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Performance of multilayer coatings in relationship to microstructure of metal layers. Characterization and optical properties of Mo/Si multilayers in extreme ultra-violet and x-ray ranges

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Abstract
Here we present a development of multilayer-based optics for the extreme ultra-violet (EUV) and x-ray range. The authors discuss results of the study of optical performance of Mo/Si coatings with regard to the microstructure of metal layers, which was analysed by x-ray diffraction (XRD) and by transmission electron microscopy (TEM). We have demonstrated an ability to control the microstructure of the multilayers via optimization of the deposition process, which is important for various EUV and x-ray applications of multilayer optics.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

Scientific, industrial and medical applications of high-energy radiation (EUV, soft and hard x-ray ranges) often require optical elements analogous to those utilized in the visible range. These are reflective and diffractive (rather than refractive) focusing optics, interference filters, beam splitters, polarizers, etc. Use of multilayer optics for the EUV/x-ray range was proposed more than 30 years ago [1]. The multilayer coatings are normally periodical structures made by alternative deposition of two or more materials with different optical indices. Expected optical properties of multilayers can be calculated by applying various algorithms based on the theory of x-ray light reflectivity [2]. Such an artificial structure can provide significant reflectance at near-to-normal incidence in the EUV/soft x-ray range due to constructive interference of signals reflected by each interface in accordance with Bragg's law [3, 4]. However, in the case of a multilayer, the Bragg formula must be corrected, taking into account the mean value of the refractive index [2, 5].

Multilayer coatings onto both sides of a thin membrane can make an interference filter of Fabry–Perot type working also in beam-splitting mode in the EUV and soft x-ray range [6, 7]. In the same range, phase retarding properties of transmission multilayers have been predicted and first studied [8].

Relatively high reflectivity in the hard x-ray range can be obtained by using multilayer-coated mirrors at grazing angles. Appropriately graded multilayer coatings were proposed for focusing or collimating of neutrons and x-ray radiation [9–12]. A random or systematic variation of the multilayer period can be used to optimize the reflectivity of the mirrors in an enlarged spectral range [13–15].

One can fabricate the selective and focusing diffraction optics for the EUV/x-ray range (such as diffraction gratings, Bragg–Fresnel zone plates etc) by consequent etching of a multilayer deposited on a substrate of a special shape and profile [16–18]. Furthermore, the ion implantation applied to multilayers can modify optical properties of implanted areas, thus making optical index gratings and various optical elements of a nano-sized 3D structure [19].

2. Technique of deposition

We use the rf (radio frequency) magnetron sputtering process for deposition of the Mo/Si multilayer coatings. This low-energy deposition technique is recognized for its reliability in preparation of high-quality multilayer coatings for the EUV and x-ray applications [20]. One can find a complete description of the technique and the deposition systems [21]. The sputtering system at L2MP provides for the control of the layer thickness on up to 300 mm diameter substrates with an accuracy responding to optical requirements for the EUV lithography. A rotating substrate holder passes in front of two targets with a certain velocity programmable for any angular segment in order to provide desired profile of the multilayer period. In addition, the use of an appropriate mask placed in between of a cathode and the substrate holder can ensure the deposition of graded multilayers. Both the shape of the mask and its position are important for the required gradient [22]. The substrate holder can be kept at different temperatures in the range from zero up to 60 °C.

3. Characterization methods

Basic methods, which are usually applied for characterization of multilayers, employ the EUV/x-ray reflectivity, x-ray diffraction (for crystalline materials) and various combinations of scanning near-field microscopy. The application of these techniques makes possible a correlation of the multilayer reflectivity in a spectral range of interest with intrinsic parameters of the multilayer such as the period, individual layer thickness, number of layers, interfacial and surface roughness and with the microstructure of materials within layers and intermixing areas. Since a complete set of these characteristics determines the performance of a multilayer as an optical element, their knowledge and, particularly, control becomes important for optimization of a multilayer coating throughout the deposition process.

