multilayered films upon IBM and can be regarded as artificially ordered layered structures, which correspond to the new solid states of high energy in the Ag-Co and Ag-Ni systems, respectively.

B. Average magnetic moment per Co/Ni atom in the ordered layered structures

In association with the above structural changes, significant modification of the magnetic property was observed. Table I lists the measured average magnetic moment per

TABLE I. Average magnetic moment per Ni/Co atom in the Ag-Ni/Co multilayers.

Specimen		σ_g (µemu)	Weight of Ni/Co (µg)	μ (μ_B)
Ag-Ni	as-deposited	234.4	1.990	1.15
	by IBM	70.56	1.994	0.35
Ag-Co	as-deposited	117.9	0.836	1.41
	by IBM	224.1	0.789	2.84

Co/Ni atom, μ_{Co} and μ_{Ni} , in the ordered layered structures as well as in the as-deposited states. Apparently, in the Ag-Co case, μ_{Co} was greatly increased and the measured average magnetic moment per Co, μ_{Co} was 2.84 μ_B , which was about twice of that in the as-deposited Co-Ag multilayered films. Besides, if it is compared to the equilibrium value in the bulk Co, i.e., $\mu_{Co} = 1.70 \mu_B$,⁹ the increase was about 67%. To our best knowledge, the measured average magnetic moment of Co was one of the highest values ever observed.¹⁰ The significant enhancement of the magnetic moment per Co atom could possibly be attributed to the reduction of the nearest-neighboring distance among the Co atoms in the new solid state. In contrast, in the Ag-Ni case, μ_{Ni} was greatly reduced to be approximately one-third of its equilibrium value, which can likely be correlated with the increase of the nearest-neighboring distance among the Ni atoms in the ordered layered structure. It should be emphasized that the magnetic measurements in AGM were made with an assembly of three identical specimens stacked together, and the precision of the measured magnetic moment of the films was estimated to be better than 2%. Meanwhile, an error involved in the ICP measurements was estimated to be less than 6%. Consequently, the total error for the magnetic moment per Co/Ni atom was lower than 8%, which was much less than the observed modifications in magnetic moments of both Co and Ni per atom.

C. Effects of irradiation dose and layer number on the structural change

To understand the underlying physics for growing the ordered layered structures, further IBM experiments were conducted. First, the effect of the irradiation dose on structural change was studied by irradiating the Ag-Co (18 layers) and Ag-Ni (20 layers) multilayered films to high doses up until 9×10^{15} Xe⁺/cm² and the above-observed ordered layered structures appeared at all irradiation stages, implying that the irradiation dose did not considerably affect the growth of the microstructure in the films upon IBM.

Second, to check the effect of the layer number, another two sets of the Ag-Co and Ag-Ni multilayered films with smaller layer numbers were also subjected to IBM, which resulted in completely different structural changes from the above observations. In the Ag-Co multilayered films with 12 layers, a dodecagonal phase was obtained at a dose of 5 $\times 10^{14}$ Xe⁺/cm², which was identified by SAD analysis (the SAD pattern is not shown). Increasing the dose to a range of $7-90\times 10^{14}$ Xe⁺/cm² resulted in the formation of a hcp and

System	Doses of IBM (Xe ⁺ /cm ²)	Layer number	Average layer thickness (nm)	Structure
		20	2.0	ordered layered structure
Ag-Ni	$5 \times 10^{15} - 9 \times 10^{15}$	16	2.5	hcp
		12	3.3	no change
	$5 \times 10^{14} - 9 \times 10^{15}$	18	2.2	ordered layered structure
Ag-Co	5×10^{14}	12	3.3	dodecagonal
	$7 \times 10^{14} - 9 \times 10^{15}$			hcp+fcc
	$5 \times 10^{14} - 9 \times 10^{15}$	6	6.7	no change

TABLE II. Structure changes as a function of the layer number and thickness in the Ag-Ni/Co multilayered films.

