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Multilayer Optics for Soft X-Rays

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ABSTRACT

We have shown that high quality Be films may be grown on α-Al_2O_3, Si(111), and Ge(111). Consistent with the relative lattice mismatches, films grown on Si(111) are of higher quality than those grown on α-Al_2O_3, and those grown on Ge(111) are of the highest quality. The epitaxial Be films grown on Ge during this grant period are the best quality Be films that we or anyone else have ever produced. Growth of these high quality films is a significant step toward single-crystal heterostructures containing Be. Such Be-containing structures may be useful, not only in EUV optics, but also in IR optics, electronic devices, and studies of thin-film superconductivity.

We have been successful in growing epitaxial Co-on-Be and Ge-on-Be, but not in making Co/Be or Ge/Be superlattices. In both cases the problem is related to limitations of our equipment. To pursue this work further would require an upgrade to our MBE apparatus.

Our very recent successes with sputter-deposited Y- and B_4C-based multilayers show that there is still much to be gained by studying and optimizing the growth of carefully chosen new material pairs. The Y/Mo reflectivity results we obtained during this grant period improved on the state-of-the-art by 50% to 100% in the λ=80–110 Å region. Our B_4C/Si multilayers are much better filters and have far superior stability compared with previous coatings for the same wavelength region. Many applications involving synchrotron radiation or laser-produced plasmas will benefit from both the optical characteristics and excellent stability of these EUV reflectors.

Epitaxial Growth of Beryllium

Our calculations for multilayer mirrors with Be spacer layers show that they should have high reflectivities for the wavelength ranges $\lambda<44$ Å and $\lambda=111–200$ Å. Other desirable properties of beryllium are its high thermal conductivity, low thermal expansion coefficient, good oxidation resistance, and good thermal stability. Our recent epitaxial growth of Be thin films on single-crystal substrates is a significant first step toward producing single-crystal multilayers.

Figure 1 shows calculated maximum attainable reflectivities for some Be-based multilayers. The curves represent the reflectivity of ideal multilayers that are optimized for each wavelength. The figure illustrates the wavelength regions where Be-based multilayers should be most useful are above its absorption edge at $\lambda\approx111$ Å, and below $\lambda=44$ Å where C-based multilayers would not perform as well. Note that the theoretical maximum reflectivity of Mo/Be at $\lambda\approx115$ Å is over 80%. Again at short wavelengths Be, this time in combination with Co, looks very good theoretically and has a lattice match that may be close enough for epitaxial growth. For these reasons, several years ago we started growing films of Be, as a first step toward the growth studies of epitaxial superlattices.

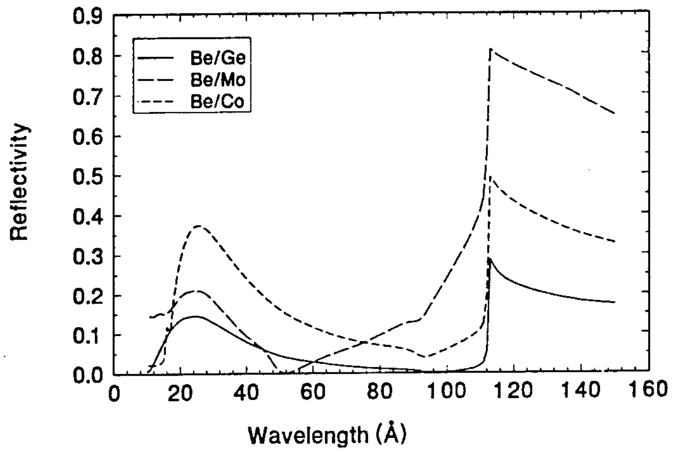

Figure 1. Calculated maximum attainable reflectivities for some Be-based multilayers. Each point on a curve represents the reflectivity of an ideal multilayer that is optimized for that wavelength.

In choosing substrates on which to grow Be films, we used two main selection criteria: (1) a small lattice mismatch with hcp Be and (2) low chemical reactivity with Be. Three materials meeting these criteria are α-Al_2O_3, Si, and Ge. Al_2O_3 and Si have very low reactivity with Be, and Ge and Be are completely immiscible. Furthermore, the lattice mismatches for Be(0001) and α-Al_2O_3(0001), Si(111), and Ge(111) are, respectively, 3.9%, 3.4%, and 1.0%. For Si(111) and Ge(111) this small mismatch occurs when the lattices are rotated 30° with respect to each other. Prior to this grant period we studied Be-on- α-Al_2O_3 and Be-on-Si in detail, and began studies of Be-on-Ge. During this grant period we filled in some gaps in the Be-on-Si studies and performed detailed studies of Be-on-Ge. The epitaxial Be films grown on Ge during this grant period are the best quality Be films that we or anyone else have ever produced.