The high resolution TEM measurements were performed on a Philips CM30/ST electron microscope at CEMES with the point resolution of 0.19 nm.

A conclusion on the influence of the multilayer microstructure on optical quality of the EUV mirrors could only be drawn if the reflectance at near-to-normal incidence is measured. The experimental run dedicated to characterization of the multilayer optics was performed at the BACH beamline of Elettra (Sincrotrone Trieste). We used the Elettra soft x-ray polarimeter (six axes) as an instrument for the EUV reflectivity and transmission measurements in the energy range from about 85 up to 115 eV. The Elettra polarimeter is identical to that of BESSY (Berlin), whose description one can find elsewhere [23].

4. The microstructure and EUV/x-ray reflectivity of the Mo/Si multilayers

Why is the actual performance of multilayer-based optical elements always diminished compared to that predicted by calculations? How does the microstructure of the multilayers govern their optical properties? Let us list the main structural factors consequent to the multilayer deposition process that may result in an additional loss of intensity due to scattering of the light passing through non-uniform regions of a multilayer:

- a variation of the layer thickness in the substrate plane and in the direction of the multilayer growth;
- lateral and vertical strains, dislocations and other defects caused by stress during the deposition process and by impurities;
- an interfacial and surface roughness of the 'substrate + multilayer' system;
- a presence of intermixing (interdiffusion) zones between layers, whose size depends on the nature of materials, their sputtering conditions and the order of deposition;
- the real state of deposited materials or, generally speaking, the microstructure of the multilayer stack, which predetermines the density and, hence, the real optical index of the material within a thin layer (the latter could be smaller than the value for the bulk material taken from x-ray tables and generally used in calculations).

Several extensions to the theory of the multilayer mirrors have been developed. They consider, for instance, the relaxed multilayers containing misfit dislocations, lateral and vertical strains, as well as the surface and interface roughness and asymmetry [24–26]. Knowledge of the microstructure and a better understanding of how it influences the optical properties of multilayers would allow one to optimize the deposition process and to bring the theory closer to the experimental practice.

To a certain extent, the morphology of thin films depends on distinctive features of a deposition process such as the flux and the kinetic energy of film-forming particles, which can vary in a very wide range. As a result, different thermalization dynamics of condensing particles can influence the layer-by-layer growth conditions, thus leading to variations in the microstructure of layers, the state of the interface between two neighbour layers and the degree of their intermixing.

In sputtering, such film properties as density, stress and surface roughness can be tuned via the energy of adatoms and the substrate temperature. An adsorption of low energy ($\leqslant$10 eV) particles generally results in amorphous layers. On the other hand, the kinetic and potential energies of condensing atoms contribute to what is called the atomic scale heating. It proved to have a significant effect on the film texture if it is greater than the binding energy and activation energy for surface diffusion [27]. It is well known that crystallization of the metal layer may affect the optical properties of the metal–dielectric system at the expense of the physical roughness increase [28].

From the results of a number of published works, which concern the microstructure of Mo/Si multilayers (see, for example [28–34], and references herein) one can deduce some general features inherent in the multilayers regardless of the fabrication technique:

- Si is normally in amorphous state;
- Mo is either in amorphous or polycrystalline state (or both phases are present);
- crystallization of Mo starts when the thickness of the metal layer exceeds some critical value, which varies with the deposition conditions;

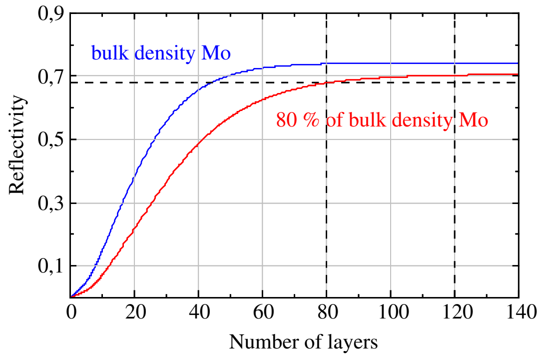

Figure 1. The normal incidence EUV reflectivity ($\lambda = 13.5$ nm) of the Mo/Si multilayer calculated as a function of the number of layers in the structure. The upper (blue) curve corresponds to the bulk crystalline state of Mo; the lower (red) curve corresponds to a reduced density of Mo in thin layers (about 80% of the bulk). The interfacial roughness is equal to zero.