a fcc nonequilibrium Ag-Co phase. While in the Ag-Ni multilayered films with 16 layers, a structural change was observed after irradiation to a dose range of 5-9 $\times 10^{15} \text{ Xe}^+/\text{cm}^2$. A new metastable phase was formed and identified to be of hcp structure. The formation of the metastable crystalline and quasicrystalline phases by IBM has been understood quite well and will therefore not be discussed in detail here.^{2,3} As was expected, in the Ag-Co (6 layers) and Ag-Ni (12 layers) multilayered films, similar IBM did not result in any new microstructure. All the IBM results are shown in Table II, from which one can see clearly that the structural changes were indeed closely related to the layer number. In other words, the number of layers in the samples played an important role in determining what kind of nonequilibrium microstructure would turn out upon IBM. From a physical point of view, it was the amount of interfacial free energy stored in the multilayered films that played a decisive role in governing the microstructural evolution in the films. As the interfacial free energy was in positive proportion to the layer number while the total thickness of the films was fixed at a constant value, the amount of interfacial free energy was therefore a controllable parameter in ionbeam manipulation of new solid microstructures in a scheme of nanosized multilayered films.

Incidentally, as mentioned in the Introduction, for the equilibrium immiscible systems, there exists a strong repulsion force, which tends to separate the two constituent metals, and the higher the temperature, the stronger the repulsion force is. In other words, to form or grow some new nonequilibrium microstructures in these systems, IBM should be conducted at relatively low temperatures. It is therefore anticipated that if the IBM would be conducted at relatively high temperature, the possibility of obtaining the nonequilibrium solid phases would be much reduced.

D. Role of interfacial free energy in nonequilibrium structure formation

Concerning the role of interfacial free energy in helping the formation of the nonequilibrium phases or microstructures in the immiscible systems, the above discussion was only a manner of phenomenological deduction based on the design and preparation of the multilayered films. It therefore requires further or direct evidence to confirm that the interfacial free energy did play a determining role in influencing the structural changes emerged in the Ag-Co/Ni multilayered films. To clarify the issue, we conducted steady-state thermal annealing of the respective films. Such a process was a neat thermal excitation scheme and involved no complicated ion irradiation process, and therefore could provide direct evidence of the existence and action of the interfacial free energy stored in the properly designed Ag-Co/Ni multilayered films in the present study. The annealing procedure began at 100 °C with increasing steps of 50 °C, and the staying time at each annealing temperature was 30 min. Take the Ag-Co sample (12 layers) as a typical example, after annealing at 250 °C for about 30 min, a new solid phase was obtained, as evidenced by the SAD pattern displayed in Fig. 4. A careful inspection of the SAD pattern indicated that an Ag-Co dodecagonal phase was formed and embedded in the polycrystalline Ag and Co matrix, which was the same observed in the Ag-Co sample upon room-temperature IBM to a dose of 5 $\times 10^{14} \text{ Xe}^+/\text{cm}^2$ mentioned above. These results not only confirmed that the interfacial free energy did drive the interfacial reaction between the Ag and Co layers, resulting in the formation of the dodecagonal phase, but also proved that the as-deposited multilayered films in the present study did possess certain amount of interfacial free energy. As the amount of the interfacial free energy was in positive proportion to the layer number of the multilayered films,⁷ the energetic sequence of the as-deposited Ag-Co multilayered films, from



FIG. 4. SAD pattern of the Ag-Co multilayered films with 12 layers after thermal annealing at 250 °C for about 30 min.

tial free energies of the multilayered films, including the interfacial free energy in the Ag-Co and Ag-Ni systems, could readily be calculated. Taking the Ag-Co system as an example, the initial free energies of the multilayered films were calculated to be 38.0, 24.4, and 11.0 kJ/mol for the samples with 18, 12, and 6 layers, respectively. These results showed that the ordered layered structure and the dodecagonal phase were indeed in the nonequilibrium states at high free energy levels in the Ag-Co system, which was also true for the Ag-Ni system. At present, though the above calculations were based on the semiquantitative model of de Boee *et al.* and the method of Alonso *et al.*, the energetic sequence of the multilayered films as well as the observed nonequilibrium phase or structure was considered to be of relevance, at least in its outline.¹²

