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This report describes work conducted during the past three years under contract AFOSR-90-0140, "Laboratory for X-Ray Optics." During this period we concentrated our effort in two areas: 1) growth of epitaxial single-crystal beryllium films; and 2) multilayers and interfaces of material pairs incorporating "new" materials for multilayer x-ray optics. To date we have succeeded in growing epitaxial single-crystal films of beryllium on α-Al_2O_3 (sapphire), silicon, and germanium using molecular beam epitaxy (MBE). This is a major development since beryllium is an excellent spacer for the difficult, but desirable, water window wavelengths (24 Å < λ < 44 Å). To our knowledge, this is the first time beryllium has been grown epitaxially on any substrate. Our best quality beryllium films were grown on silicon. The ability to grow a single crystal layer of beryllium is a promising first step toward a beryllium-based single-crystal mirror. In the area of new material pairs, we began by using theoretical reflectivity calculations to assemble a list of promising new material pairs. We then prioritized this list by using what was already known about the materials, such as phase diagrams and diffusion studies. These material pairs then were studied by sputtering and/or MBE. We obtained good soft x-ray reflectivity results from silicon/boron, titanium/tungsten, yttrium/molybdenum, and boron carbide/palladium. One yttrium/molybdenum mirror had a measured normal-incidence reflectivity of 25.6% at 113 Å, the highest reflectivity we are aware of for any mirror operating at λ < 125 Å. Our development of low-angle x-ray diffraction techniques focused on nonspecular x-ray scattering as a characterization tool. The non-specular behavior can be understood in terms of current theories of x-ray scattering from multilayers with correlated interface roughness. We also have fabricated multilayer mirrors, by sputtering, for several soft x-ray laser experiments during this period.

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ABSTRACT

During this period we concentrated our effort in two areas: 1) growth of epitaxial single-crystal beryllium films; and 2) multilayers and interfaces of material pairs incorporating "new" materials for multilayer x-ray optics. To date we have succeeded in growing epitaxial single-crystal films of beryllium on α-Al_2O_3 (sapphire), silicon, and germanium using molecular beam epitaxy (MBE). This is a major development since beryllium is an excellent spacer for the difficult but desirable water window wavelengths (24 Å < λ < 44 Å). To our knowledge, this is the first time beryllium has been grown epitaxially on any substrate. Our best quality beryllium films were grown on silicon. The ability to grow a single crystal layer of beryllium is a promising first step toward a beryllium-based single-crystal mirror. In the area of new material pairs, we began by using theoretical reflectivity calculations to assemble a list of promising new material pairs. We then prioritized this list by using what was already known about the materials, such as phase diagrams and diffusion studies. These material pairs then were studied by sputtering and/or MBE. We obtained good soft x-ray reflectivity results from silicon/boron, titanium/tungsten, yttrium/molybdenum, and boron carbide/palladium. One yttrium/molybdenum mirror had a measured normal-incidence reflectivity of 25.6% at 113 Å, the highest reflectivity we are aware of for any mirror operating at λ< 125 Å. Our development of low-angle x-ray diffraction techniques focused on non-specular x-ray scattering as a characterization tool. The non-specular behavior can be understood in terms of current theories of x-ray scattering from multilayers with correlated interface roughness. We also have fabricated multilayer mirrors, by sputtering, for several soft x-ray laser experiments during this period.

Epitaxial Growth of Beryllium

High quality beryllium thin films are desirable for possible applications in several fields. Beryllium is of great interest to the x-ray optics community because of its extremely low absorption in the soft x-ray region. Calculations for multilayer mirrors with beryllium spacer layers show that they should have very high reflectivities for the wavelength ranges $\lambda < 44$ Å and $\lambda = 111$-200 Å. Such mirrors would be useful for x-ray microscopy, astronomy and lithography. Beryllium also is useful for infrared (IR) optics due to its high reflectance, high thermal conductivity, low thermal expansion coefficient, good oxidation resistance and good thermal stability. Single-crystal x-ray and IR mirrors may have advantages over the typical amorphous and polycrystalline films and multilayers that are presently used. The very low electrical resistivity of beryllium at low temperatures and its low density (1.85 gm/cm^3) makes beryllium films attractive for use for electromagnetic shielding in space applications. Our recent epitaxial growth of beryllium thin films on single-crystal substrates is a significant first step toward producing single-crystal multilayers.