- the interface is non-symmetrical (i.e. Mo-on-Si and Si-on-Mo intermixing layers are of different thicknesses and of various microstructures);
- the presence of silicides and oxides in the crystalline phase within intermixing areas.

What is the significance of whether Mo is in the crystalline phase or in the amorphous one? Let us show how the state of the metal layer determines the performance of reflecting coatings. It is important to recall that optical properties of a thin film differ from those of a bulk material. Two boundary cases are shown in figure 1, which represents a simulation of the normal incidence reflectivity of the Mo/Si multilayer at 13.5 nm as a function of the number of layers with no respect to the interfacial roughness. The blue upper curve corresponds to the bulk crystalline state of the Mo layer (bcc phase). The reflectivity was calculated by using the corrected refraction index for Mo from the CXRO data bank [35]. The red lower curve is reconstructed in assumption that the density of molybdenum in a thin layer is 80% of that of the bulk material according roughly to the mean value estimated from the results of density measurements of a few-nanometre-thick amorphous Mo film [36].

Thus, most of the real Mo/Si multilayers fall into the area between two curves depending on the microstructure of the metal layer. Generally, it means that the number of periods in the stack is a somewhat more important parameter that one could think of. We would rather suggest depositing at least 60 pairs of Mo/Si in order to get a maximum reflectivity at 13.5 nm independently of what the state of the metal layers is.

In our calculations of the Mo/Si reflectivity in the EUV range, we did not take into account a roughness of the interface between two layers. For normal incidence geometry, the better reflectivity peculiar to the multilayer with polycrystalline Mo is solely due to higher contrast in the refractive indices of the materials. On the other hand, the interface between two materials in an amorphous state is more distinct. Moreover, we suppose that the formation of Mo silicides within interdiffusion zones is less favourable in amorphous multilayers. A gain in the multilayer reflectivity due to reduced interfacial roughness will be more significant in grazing geometry and at shorter wavelengths. Many x-ray applications impose rather strict requirements on the multilayer roughness, which can hardly be met if no efforts are performed to compensate any possible increase of the roughness caused by structural changes during the multilayer growth. Various techniques and methods proved to be useful for this purpose, like a buffer layer deposition or ion beam polishing [37–39], but they make the multilayer deposition process more complicated, thus more expensive and time consuming.

5. Results and discussion

We optimized the rf-sputtering process empirically such that the Mo layer state could vary from amorphous to polycrystalline and vice versa, not being a function of the layer thickness up to about 3.5 nm. For a given distance between the cathode and the substrate, which was about 8 cm, we used a variation of either the substrate temperature or the working gas pressure. The latter is faster but it takes some time to calibrate the deposition rate at different values of the Ar pressure. The substrate temperature varied in the range from zero to 60 °C. This allows us to prevent uncontrolled heating of the substrate by plasma radiation, and also to influence thermalization conditions for sputtered adatoms arriving on the substrate.

Previously, the effect of increasing the interfacial roughness in multilayers prepared with increased gas pressure was observed in a far higher pressure range (in the order of 10^{-2} mbar), typical for the dc mode of the magnetron sputtering process [21, 36]. It is explained by reduced flux and the loss of the energy of sputtered particles due to increasing number of collisions with gas atoms, which result in decreasing density and porous structure of deposited films. Thus, the interdiffusion goes more easily, smearing the interface between two materials. On the other hand, changes of neither the roughness nor the structure of multilayers were reported in other publications, where the sputtering process was performed at very low working pressure (in the order of 10^{-4} mbar) [32].

