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Interface engineering of short-period Ni/V multilayer X-ray mirrors

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Abstract

Low-energy ion-assisted magnetron sputter deposition has been used for the synthesis of highly reflective Ni/V multilayer soft X-ray mirrors. A low ion energy and a high ion-to-metal flux ratio were employed in order to stimulate the adatom mobility while minimizing ion-induced intermixing at the interfaces. An analytic model, based on the binary collision approximation, was used in order to gain insight into low-energy ion–surface interactions as a function of ion energy and ion-to-metal flux ratio. The model predicted a favorable region in the ion energy-flux parameter space where only surface atomic displacements are stimulated during growth of Ni and V for multilayers. For a series of Ni/V multilayer mirrors with multilayer periods about $\Lambda = 1.2$ nm, grown with a continuous ion assistance using energies in the range 7–36 eV and with ion-to-metal flux ratios $\Phi_{\mathrm{Ni}} = 4.7$ and $\Phi_{\mathrm{V}} = 20.9$, specular and diffuse X-ray scattering analyses revealed that ion energies of ~27–31 eV produced the best trade-off between reduced interfacial roughness and intermixing. However, it was also concluded that an interface mixing of about ±1 atomic distance is unavoidable when a continuous flux of assisting ions is used.

To overcome this limitation, a sophisticated interface engineering technique was employed, where the first 0.3 nm of each layer was grown with a high-flux low-energy ion assistance and the remaining part was grown with a slightly higher ion energy. This method was demonstrated to largely eliminate the intermixing while maintaining the smoothening effect of ion assistance. Two Ni/V multilayer soft X-ray mirror structures, one with 500 periods designed for near-normal incidence and one 150 periods reflecting polarizer at the Brewster angle, were grown utilizing the interface engineering concept. Both the near-normal incidence reflectivity as well as polarizability were improved by a factor of 2 as compared to previously reported data for an X-ray energy of $E = 511$ eV.

Keywords: Nickel; Multilayer; Vanadium; X-ray mirrors; Sputtering

1. Introduction

The continuously increasing understanding of thin film nucleation and growth has lead to many devices where multilayers with ultrathin (0.5–2 nm thick) layers are indispensable for the performance. Hard drive read heads, quantum well lasers, and high electron mobility transistors are a few state-of-the-art examples of technology that would not exist without the ability of growing multilayers with extremely thin, smooth, and abrupt interfaces. The first application of synthetic multilayers was as Bragg reflectors for X-rays. Such X-ray multilayer mirrors have been used as grazing incidence mirrors in many applications like telescopes, hard X-ray optics, spectrometers, etc. However, normal-incidence Bragg reflectors have the potential to be used as optical elements in instrumentation utilizing normal-incidence mirror designs. Imaging instruments based on such designs can have up to three orders of magnitude higher optical resolution than a corresponding grazing incidence design. This has been fully taken advantage of in the design of the next generation lithography steppers for IC fabrication that will utilize a cascade of aspheric normal-incidence multilayer mirrors operating with extreme ultraviolet radiation ($\lambda = 13$ nm, ~95 eV). Also for applications in the soft X-ray spectrum ($\lambda = 1–10$ nm, $\sim E = 100–1000$ eV), such as soft X-ray microscopes, deep space telescopes, spectrometers, optics for free-electron lasers and higher harmonic generation attosecond laser spectroscopy, there is a driving force to implement near-normal multilayer

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mirrors. However, the progress in finding ways of making normal-incidence multilayer X-ray mirrors with any appreciable reflectance for soft X-rays has been very slow due to an extreme sensitivity to interface imperfections when the X-ray wavelength approaches atomic dimensions. Since normal-incidence Bragg reflectors utilize interface spacings of about 1/4 of the reflected light wavelength, it follows that the required layer thicknesses in the soft X-ray domain fall in the range [0.25–2.5 nm] and it is evident that interface imperfections, like roughness or intermixing, will have increasingly detrimental effects on any multilayer mirror performance for X-ray energies close to the upper limit of the range.

Despite these difficulties, small but non-zero near-normal multilayer reflectances have been reported for energies as high as 775 eV ($\lambda = 1.6$ nm) [1]. In order to obtain very smooth interfaces that minimize the diffuse scattering from the mirrors, it is common to utilize ion-assisted deposition, either by post deposition ion polishing of each layer utilizing a separate ion-gun [2] with ion energies of a couple of hundred electron volts, or by applying an ion flux during deposition in order to assist the deposited atoms to reach local minimum energy positions [3–6]. It is desired that each deposited adatom is given the possibility to diffuse to such positions in order to minimize the roughness of the growing film.

