Study of surfactant mediated growth of Ni/V superlattices

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The Ni/V multilayers are useful as soft x-ray mirrors, polarizers, and phase retarders. For these applications, it is necessary that the interfaces roughness and interdiffusion must be as small as possible. The V-on-Ni and Ni-on-V interfaces are asymmetric due to the difference in the surface free energy of Ni and V. In this work, we report Ag surfactant mediated growth of Ni/V superlattices prepared using ion beam sputter deposition technique. These superlattices were studied using x-ray and neutron scattering techniques. It was found that when added in an optimum amount, Ag surfactant results in reduced interface roughness and interdiffusion across the interfaces. Obtained results can be understood with the surfactant floating-off mechanism leading to a balance in the surface free energy of Ni and V. © 2013 AIP Publishing LLC. [<http://dx.doi.org/10.1063/1.4813135>]

I. INTRODUCTION

Artificially prepared thin film multilayers are of great interest because of underlying fundamental physics and their applications in the magnetic devices and as an optical component for x-rays and neutrons.^{1–3} The inherent advantage of such multilayers allow one to tune thickness and period covering a wide wavelength region.⁴ To achieve the optimum performance of such multilayers, the criterion of A/B (A: low-absorption element/B: an element such that high reflection at the interface) type multilayer is generally followed.^{5,6} The Ni/V multilayers are useful as soft x-ray optical components in the water window region (277–525 eV) and as polarizer/retarder.^{7–9} The water window region provides a good contrast for imaging of biological specimens and the polarizer/retarder allow the conversion of linearly polarized light into circularly polarized light, or vice versa. Realization of Ni/V multilayers as a soft x-ray mirror can only be obtained when the layer thicknesses are typically below 3 nm.^{10,11} At such thicknesses, the interface imperfections like the interface roughness and the intermixing, become very significant and affect the performance of multilayer devices due to the loss of specular reflectivity.^{12,13} Moreover, during the growth of the multilayer, A/B and B/A interfaces are not symmetric due to the inherent difference in the surface free energy (γ) of the elements.¹⁴ Hence, improvement of the interface roughness and intermixing is a very important issue in multilayer based devices. It has been found in the literature that V based superlattices consisting of Fe (Mo) can be grown with high structural quality due to the moderate lattice matching.^{15–17} Remhof *et al.* has demonstrated that the interfacial quality in the Fe/V superlattice can be improved using hydrogen as a surfactant.¹⁸

During the deposition of the multilayer, growth mode is determined by several factors, out of which the γ is an intrinsic property and plays a vital role in determining the growth mode. In general, alternate interfaces in multilayer structure are asymmetric due to the difference in the surface free energy

(γ) of the constituent element of the multilayer.^{14,19} An element with low γ will wet the surface of elements with high γ and make a smooth interface, whereas high γ element agglomerates over the low γ element surfaces and make a rough interface. Such asymmetry of the interface roughness is always present during the growth of the multilayer, no matter what deposition technique is used. In Ni/V multilayers polycrystalline V ($\gamma = 2.6 \text{ Jm}^{-2}$) has somewhat larger γ than polycrystalline Ni ($\gamma = 2.4 \text{ Jm}^{-2}$) which might lead to rough V-on-Ni and smooth Ni-on-V interface.^{20,21} As such the difference between γ of Ni and V is small, still when depositing large number of Ni/V repeats, even this difference can be significant as obtained in case of Ni/Ti multilayers ($\gamma_{\text{Ni}} = 2.4 \text{ Jm}^{-2}$ and $\gamma_{\text{Ti}} = 2.1 \text{ Jm}^{-2}$).³ A novel and simple way of inducing a layer by layer growth with smooth and symmetric interface is the addition of surfactant into the multilayer.^{22–24} Introduction of third element known as surfactant minimizes the difference of the γ of the elements and induce layer by layer growth.^{24–30} Various types of surfactants, e.g., Ag ($\gamma = 1.2 \text{ Jm}^{-2}$), Sb ($\gamma = 0.6 \text{ Jm}^{-2}$), Bi ($\gamma = 0.6 \text{ Jm}^{-2}$), Pb ($\gamma = 0.6 \text{ Jm}^{-2}$), etc. (γ values for polycrystalline case) have been used in different types of multilayers.^{3,25,31–35} It has been demonstrated that surfactant floats at the surface balancing the γ of the elements, suppress surface diffusion and prevents island formation.^{18,36–40} To act as a good surfactant an element must have relatively smaller γ and larger volume which promotes layer by layer growth and inhibits its incorporation. Trapping of surfactant atom into the underlayer during the growth of the multilayer is avoided due to the cost of extra energy.³⁰ The thickness of Ag or Pb surfactant to act as a good surfactant have been reported to be about a monolayer (ML) in Cu/Co and Ni/Ti multilayers.^{3,25,35}