In our experiment, we varied the Ar pressure within an intermediate range between 1×10^{-3} and 4×10^{-3} mbar, where the mean free path for sputtered atoms is of the same order (and few times smaller) than the distance between the cathode and the substrate. In accordance with the well known Thornton scheme [21], we tried to tune the energy of Mo atoms arriving on the substrate close to the crystallization threshold for various thermal regimes of the surface. Therefore, relatively small changes (found empirically) of either the Ar pressure or the substrate temperature during the deposition process led to structural changes occurring within the metal layer.

We have selected the samples for EUV measurements upon the results of the TEM study of their microstructure. The presence of the crystalline phase of Mo and of Mo silicides in the multilayers was also analysed using the XRD technique. The formation of intermixing zones, their composition and microstructure depend, in fact, on deposition parameters and should be bounded to the state of the materials. In order to distinguish an influence of the structure of metal layers on optical reflectivity of the Mo/Si multilayer mirrors, we used a simple 'two layers in period' model of multilayers. In this model, each layer has an effective thickness, density and refractive index, and the interdiffusion is accounted for by the statistical parameter of the interfacial roughness. The latter can be estimated from the low angle x-ray reflectivity and diffraction measurements (the fitting procedure is described in [31]). The substrate and the multilayer surface can also be analysed by AFM (atomic force microscopy).

The EUV reflectivity of the Mo/Si multilayers was studied in relationship to the state of the metal layer, which is either amorphous, polycrystalline or mixed. Here we present first the results of characterization for two typical samples: Ma40-A and Ma40-C. We use such a name assignment to point to the number of periods and the microstructure of the metal layer, which we consider either amorphous (A) or polycrystalline (C). Their TEM images are shown in figures 2(a) and (b). One more sample with amorphous Mo layers and a bigger number of periods ($N = 60$) was tested as well (Ma60-A).

The choice of the value of d-spacing was driven by a limitation of the experimental geometry (incident angle $\leqslant 80°$) due to a particular design of the detector holder inside the Elettra polarimeter. The low angle x-ray (Cu Kα radiation, $\lambda = 1.54$ Å) reflectivity curves (not given here) of these samples were best fitted with the following parameters.

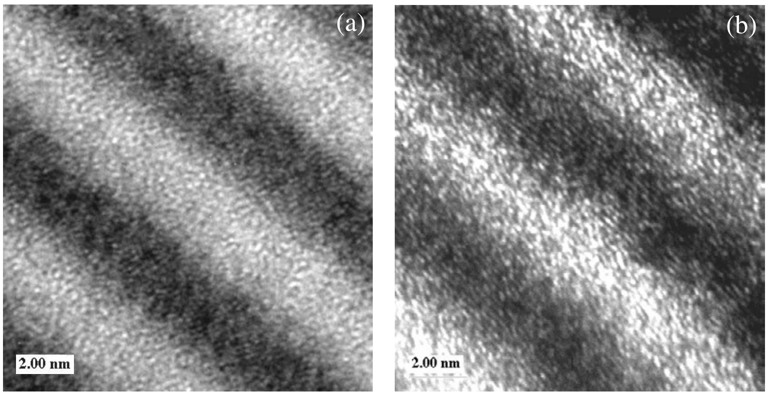

Figure 2. HRTEM images of the multilayers: Ma40-A (Mo amorphous)—(a); Ma40-C (Mo polycrystalline)—(b). Mo layers are dark, Si layers are light.

- Ma40-A: 40 periods, $d = 76.3$ Å, $d_{\mathrm{Si}} = 43.5$ Å, $d_{\mathrm{Mo}} = 32.8$ Å;
- Ma40-C: 40 periods, $d = 74.8$ Å, $d_{\mathrm{Si}} = 43.5$ Å, $d_{\mathrm{Mo}} = 31.3$ Å;
- Ma60-A: 60 periods, $d = 76$ Å, $d_{\mathrm{Si}} = 43.5$ Å, $d_{\mathrm{Mo}} = 32.5$ Å.