We should note that although solid beryllium is relatively harmless, inhalation of its dust can lead to berylliosis, a debilitating and chronic lung disease. We worked with our Office of Risk Management to devise a set of appropriate laboratory procedures, which are carefully followed to prevent beryllium dust exposure. The molecular beam epitaxy (MBE) apparatus we use for this work is housed in a class-1000 clean room and has a separate introduction chamber so that the growth chamber is not vented when introducing or removing samples.

We used crystalline α-Al_2O_3(0001) (sapphire) and Si(111) substrates for our studies because of their nearly 2:1 lattice match with hcp-Be. The basal planes of α-Al_2O_3 consist of equilateral triangular arrays of atoms with spacing nearly twice that of the spacing between the atoms in the hcp-Be(0001) planes. The (111) planes of silicon also have a close lattice match, but only when rotated by 30 degrees with respect to the Be(0001). Using these relative alignments, the in-plane lattice mismatch is only 3.9% for beryllium and 3.1% for silicon.

All of the beryllium films were grown at pressures of $\sim 2 \times 10^{-10}$ torr in our Riber-1000 MBE machine while simultaneous reflection high-energy electron diffraction (RHEED) observations were made. We grew films on Al_2O_3 and silicon at several substrate temperatures from 10 °C to 500 °C and at several deposition rates from 3.4 Å/min to 18.6 Å/min. After deposition we characterized the samples *ex situ* with a variety of x-ray diffraction (XRD) techniques, optical microscopy, scanning electron microscopy (SEM), atomic force microscopy (AFM), and ion beam analysis with both He^+ and proton backscattering.

In all the cases we have studied to date, the RHEED patterns indicate that beryllium forms an epitaxial overlayer with the hcp-Be [0001] direction parallel to α-Al_2O_3 [0001] and Si [111]. In-plane we find that the hcp-Be lattice is rotated 30 degrees with respect to the hcp-oxygen lattice of the α-Al_2O_3, that is Be[1$\bar{1}$00] $\parallel$ α-Al_2O_3[1$\bar{2}$10], for all of the samples except for the one deposited at 500 °C. In the case of the 500 °C sample, the hcp-Be lattice is aligned with the α-Al_2O_3 lattice. The beryllium grew on silicon with a 30-degree rotation with respect to the triangular lattice of (111) silicon atoms for all temperatures studied.

Our XRD studies of these films also indicate that the beryllium has grown epitaxially. High-angle θ-2θ diffraction showed only the α-Al_2O_3(0006) substrate peak and Be(0002) peak. Figure 1 shows θ-2θ XRD spectra and rocking curves for two samples. The solid lines are data for the sample made on Al_2O_3