In this work, we have used Ag ($\gamma = 1.2 \text{ Jm}^{-2}$) as a surfactant and studied its effect on interface quality and thermal stability in Ni/V multilayers. Since surfactant mediated growth of Ni/V multilayers is not reported earlier, in order to optimize the thickness of Ag we have deposited Ni/V multilayers with different ML thickness of Ag surfactant. Without surfactant and with optimized thickness of Ag surfactant Ni/V multilayers have been studied using x-ray diffraction,

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synchrotron x-ray, and neutron reflectivity techniques. Reflectivity is non destructive technique and measures the interface roughness and interdiffusion with Ångström level accuracy. It was found that presence of Ag surfactant in Ni/V multilayers improves the interfacial quality and reduces interdiffusion.

II. EXPERIMENTAL

Ion beam sputtering (IBS) technique was used to deposit Ni/V multilayers with Ag surfactant on Si (100) substrate. An identical sample without Ag surfactant was also prepared for comparison. Ar^+ ions of energy of 1 K eV were produced using radio frequency (rf) ion beam source (Veeco 3 cm rf-source). Ni, V, and Ag targets (purity 99.999%) were mounted on a rotary motion feedthrough (to switch over from one to another target), which can hold up to four different targets at a time. Ar^+ beam was kept incident at an angle of 45° with respect to the target surface. The base pressure in the chamber prior to the deposition was about 5×10^{-8} mbar and during the deposition pressure was about 5×10^{-4} mbar due to the flow of 4 sccm Ar gas (purity 99.9995%). The targets were sputtered at room temperature, alternatively to deposit multilayer structure. The deposition rates achieved were 2.2 nm min^{-1} , 1.6 nm min^{-1} , and 6 nm min^{-1} for Ni, V, and Ag, respectively. The nominal structure of the multilayer is $\text{Ni (10 nm)}/x/[\text{Ni (3 nm)}/\text{V (3 nm)}]_{10}$ with $x=0$ (without surfactant) or $x \approx 0.5 \text{ ML}$, 1 ML , and 2 ML Ag (with surfactant). We have deposited relatively thick Ni/V multilayers than normally used in soft x-ray mirror applications so that Bragg peak in the neutron reflectivity pattern appear at lower angles.

The X-ray diffraction (XRD) measurements were carried out using 1.54 Å x-rays in the θ - 2θ geometry using a standard diffractometer (Bruker D8 Advance) equipped with a fast 1D detector based on silicon drift technology (Bruker LynxEye). The unpolarized neutron reflectivity (NR) measurements were performed at NARZISS reflectometer at SINQ/PSI, Switzerland using neutrons of wavelength 5 Å . In order to avoid the effect of randomly oriented magnetic domains in Ni/V multilayers under zero magnetic field, 800 G of magnetic field was applied parallel to the sample surface, which is sufficient to saturate the sample magnetically at room temperature.⁴¹ In addition to NR measurements, the polarized neutron reflectivity (PNR) measurements were performed under the same magnetic field. The direction of magnetic field was kept parallel to the surface of the samples. Further, synchrotron x-ray reflectivity (XRR) measurements were also performed to probe the interface structure. Since laboratory source x-ray ($\text{CuK}\alpha$) contrast for Ni and V is rather poor, synchrotron XRR measurements were done at Indian beamline BL-18B, photon factory, Japan at photon energy of 7 keV.