We have not detected any significant variation of the multilayer roughness unless the metal layer structure is changed. The rms values of the interfacial roughness estimated from the fit are 1.6 Å (Mo on Si), 2.4 Å (Si on Mo amorphous) and 3.2 Å (Si on Mo polycrystalline). The statistical error for the values of period is 0.1 Å.

The data for amorphous Mo/Si multilayers were best fitted with slightly reduced values of both *delta* and *beta* compared to those from the Henke table for the complex refractive index of Mo. In the fitting procedure, no minimization was done and that is why we do not give these numbers in present paper. But we would like to recall that the tabulated values for the metals utilized in the fabrication of thin films and multilayers were determined semi-empirically starting from rather thick films having the bulk crystalline structure (see in [35], for instance). Thus, an inhomogeneity of the refractive index due to a given microstructure of thin-layered materials and its possible reduction on the score of reduced density of metals in few-nanometre thin films should be concerned in simulations of the optical performance of multilayer-based elements.

We also succeeded in preparing samples with the Mo layers in two different states. Two phases of the metal layers coexisting in the stack are clearly seen in the TEM images presented in figure 3. In this case, the deposition rate was kept constant so one can expect to observe a contraction of the multilayer period (a few per cent) due to crystallization of Mo.

Simulated and experimental low angle x-ray reflectivity curves for this sample are shown in figure 4. The best fit for the sample Ma40-D (here D is for double structure) gives the following parameters:

$$d_1 = 76.2\ \text{Å},\ \ d_2 = 74.0\ \text{Å},\ \ d_{\mathrm{Si}} = 43.5\ \text{Å},\ \ d_{\mathrm{Mo}} = 32.7(30.5)\ \text{Å}.$$

The presence of two dissimilar structures in the stack is confirmed by doubled Bragg peak character of the reflectivity curve and the same is seen on the corresponding part of the electron diffraction micrograph (also presented in figure 4). This result is very similar to x-ray measurements of the Mo/Si multilayers whose microstructure was modified by ion beam implantation, the technique proposed as an alternative to the ion etching for fabrication of x-ray optical elements [40].

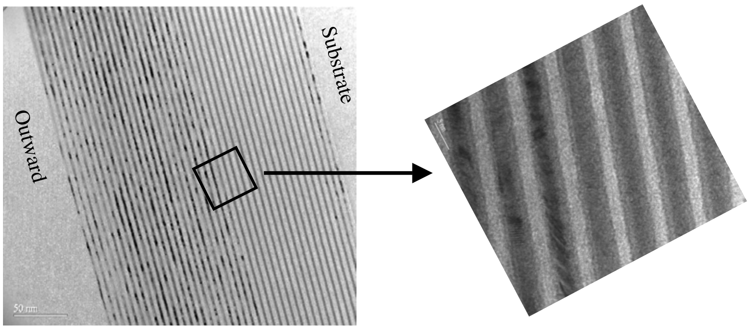

Figure 3. TEM images of the double-structured multilayer (Ma40-D). The inset is the zoomed part of the phase transition within the stack.