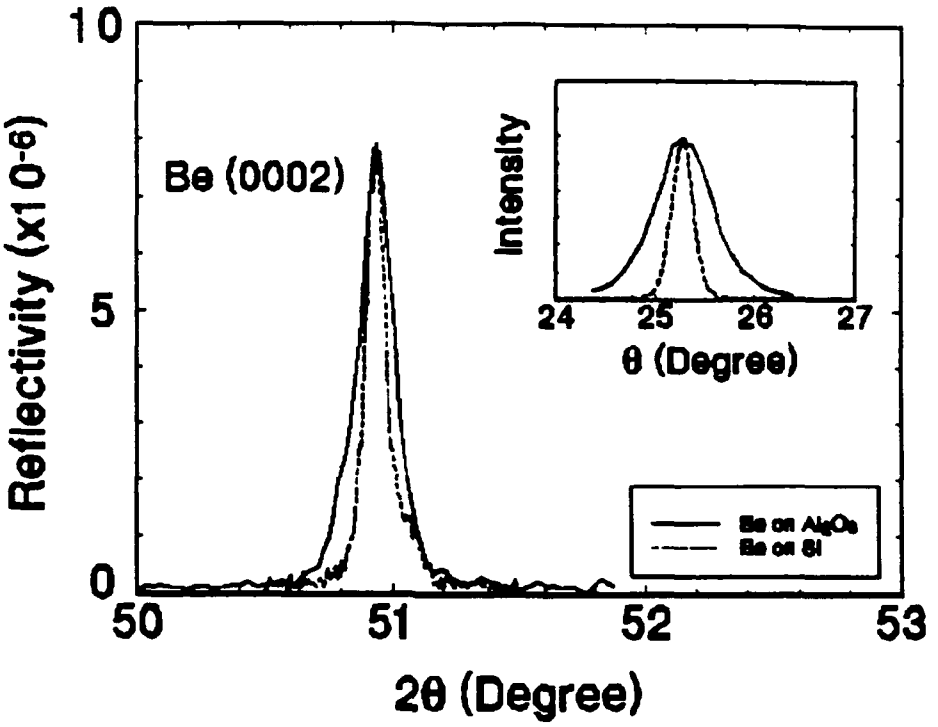

Figure 1. Zero to 2θ diffraction and rocking curves (inset) for epitaxial beryllium on silicon and Al_2O_3. Analyses of the θ-2θ spectra yield coherence lengths nearly equal to the film thicknesses.

at T = 10 °C and at a beryllium deposition rate of 3.8 Å/min. The dashed curves are for a beryllium film grown on silicon at T = 300 °C at a rate of 5.7 Å/min. When corrected for instrumental broadening, the 2θ full-width-at-half-maximum for each θ-2θ curve shown in Fig. 1 corresponds to a structural coherence length nearly equal to the total thickness of the corresponding film. That is, these data are consistent with the films being single-crystals. The rocking curves taken about the same peaks are shown in the inset. The film grown on Al_2O_3 has $\delta\theta = 0.65$ degrees and the film grown on silicon has $\delta\theta = 0.24$ degrees. A width of 0.65 degrees is approximately an order of magnitude narrower than that for a highly textured polycrystalline metal film. Crystalline perfection giving rise to a width of only 0.24 degrees is rarely seen in metallic thin films, and indicates the very high quality of these samples. All of the films grown on Al_2O_3 had similar $\delta\theta$ and $\delta 2\theta$ except for the sample grown at 500 °C, which had significantly narrower peak widths of $\delta\theta = 0.075$ degrees and $\delta 2\theta = 0.10$ degrees. Unfortunately, the 500 °C sample was composed of large beryllium islands rather than being a uniform film.

Ion beam channeling measurements were made on several of the samples by J.A. Leavitt, L.C. McIntyre, M.D. Ashbaugh, Z. Lin, J. Frank and S. Champlin at the University of Arizona's Ion Beam Analysis Laboratory. The backscattering yield was measured both with the beam aligned along the α-Al_2O_3[0001] or Si[111] direction and for a "random" case in which the beam was tilted 7 degrees off axis and the sample rotated during the measurement. To make a channeling measurement on the small atomic mass beryllium overlayer the large resonance in the ^1H on ^9Be cross section at 2.525 MeV was exploited

to provide a beryllium signal that was measurable above the background caused by the substrate. The channeling ratio χ, i.e. the ratio of the backscattering yield in the aligned and random directions, indicates the crystalline quality—smaller χ indicates fewer defects. All of the epitaxial samples channeled, but the best to date has $\chi = 0.33 \pm 0.02$. This is for a beryllium film grown on Si(111) at T = 300 °C. This small channeling ratio implies that the beryllium-on-silicon film is a very high quality crystal, which agrees with our x-ray data. The observation of channeling in these films is strong evidence that the beryllium overlayers have long-range order and are aligned with the crystalline planes of the substrate.

We also have succeeded in growing epitaxial beryllium on germanium. The germanium surface was prepared by epitaxial growth of a germanium buffer layer on Si(111) substrates. The quality of these beryllium films is not as high as for beryllium on silicon, but it is better than for beryllium on Al_2O_3. Since the mismatch between beryllium and germanium is less that for silicon, ($\sim 1\%$ in-plane) it is somewhat surprising that the beryllium grown on germanium is less perfect than when grown on silicon. The difference may be due to the germanium buffer layer being not of the same quality as the silicon substrates. We will continue this project by working to improve the starting germanium surface to improve the quality of the beryllium films grown on germanium.