III. RESULTS AND DISCUSSION

A. Optimization of Ag surfactant

As mentioned already, Ag with a very small surface free energy and a larger atomic volume than Ni and V makes it a

suitable element to act as a surfactant during the growth of Ni/V multilayers. Since till now surfactant mediated growth of Ni/V multilayers has not been investigated and such an investigation will help to optimize the necessary amount of Ag surfactant for improving the quality of such systems. Typically, it has been reported for various other multilayer structures, which around the thickness of about 1 ML, addition of surfactant yield the best results.^{3,25,35} Therefore, to optimize the amount of Ag surfactant, we added about 0.5 ML, 1 ML, and 2 ML of Ag surfactant during the growth of the multilayer and also prepared a sample without surfactant for comparison. Fig. 1 shows neutron reflectivity pattern of these samples. Bragg peak appearing around momentum transfer vector, $q_z = 0.096 \text{ Å}^{-1}$ ($q_z = 4\pi \sin \theta / \lambda$; θ is the incidence angle and λ is the wavelength of neutrons) results due to the constructive interference of neutrons reflected from each interface, i.e., Ni-on-V and V-on-Ni in this case. Intensity of the Bragg peak is determined by scattering length density (SLD) contrast of the elements (here Ni and V) and interface sharpness. It may be noted that nominal structure of the Ni/V multilayers prepared with and without Ag surfactant are same except a small amount of Ag surfactant is placed at the bottom of the multilayer. Under the Born approximation reflectivity from the multilayer is given by⁴²

$$R(q_z) = R_0(q_z)e^{-q_z^2 \sigma^2}, \quad (1)$$

where R is the reflectivity of the multilayer, σ is the average interface roughness, and R_0 is the reflectivity of the multilayer with perfectly smooth interfaces. The exponential term is Debye-Waller type factor, which takes into account the decay in the reflectivity with interface roughness. As can be seen from Fig. 1, the intensity of the Bragg peak is higher for the sample deposited with 1 ML of Ag surfactant than the samples deposited without surfactant or with 0.5 ML and 2 ML Ag surfactant. This indicates the sharper interface of Ni/V is obtained with addition of 1 ML of Ag surfactant. Inset of the Fig. 1 compares the reflectivity at Bragg peak after subtracting the background due to Fresnel reflectivity which decays with

FIG. 1. NR pattern of as-deposited Ni/V multilayers deposited with different amount of (0 ML, 0.5 ML, 1 ML, and 2 ML) Ag surfactant. Inset compares the reflectivity at Bragg peak.

q_z^{-4} power law behavior. An enhancement in Bragg peak reflectivity for the sample with 1 ML of Ag surfactant reflects the optimized amount of Ag to act as a surfactant in Ni/V multilayer. However, the reflectivity in Ni/V multilayer deposited with 0.5 ML and 2 ML of Ag are comparable to the sample prepared without surfactant. The quantitative information of the difference in the roughness of different interfaces in the sample prepared with (1 ML Ag) and without Ag surfactant Ni/V multilayer is discussed in Secs. III B–III D.

B. X-ray diffraction

Fig. 2 shows the XRD pattern of as-deposited Ni/V multilayers prepared with and without Ag surfactant. XRD pattern shows a textured growth about Ni (111) orientation as fundamental peak of the multilayers. Observed pattern suggest that Ni/V has grown as a superlattice. One or more satellite peaks on either side of (111) fundamental peak can be clearly seen in Fig. 2. The crystallite size was calculated from the full width at half maxima (FWHM) of the fundamental peak using Scherrer formula.⁴³ It was found that crystallite size for the sample prepared with Ag surfactant (17.6 nm) is slightly larger than the sample prepared without surfactant (16.2 nm). It may be noted that size of crystallite is more than two times the nominal period of the multilayer which clearly indicates the strong structural coherence of Ni and V layers resulting in superlattice structure. It is well known that in superlattice structure satellite peak depends on thickness fluctuation or composition gradient at the interface.^{44–46} Interface roughness in the multilayer are nothing but the random distribution of atoms due to island formation and intermixing at the interface during deposition. Inset of the Fig. 2 compares the relative intensity of the satellite peaks. In literature, it has been established that sharper interface results in larger satellite peak.^{17,47,48} A qualitative information about the interface structure may be obtained by evaluating the relative intensity (RI) using the equation⁴⁸