We grew all of our Be samples with our Riber-1000 MBE machine (base pressure of 2×10^{-10} Torr). Each film was grown while monitoring the surface structure with reflection high-energy electron diffraction (RHEED). RHEED analysis reveals the epitaxial orientation of the Be films as well as qualitative information about the roughness of the surface. We also analyzed each of these Be films *ex situ* with electron and atomic force microscopy, x-ray diffraction, Rutherford backscattering analysis, and ion beam channeling. Our work shows that epitaxial Be films of varying quality can be grown on these substrates.

For all three substrates studied, we found the Be crystalline quality is strongly dependent on the substrate temperature during deposition. The best growth temperatures, as well as structural parameters determined from x-ray diffraction and ion beam channeling, are given in Table I. X-ray linewidths improve for Be grown at even higher temperatures, but the increased mobility results a three-dimensional growth mode and therefore very rough films.

Table I. Summary of our best Be films grown on each of the three substrates considered to date.

Substrate	Growth Temperature (°C)	Rocking Curve FWHM	Channeling χ_{min}
α-Al_2O_3(0001)	125	0.51±0.02	0.81±0.02
Si(111)	300	0.24±0.02	0.33±0.02
Ge(111)	300	0.19±0.02	0.18±0.02

High-angle θ-2θ x-ray diffraction and RHEED analysis indicate that the beryllium films are preferentially oriented with the (0001) planes parallel to the α-Al_2O_3(0001), Si(111), or Ge(111) substrate planes. We performed x-ray rocking curve measurements on the (0002) peaks, and ion beam channeling measurements along the [0001] direction to judge the crystalline quality of the films. The FWHM of these rocking curves decreases with increasing substrate temperature. As shown in Table I, the FWHM for optimized Be-on-Si growth is approximately a factor of two narrower than for Be-on-α-Al_2O_3. For Be-on-Ge it is narrower still. The ion beam channeling results are described by the parameter χ_{min}, which is the ratio of the backscattering yield in the aligned and random directions. This ratio will be lower for better crystalline quality Be overlayers. To make a channeling measurement on the small atomic mass Be overlayer, the resonance in the ^1H on ^9Be cross section at 2525 KeV was exploited to provide a Be signal which was measurable above the background caused by the substrate. Observation of channeling in these films is direct evidence that the Be overlayers have long range order and are aligned with the substrate. As shown in the Table, the channeling results are in agreement with the trend observed in the rocking curve FWHM data. Not surprisingly, our results show that the crystalline perfection improves with improving lattice match.

We have been successful in growing epitaxial Co-on-Be, but not in making Co/Be superlattices. We find that Co suffers from island growth at temperatures significantly above 100 °C, while growth of single-crystal Be is best at 300 °C. Although we can heat quickly from 100 °C to 300 °C, we cannot quickly return the substrate temperature to 100 °C. This limitation of our equipment makes it difficult to produce good Co/Be samples with many layers. Similar problems were encountered in our attempts to grow Ge/Be superlattices. To pursue this work further would require an upgrade to our MBE apparatus. One possibility is a new sample manipulator that would allow fast changes in substrate temperature. Another possible way to proceed is to find a means to grow single-crystal Be at lower temperatures, such as the use of ion-assisted deposition.

To summarize this section, we have shown that high quality Be films may be grown on α-Al_2O_3, Si(111), and Ge(111). Consistent with the relative lattice mismatches, films grown on Si(111) are of higher quality than those grown on α-Al_2O_3, and those grown on Ge(111) are of the highest quality. Growth of these high quality films is a significant step toward single-crystal heterostructures containing Be. Such Be-containing structures may be useful, not only in EUV optics, but also in IR optics, electronic devices, and studies of thin-films superconductivity.

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 mirrors for the entire wavelength region of interest, several different spacer materials are needed, each of which will be optimal over only a small wavelength region. For a given spacer, several absorbers typically yield similar theoretical reflectivity curves. Our approach to date 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. During this grant period we have had success with two new pairs made by sputter deposition: Mo/Y and B_4C/Si.