In summary, we have used MBE to grow single-crystal (0001) hcp-Be epitaxially on α-Al_2O_3(0001), Si(111), and Ge(111) for several growth conditions. To our knowledge, these are the first reports of beryllium epitaxy on any substrate material. Although we have grown good quality epitaxial beryllium films on all three substrates, the films grown on silicon have significantly better crystalline perfection. These studies are important first steps toward growing high quality beryllium-based multilayers and superlattices. The Al_2O_3 results have been published in *Applied Physics Letters* and a second manuscript, including growth studies on silicon, has been submitted.

Multilayers Made With New Material Combinations

A typical material pair has a small region of high reflectivity for $\lambda > \lambda_{edge}$, where λ_{edge} is the wavelength of an absorption edge in the spacer material. Thus, to make optics for the entire soft x-ray region, several different spacer materials are needed, each of which is optimal over only a small wavelength region. For a given spacer, several absorbers typically yield similar reflectivity curves. Our approach has been to study each spacer material in turn, combining them with several of the most promising absorbers. We chose the absorbers to study based on optical constants, binary phase diagrams, literature searches for growth properties, and previous experience in our laboratory.

To test a new material pair we normally begin by sputter-depositing several test multilayers of that pair under various sputtering conditions. Recently we made test multilayers of titanium with cobalt, nickel, copper, tungsten; yttrium with molybdenum, silver, palladium, and niobium; carbon with cobalt; and boron carbide with palladium. Of these combinations, we obtained good soft x-ray reflectivity results from titanium/tungsten, yttrium/molybdenum, and boron carbide/palladium. One yttrium/molybdenum mirror had a measured normal-incidence reflectivity of 25.6% at 113 Å, the highest reflectivity we are aware of for any mirror operating at $\lambda < 125$ Å. Several of the other pairs worked well enough to merit further study. Since yttrium/molybdenum and yttrium/niobium have a lattice match for one crystal orientation, they merit study by MBE.

Boron is potentially useful as a spacer in multilayer x-ray optics for wavelengths between the boron K-edge at 67 Å and approximately 125 Å. Boron-based multilayer mirrors have theoretical

reflectivities >50% for wavelengths between 65 Å and ~120 Å. Such multilayers would be quite useful, since this wavelength region lies above that for which tungsten/carbon functions, and below that for which molybdenum/silicon works well. To investigate the usefulness of this element as a spacer, we have fabricated multilayers incorporating boron by alternately depositing several material pairs, including Ag/boron, palladium/boron and boron/silicon. We are interested in the Ag/boron system as a possible crystalline superlattice x-ray mirror. Unfortunately, the tendency of Ag to form islands has frustrated our efforts to date to grow both single crystal boron overlayers and quality non-epitaxial multilayers. The palladium/boron system shows promise as an amorphous multilayer system. Our work shows that the palladium reacts with the boron to form an amorphous, palladium-rich boride. However, calculations indicate that the ideal reflectivity of the reacted multilayers may be as high as 51% at 80 Å. In the boron/silicon system, boron functions as the absorber layer for wavelengths greater than approximately 125 Å, resulting in a narrow bandpass mirror. We have produced test multilayers of Pd_xB/boron and silicon/boron. Low-angle x-ray diffraction characterization of the test multilayers and surface analysis studies of single interfaces show that the samples are layered with relatively sharp interfaces.

Studies of Interfaces with Boron

We have studied the growth of several candidate materials on boron to determine their growth modes, the chemical sharpness of the interfaces, and the structure of the layers formed. The films were deposited in ultrahigh vacuum and studied with *in situ* Auger electron spectroscopy (AES), x-ray photoelectron spectroscopy (XPS), and RHEED. Scanning tunneling microscopy (STM) characterization and Rutherford backscattering calibrations were performed after removing the samples from the vacuum system. For all of these studies amorphous boron films were first formed by depositing at room temperature onto oxidized silicon wafers. Following deposition of each boron film, overlayers of palladium, Ag, or silicon were deposited at substrate temperatures of ~50 °C and immediately studied with AES and XPS. In the case of palladium, we find that at room temperature it reacts with the boron to form a smooth, amorphous Pd_xB_{1-x} layer, with $x \approx 0.9$. With annealing the boride becomes more boron rich, with x dropping to ~0.55. This reaction occurred for all palladium coverages studied, from 2.3 Å to 230 Å. However, our calculations show that the Pd_xB_{1-x} should perform as a good absorber layer as part of Pd_xB_{1-x}/boron multilayer mirrors. We find that silicon deposited onto boron forms a sharp interface and an amorphous overlayer of pure silicon. Depositing Ag on boron results in a polycrystalline layer, composed of large Ag islands.