$$RI = (I_{-1} - I_{back}) / (I_0 - I_{back}), \quad (2)$$

FIG. 2. XRD pattern of as-deposited Ni/Vi superlattices deposited with and without Ag surfactant. Scattered points are measured data and solid lines are fit to them. Inset compares the intensity of the superlattice peaks.

where I_{-1} is the intensity of first order satellite peak at lower angle with respect to the fundamental peak I_0 , and I_{back} is the intensity of the background. RI of the samples prepared with and without Ag surfactant were found to be 0.51 ± 0.01 and 0.47 ± 0.01 , respectively, which indicates the sharper interface with the addition of Ag surfactant.

In order to get more detailed information, XRD patterns were fitted using superlattice refinement from the x-ray-diffraction computer programme (SUPREX) developed by Fullerton *et al.* under kinematical formalism.¹⁵ In SUPREX individual layer is considered by number of atomic planes separated by lattice spacing. During the fitting we have taken standard A/B type superlattice with a crystalline/crystalline model. The obtained parameters from the fitting of the SUPREX are number of atomic planes (N), interplanar distance (d), and discrete interface roughness (σ), which is actually thickness fluctuation in term of ML, rest all other parameters were kept constant. Position of the superlattice peaks depend on the period of the multilayer which is sensitive to the parameter N and d , while σ controls the intensity and broadening of the peak. Fitted parameters are given in Table I. It may be seen from the Table I that values of σ_V and σ_{Ni} are significantly different for the sample prepared without surfactant, while it becomes almost equal for the sample prepared with Ag surfactant. This clearly indicates that addition of Ag surfactant has induced layer by layer type mode growth and asymmetric fluctuation in the layer thickness due to the difference in the γ of Ni and V, during the growth of the Ni/V superlattice is suppressed.

C. Neutron and X-ray reflectivity

In order to obtain precise determination of interface structure and magnetic properties of Ni/V superlattices NR, PNR, and XRR measurements were performed on the samples prepared with and without Ag surfactant. As mentioned above, to increase the SLD of Ni and V, synchrotron radiation was used to perform XRR measurements. XRR can measure reflectivity up to higher q_z (momentum transfer vector) value, which is limited in NR due to poor flux of neutrons.

Fig. 3(a) shows the NR pattern and its fit of as-deposited Ni/V superlattices prepared with and without Ag surfactant. NR patterns were fitted using a computer software SimulReflec.⁴⁹ During the fitting of the NR data, bilayer period (ℓ) and interface roughness (σ) were varied and rest of the parameters were fixed equal for both the samples prepared with and without Ag surfactant. The SLD of Ni and V layers were kept fixed at their nominal theoretical values. However, thin layer of silver oxide of 0.5 nm was added on

TABLE I. Bilayer period (ℓ), interplanar spacing (d), and discrete interface roughness (σ) obtained from fitting of the XRD data of as-deposited Ni/V superlattices prepared with and without Ag surfactant using SUPREX programme.