E. Proposed interpretation for the growing of the ordered layered structures

It is commonly known that IBM process can generally be divided into two steps: i.e., the first step of the atomic collision triggered by impinging ions and followed by a second step of relaxation.¹³ In the first step, the irradiating ions triggered a series of atomic collisions-namely, an atomic collision cascade-which was responsible for the intermixing between the Ag and Co/Ni metal layers. After receiving an adequate ion dose, a mixture of Ag-Co/Ni was obtained and it was in a highly energetic and disordered state. According to the atomic collision theory, the relaxation period is extremely short lasting only for $10^{-10} - 10^{-9}$ sec, which allowed only a limited rearrangement of the atoms in the Ag-Co/Ni mixture, and therefore the mixture could not undergo straightforward to its corresponding equilibrium state: instead, it resided in a possible intermediate state: i.e., the observed ordered layered structure in the present study.

Concerning the effect of the interfacial free energy, for the Ag-Co (18 layers) and Ag-Ni (20 layers) multilayered films, the interfacial free energy was high enough to elevate the initial energetic state of the Ag-Co/Ni multilayered films presumably a little lower than that of the Ag-Co/Ni ordered layered structure, which was formed by IBM, yet has so far not been obtained by thermal annealing. It is therefore reasonably understood that IBM could not only induce intermixing between the Ag-Co/Ni layers, but also provide some additional irradiation energy, which helped the multilayered films to reach the respective energetic level for forming the Ag-Co/Ni ordered layered-structure. While for the Ag-Co multilayers (12 layers) the interfacial free energy was of medium and presumably close to that of an amorphousquasicrystalline state,⁷ a dodecagonal phase was therefore formed not only by IBM, but also by thermal annealing.

Moreover, in the as-deposited Ag-Co (18 layers) and Ag-Ni (20 layers) multilayered films, the interfaces were in-

termediate layers consisting of atomic mixture probably in a disordered state and the intermediate laver could be a couple of atomic layers thick.¹⁴ According to Lin et al.¹⁵ and Li et al.,¹⁶ the interfacial reaction driven by the interfacial free energy could extend to about 4-5 nm for some Cu-based systems with positive heat of formation, which was confirmed experimentally by direct TEM observations.¹⁷ In the present study, the individual layer thickness in the Ag-Co/Ni multilayered films was designed to be 2-3 nm: the interfacial free energy might therefore be high enough to drive the intermixing between Ag and Co/Ni thin layers thoroughly in the films. Incidentally, it has been argued very recently that for two immiscible metals, when one metal was deposited onto the surface of another metal, intermixing could be extended around 1-2 nm through the so-called "capping" or "burrowing" mechanism.^{18,19,20} These proposed mechanisms may be of help to understand the detailed atomic motion mechanism in the nanosized metal-metal multilayers upon IBM and/or SSR in the equilibrium immiscible systems: however, much more studies are still necessary and should be done before an exact mechanism is clarified.

It should be mentioned that some other factors might also influence the formation of the metastable phase and the ordered layered structure in the Ag-Co/Ni systems, e.g., the mechanical stresses due to the size mismatch, interface roughness, substrate temperature of IBM and radiationenhanced diffusion, etc. As these possible factors emerged in all the studied multilayered films with different layer numbers, the experimental results described above evidently suggested that the interfacial free energy was a major and controllable factor in manipulation of the ordered layered structure of high energy by IBM in the immiscible Ag-Co and Ag-Ni systems.

IV. SUMMARY

In summary, we have shown that in equilibrium immiscible Ag-Co and Ag-Ni systems, interface-assisted ion-beam mixing was capable of manipulating some new solid-state microstructures, especially the ordered layered structures, corresponding to the new magnetic states of Co and Ni atoms, respectively. The amount of the interfacial free energy could be artificially designed to elevate the initial metalmetal multilayers up to a desired high-energy level and could therefore be considered as a controllable parameter in ionbeam manipulation. Besides, the significant changes in average magnetic moment of Co/Ni atoms in the respective ordered layered structures raise an interesting issue, which certainly deserves much further theoretical investigation.

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