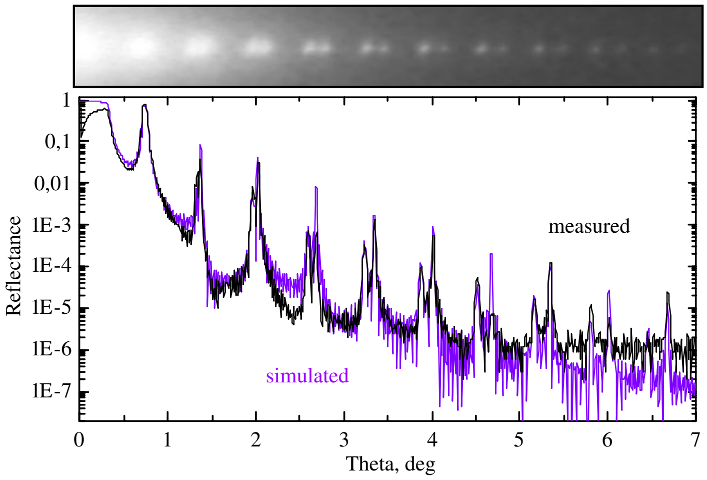

Figure 4. Low angle x-ray (Cu Kα) reflectivity curves of the Mo/Si multilayer with the double structure: black—measurement; violet—simulation. Above—corresponding part of the electron diffraction micrograph of the same multilayer.

Figure 5 shows the XRD patterns of the Mo/Si multilayers peaked around 40°, which is the signature of the (110) crystallographic plane in the bcc lattice of Mo. One can note that the crystalline phase is also present in the multilayers that we consider amorphous, even though its fraction is not significant. It was not possible to avoid the crystallization of Mo in a few layers close to the substrate, but the rest of the stack is practically free of crystallites.

The EUV reflectivity curves of the multilayers at the wavelength of 13.5 nm as a function of the incident angle and reflectance spectra measured at fixed angle of incidence (72°) are shown in figures 6(a) and (b). For the Mo/Si multilayer of 60 periods (i.e. 120 layers), Ma60-A, the peak reflectivity achieves nearly 70%, which levels with the best results for the near-to-normal incidence Mo/Si mirrors (even though it cannot be directly compared because of slightly different period).

It is worth to note that the term 'polycrystalline' used in our paper is conventional. It does not necessarily imply that the metal layer is totally crystallized. On the contrary, the Mo crystallites in Mo/Si multilayers have a notable columnar and/or granular structure. Crystallized in this way, the Mo layer has in turn a reduced density compare to bulk Mo.

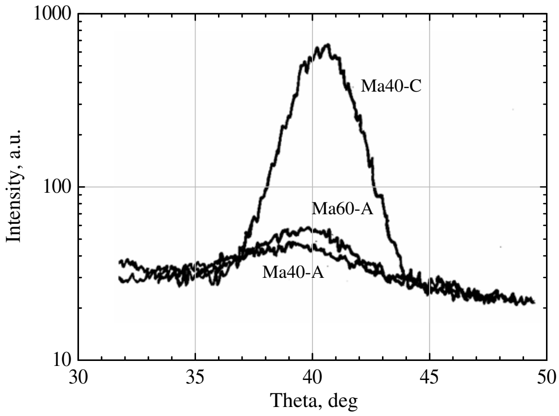

Figure 5. Large angle x-ray diffraction patterns of Mo/Si multilayers. Peaks around 40° correspond to Mo(110). The higher peak is for Ma40-C; the lower peaks belong to Ma60-A and Ma40-A correspondingly.