Sample	ℓ (± 1 Å)	d_{Ni} (± 0.01 Å)	d_V (± 0.01 Å)	σ_{Ni} monolayer	σ_V monolayer
No Ag	65	2.04	2.15	1.68	2
with Ag	64	2.04	2.14	1.72	1.76

FIG. 3. Neutron reflectivity (a) and polarized neutron reflectivity (b) pattern of as-deposited Ni/V superlattices deposited with and without Ag surfactant. The pattern on y-axis has been shifted for clarity. Inset shows the reflectivity at Bragg peak in neutron reflectivity.

the top of the superlattice to fit the sample prepared with Ag surfactant. The obtained fitting parameters are given in Table II. As can be seen from the table, V-on-Ni and Ni-on-V interface roughness become almost equal with addition of Ag surfactant. In order to see the effect of Ag surfactant on magnetic moment in Ni/V superlattices, PNR measurements were performed under the saturation magnetic field. Fig. 3(b) shows the PNR pattern and its fit of the sample prepared with and without Ag surfactant. PNR probes precise information about the absolute magnetic moment per atom in a magnetic sample, independent of substrate magnetism (often paramagnetic or diamagnetic).⁵⁰ This makes PNR a unique technique to measure absolute magnetic moment in magnetic samples. Fitting of the PNR patterns were also performed using SimulReflec.⁴⁹ It was found that Ni magnetic moment in both the samples prepared with and

TABLE II. Bilayer period (ℓ) and interface roughness (σ) obtained during the fitting of the NR and XRR data of as-deposited Ni/V superlattices prepared with and without Ag surfactant.

	Sample	ℓ nm (± 0.1)	$\sigma_{\text{Ni-on-V}}$ nm	$\sigma_{\text{V-on-Ni}}$ nm	$\sigma_{\text{substrate-film}}$ nm
NR	No Ag	6.4	0.72 ± 0.05	1.15 ± 0.05	0.5 ± 0.05
	with Ag	6.5	0.74 ± 0.05	0.8 ± 0.05	0.5 ± 0.05
XRR	No Ag	6.4	0.6 ± 0.1	1.2 ± 0.1	0.5 ± 0.1
	with Ag	6.4	0.6 ± 0.1	0.7 ± 0.1	0.5 ± 0.1

without Ag surfactant is $0.4 \mu_B$. This indicates that Ag surfactant has no significant influence on the magnetic moment of Ni, as expected.

Fig. 4 shows the XRR pattern of the samples. Here, again an enhancement of the reflectivity at the Bragg peak is a measure of improvement of the interfaces with addition of Ag as surfactant. Fitting of the XRR patterns were done using Parratt's formalism.^{51,52} Fitting parameters are given in Table II. It can be seen from the table that V-on-Ni and Ni-on-V interface roughness are indeed significantly different while it becomes almost equal for the sample prepared with Ag surfactant. Asymmetry in the interface roughness of the sample prepared without surfactant is due to the difference in the γ of V and Ni. When Ni atoms are deposited on the surface of V, it wets the surface of V as $\gamma_V > \gamma_{\text{Ni}}$. On the other hand, when V is deposited on the surface of Ni, it will agglomerated on Ni surface. Hence during the growth of the multilayer higher γ of V leads to rough V-on-Ni interface whereas low γ of Ni results in relatively smooth Ni-on-V interface. In case of the sample prepared with Ag surfactant, very low γ of Ag (1.2 Jm^{-2}) than both V (2.6 Jm^{-2}) and Ni (2.4 Jm^{-2}) helps in removing the asymmetry of the interface roughness. In this condition, when the growth of the multilayers takes place, the upcoming atoms basically see the lower γ of surfactant atoms instead of the other element of the multilayer. This leads to wetting of the upcoming layer. If the surfactant atoms float off to the surface, the deposition of the next element will also see the γ of the surfactant. In this situation, the layer-by-layer type growth is induced, resulting in a symmetric interface in a multilayer.^{3,22,36} Hence, symmetric interface roughness of V-on-Ni and Ni-on-V were obtained due to the balance of the γ of Ni and V with the presence of Ag surfactant. Interface roughness obtained with XRR and NR data is almost similar and correlates well with those obtained from XRD measurements.