Non-Specular X-Ray Scattering from Multilayer X-Ray Mirrors

We have observed resonant non-specular x-ray scattering effects in silicon/molybdenum multilayers and we have described the experimental techniques and results in the previous report. Using a simple analysis of the data, based on recent theories of x-ray scattering from periodic multilayers, we previously used our measurements to determine that correlated roughness was present in these multilayers for spatial periods $l > 200$ Å. To extract more structural information, a specific model must be chosen. The model must include the power spectra of the interfaces and describe how the roughness is propagated through the stack. In collaboration with Daniel Stearns at Lawrence Livermore Laboratory, this type of detailed analysis of the data for one high quality silicon/molybdenum multilayer (sputter-deposited at 3 mtorr Ar pressure) has now been completed. We have used a growth model developed by Stearns where the growth is parameterized in terms of a relaxation parameter ν describing the damping of features with increasing film thickness and Ω representing the volume of a growth unit. The results are the power spectral density (PSD) of the roughness as a function of spatial frequency and number of layers and a

correlation number as a function of spatial frequency. The behavior of the correlation number is particularly interesting since it represents the number of bilayers over which the roughness is correlated at a particular spatial frequency. These results show that roughness with $l > 1000$ Å is correlated over hundreds of bilayers, but the correlation number drops rapidly with increasing spatial frequency. For $l < 100$ Å the roughness is effectively uncorrelated (correlation number < 2 bilayers).

We now have developed this technique to the point that it can be used to determine the magnitude of the roughness and its origin. By combining non-specular scattering results with transmission electron microscopy (TEM), STM, and/or AFM results, and studying a series of samples, we could separate the substrate effects from the intrinsic roughness generated by the multilayer. This information will be invaluable in improving both the substrate surface roughness and the intrinsic multilayer roughness.

Sputtered Mirrors

Molybdenum/silicon multilayer mirrors, which we designed and fabricated to match the 236 Å germanium lasing transition, were used in x-ray laser experiments at Rutherford Appleton Laboratory in April 1991. The laser output brightness at 236 Å was typically increased by 30-fold when our cavity mirror was used, in spite of the fact that the multilayer was vaporized during the pulse. These experiments showed an exponential increase in the gain as a function of plasma length and the first demonstration of saturation of gain for a soft x-ray laser. In addition, the gain-length product of 21 is the highest demonstrated to date for any x-ray laser. These laser results were published by A. Carillon, et al. in Phys. Rev. Lett. **68**, 2917 (1992). We also provided mirrors for 182 Å to Szymon Suckewer and Charles Skinner at Princeton Plasma Physics Laboratory for plasma diagnostics experiments.

In addition to the characterization we performed in our laboratory, we sent silicon/molybdenum mirrors to collaborators at Sandia National Laboratories for characterization with high resolution transmission electron microscopy (HTEM). (We now have our own HTEM so this is no longer necessary.) These mirrors complete a series of molybdenum/silicon multilayers made, at various pressures and with various layer thicknesses, to study the effects of these variables on the structure and performance of these mirrors. The results indicate that varying the sputtering pressure has a dramatic effect on the multilayer structure. Higher sputtering pressures cause the roughness to increase. Since the higher sputtering pressure reduces the energy of the depositing atoms, the surface mobility is reduced causing a columnar-like growth mode. The best mirrors are those produced in an atmosphere of 3.0 mtorr Ar (the lowest pressure studied).

R. Watts C. Tarrio and T. Lucatorto of NIST made the reflectivity measurements on most of the sputtered mirrors at the SURF-II beamline. C. Montcalm, H. Pepin and M. Chakar of the Université du Québec made reflectivity measurements on several of the multilayers made of "new" materials. In addition, C. Montcalm visited our laboratory for two months in early 1993 and participated in sputter-deposition of "new-materials" multilayers.

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Degrees Awarded

Judith Ruffner Ph.D., passed oral exam February 12.