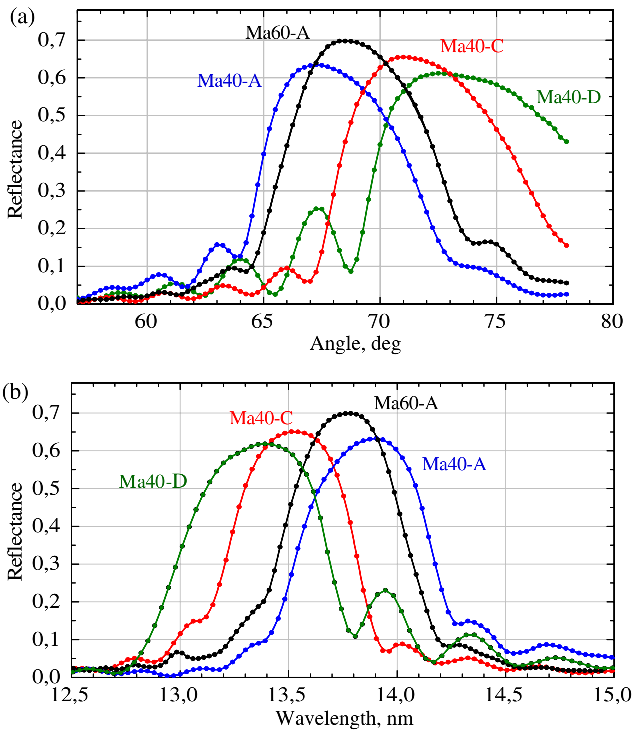

Figure 6. Reflectance of the Mo/Si multilayers at 13.5 nm as a function of incident angle (a) and the reflectivity spectra of the multilayers at fixed incident angle 72° (b). Legend: Ma40D—green; Ma40A—blue; Ma40C—red; Ma60A—black.

Moreover, the crystallization increases the physical roughness on the boundary with neighbour Si layer. This is why the reference to figure 1 should not be directly applied.

We compare the reflectivities in two cases: first—of the polycrystalline and amorphous Mo/Si multilayers with the same period number, and second—of the samples of a similar amorphous structure but with different period numbers. In the former case, the smaller value of the period of the Ma40C multilayer promises about 0.3% higher peak reflectivity than Ma40A according to simulations, which do not consider the differences in the microstructure and roughness. Thus, the better reflectance (by about 2%) of the polycrystalline sample is mainly due to higher optical contrast of refractive indices, which dominates a possible 1% loss in the reflectivity because of increasing roughness. In the latter case, the improved reflectance is because of higher number of periods (60 versus 40) in the stack being accompanied, probably, with increasing average density of amorphous metal layers. The reflectance of the double-structure multilayer proved to be the worst of four samples, as expected. Speculating on what reflectance of the 60-period polycrystalline Mo/Si multilayer could be attained, we would suppose it to be a bit higher than Ma-60A under the condition that interfacial roughness will not increase more.

As far as the fabrication of the Mo/Si multilayer mirrors is of great concern for EUV lithography, the conclusion to be drawn is as follows. If it is not possible to provide the uniform growth of crystalline metal layers with reduced interfacial roughness, then it would be highly desirable to ensure the formation of amorphous Mo layers of density as close to the bulk one as possible.

The fundamental mechanisms of atomic and molecular assembly during the growth of thin films (and multilayers) and analysis of microstructure evolution deserve more thorough study. It can exploit advances in *ab initio* computation of structure, molecular dynamics simulations of structure evolution and the kinetic Monte Carlo method to develop a detailed understanding of the key processes and their sensitivity to the deposition conditions [41].

Significant progress is likely possible by coupling the modelling capability with direct diagnostics of various parameters of the deposition process like ion density, electron temperature and plasma potential, energy distribution and flux of sputtered atoms, ions and neutrals impinging the substrate, stress measurements, etc.

6. Conclusion

We have demonstrated the capability to control the multilayer microstructure during the deposition process. The characterization of the Mo/Si multilayers with various microstructures was performed. It proved to be possible to fabricate the multilayers with up to 3.5 nm thick amorphous metal layers and reduced interfacial roughness. Still, it would be desirable to ensure the formation of amorphous Mo layers of density as close to the bulk one as possible.

The rf-magnetron sputtering technique, which is relatively simple and rapid, has a potential of being chosen as an industrial process for the multilayer coatings. Exceptional stability of the deposition process together with the possibility to control the microstructure of individual layers ensure a high repeatability of the layer thickness along a stack with a great number of periods.

Further investigations, which are in progress, would answer the question of how far one can go in the multilayer microstructure engineering via optimization of the deposition process. New promising directions would be those that enable control of adatoms in the 1–20 eV range to ensure the growth of multilayers with desirable microstructure and smooth interfaces.

Acknowledgments

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