In most reports utilizing ion-assisted magnetron sputter deposition, the used ion energies and their fluxes are not stated. The deposition process is usually optimized with respect to a negative substrate bias V_s that attracts the ions from the plasma. The optimal bias is often found to be several hundred volts and since the plasma, hosting the ions, usually is at a potential of a few electron volts, the ion energy can be considered to be approximately qV_s, where q is the unit charge and the valency of the ions is assumed to be unity. This crude treatment is often adequate for relatively thick films where the detailed atomic processes occurring at or just below the surface are not of direct importance. However, a concurrent low-energy (<65 eV) ion flux during sputter deposition has been shown to create an undesired intermixing of the interfaces in Cr/Sc multilayer mirrors designed for an X-ray wavelength of 3.374 nm, i.e. with layer thicknesses <1 nm along with the desired smoothening effect [5]. In order to minimize the intermixing, much effort was spent on minimizing the ion energy to below 30 eV which, in turn, has called for higher ion-to-metal flux ratios (>1) [4,7].

In the first part of the present work, we have developed the model of ion–surface interactions of Brice et al. [10] which is based on the binary collision approximation to predict and explain the experimentally observed interface development in ion-assisted growth of Ni/V multilayer mirrors with layer thicknesses as thin as 2–4 atomic distances. In the second part of the article, we demonstrate how the interfaces can be engineered by implementing a novel deposition process where the ion energies are varied within each of the individual layers. The interface engineering concept is demonstrated by the growth and characterization of two highly reflecting Ni/V mirror structures optimized for maximum reflectivity at near-normal incidence and at the Brewster angle, respectively, for an X-ray energy corresponding to the V *2p* absorption edge ($E = 511$ eV).

2. Theoretical considerations

As an ion penetrates into a medium it immediately starts to interact with it and different mechanisms of energy and momentum transfer take place. The most important effects are electronic excitation/ionization, generation of plasmons and phonons, displacement of atoms, sputtering of surface atoms and emission of secondary electrons [8]. These interactions can be divided into nuclear (elastic) collisions and electronic excitations (inelastic), and are characterized by the respective scattering cross-sections. The total energy loss per unit path length, or stopping power, of the incoming projectile can be written as [9]

$$-\left(\frac{\mathrm{d}E}{\mathrm{d}z}\right) = S_\mathrm{n}(E) + S_\mathrm{e}(E), \tag{1}$$

where $S_\mathrm{e}(E)$ is the energy loss rate to electronic excitations and $S_\mathrm{n}(E)$ is the nuclear energy loss. If sequential binary collisions are assumed the amount of energy, causing a target atom displacement, that one ion transfer to the surface $0 \le z \le z_\mathrm{s}$ and the underlying bulk $z > z_\mathrm{s}$ is given by [10–12]

$$E_\mathrm{s}\left(E, E_\mathrm{d}^{(\mathrm{s})}\right) = \int_{E(z_\mathrm{s})}^{E} \frac{S_\mathrm{d}\left(E', E_\mathrm{d}^{(\mathrm{s})}\right)}{S_\mathrm{n}(E') + S_\mathrm{e}(E')} E', \tag{2}$$

and

$$E_\mathrm{b}\left(E, E_\mathrm{d}^{(\mathrm{b})}\right) = \int_{0}^{E(z_\mathrm{s})} \frac{S_\mathrm{d}\left(E', E_\mathrm{d}^{(\mathrm{b})}\right)}{S_\mathrm{n}(E') + S_\mathrm{e}(E')} E', \tag{3}$$

respectively. E and $E(z_\mathrm{s})$ are the energies of the ion initially and at a distance z_s normal from the target surface; $S_\mathrm{d}(E, E_\mathrm{d})$ is the energy loss correlative to the atomic displacement, and $E_\mathrm{d}^{(\mathrm{s})}$ and $E_\mathrm{d}^{(\mathrm{b})}$ are the required energies to displace one atom on the surface and in the bulk, respectively. The remaining part of the ion energy, which is not lost to displacements, is transferred to electronic excitations and also dissipates to the "lattice" as phonons (heat).

The average energy loss when an ion travels a distance dz in a material with atomic density N is obtained by integrating the product of energy transfer to a recoil, T, and the differential energy scattering cross-section, σ_s, over all possible values of T, i.e.

$$S_\mathrm{d}(E, E_\mathrm{d}) = N \int_{T_\mathrm{min}}^{T_\mathrm{max}} T\sigma_\mathrm{s}. \tag{4}$$

The energy transfer is a function of the incident ion energy and the maximum energy transfer occurs for a head-on collision with the target atom. It can be derived assuming an elastic collision and from the conservation of energy and momentum that

$$T_\mathrm{max} = \gamma E = \frac{4M_1M_2}{(M_1 + M_2)^2} E, \tag{5}$$