D. Thermal stability of Ni/V superlattices

Thermal stability of the Ni/V superlattices was studied by performing XRD and NR measurements on samples

FIG. 4. XRR pattern of as-deposited Ni/V superlattices deposited with and without Ag surfactant.

annealed simultaneously at different temperatures in vacuum furnace (base pressure $\sim 1 \times 10^{-6}$ mbar or better) for 1 h at each temperature. Fig. 5 shows the XRD pattern of Ni/V superlattices after annealing it at 473 K, 573 K, and 623 K along with its fitting. The samples annealed at 473 K were fitted assuming crystalline/crystalline model as done for as-deposited samples. There is no significant change after annealing the samples at 473 K except small increase in the discrete interface roughness. After annealing the samples at 573 K a new peak about $2\theta = 32^\circ$ (shown as star) was found for the sample prepared without surfactant which get enhanced at further annealing of 623 K. The new peak around $2\theta = 32^\circ$ may occur due to metastable alloy formation triggered by intermixing due to the excess γ at the interface. It is interesting to note that no such peak was observed for the sample prepared with Ag surfactant even at 623 K which may be due to the minimization of the difference in γ of V and Ni with addition of Ag surfactant. Such type of intermixed metastable phase in Cu/Co multilayers due to the difference in the γ of Cu (1.8 Jm^{-2}) and Co (2.55 Jm^{-2}) have been observed previously^{25,26,53} while no such phase was observed when Ag surfactant was added during the deposition.

Fitting of the samples annealed at 573 K and 623 K were done using interdiffusion model (taking A-B and B-A interface width equal) in SUPREX programme.¹⁵ Fitting of the samples annealed at 573 K, interface width was varied keeping all other parameters fixed. It was observed that interface width is about 2.2 ML and 3.7 ML for the samples prepared with and without Ag surfactant, respectively. Further, at 623 K interface width for the samples prepared with and without Ag surfactant was found to be about 3 ML and 5 ML, respectively, but interplanar spacing of V and Ni atomic planes are also changing. In order to get the interdiffusion length precisely at the interface, NR was performed on the fresh samples annealed again at 573 K, which is an onset of the appearance of intermixed peak in the sample prepared without surfactant. Fig. 6 shows the NR pattern of the samples prepared with and without Ag surfactant in as-deposited state and after annealing at 573 K. Reduction in the reflectivity at the Bragg peak may be quantified in terms of the interdiffusion length at the interface using the expression^{54,55} $I(t) = I(0) \exp(-8\pi^2 D t / \ell^2)$, here $I(0)$ is the

FIG. 5. XRD pattern of as-deposited and annealed Ni/V superlattices deposited with and without Ag surfactant. Scattered points are measured data and solid lines are fit to them. Scales are same for each graph.

FIG. 6. NR pattern of as-deposited and annealed Ni/V superlattices deposited with and without Ag surfactant. Inset shows the comparison of Bragg peak in linear scale.

intensity before annealing and $I(t)$ is the intensity after annealing time t at temperature T , ℓ is the bilayer periodicity. The diffusion coefficient (D) is obtained using this equation and inter diffusion length (L) can be calculated with the expression $L^2 = 2Dt$ in the direction normal to the surface of the sample.⁵⁶ Diffusion coefficient for the samples prepared with and without Ag surfactant was obtained about $9.76 \times 10^{-5} \text{ nm}^2/\text{s}$ and $2.21 \times 10^{-4} \text{ nm}^2/\text{s}$, respectively. It may be noted that diffusion coefficient reduces significantly with addition of Ag surfactant. The interdiffusion length for with and without Ag surfactant samples were calculated to be about $0.84 \pm 0.01 \text{ nm}$ and $1.26 \pm 0.01 \text{ nm}$, respectively. This result clearly shows that addition of Ag surfactant reduces the intermixing at the interface of the Ni/V superlattices.

IV. CONCLUSION

In this work, we have deposited Ni/V superlattice structure with and without Ag surfactant. It was observed that addition of Ag surfactant in Ni/V superlattice results in increased (neutron and x-ray) reflectivity at Bragg peak which has important implication in x-ray mirror applications. Thermal stability measurements show that addition of Ag surfactant results in suppression of interdiffusion and improved thermal stability.

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