We chose to study Mo/Y mirrors because Y is a good spacer from λ=124 Å down to approximately λ=75 Å. A Mo/Y mirror we recently made and measured at the Wisconsin Synchrotron Radiation Center, in a collaborative effort between our group and a group from NRC-Canada, had a normal-incidence reflectivity of 46% at 114 Å. Another mirror made in the same series of experiments had *R*=32% at 95 Å. Although there is still much work to be done to optimize Mo/Y multilayers, already our measurements show that this new pair performs better in the $\lambda \approx$100 Å region than any previously-reported pair.

Figure 2 illustrates the importance of these Mo/Y results. It shows the best near normal-incidence reflectivities measured to date in the wavelength region λ=40–140 Å. The circles represent the state-of-the-art before our Mo/Y work. It is clear that this new material pair has dramatically increased the potential for applications in the region between the yttrium M-edge and the beryllium K-edge, *i.e.* λ=80–111 Å. The fact that these results and the Mo/Be result shown in the figure were just obtained in 1994 supports our argument that there is

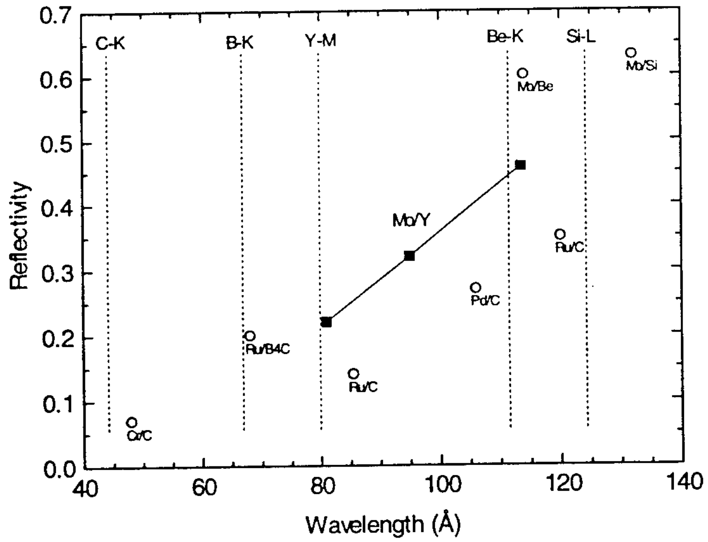

Figure 2. Near normal-incidence reflectivities measured to date. Our Mo/Y work (■) resulted in a great improvement over the previous state-of-the-art (O).

much to be gained by exploring new material pairs.

We also recently studied sputter-deposited B_4C/Si multilayers. Like Mo/Si, these multilayers perform well for λ>124 Å where the absorption of Si is very low. However, our interest in this new material pair stems from applications that require high selectivity more than maximum reflectivity. Compared to Mo/Si multilayers, B_4C/Si have a much narrower bandpass ($\delta\lambda$) and better rejection of off-peak light, albeit by sacrificing maximum peak reflectivity (R_o). However, mirrors with better filtering characteristics are needed for plasma diagnostics and for a variety of instruments that rely on synchrotron radiation from bending magnets. We made mirrors for three different peak wavelengths (λ_o) and angles: λ_o=133 Å at near-normal-incidence; λ_o=182 Å at 45°; and λ_o=236 Å at 45°. Because the Brewster angle is approximately 45° for these mirrors, the 45° mirrors act as polarizing filters. We have shown that B_4C/Si multilayers are far superior in this respect.

Table II summarizes the measured performance of our B_4C/Si mirrors with a comparison to similar Mo/Si mirrors. As can be seen, the B_4C/Si peaks are much narrower than for Mo/Si, ranging between 1.7 and 2.7 times narrower. In addition, the tails on both sides of the reflectivity maxima are typically one to two orders-of-magnitude lower than for Mo/Si, providing much better off-peak rejection. Although the reflectivities are good, as expected they are lower than for Mo/Si. Another very important property of these multilayers is that they exhibit excellent stability upon annealing at temperatures up to 600 °C. This result is in contrast to, and an important improvement upon, the behavior of Mo/Si multilayers, which undergo significant structural changes and a contraction in Λ upon annealing at 400 °C for only 30 minutes.

Table II. Measured performance of our B_4C/Si mirrors compared to similar Mo/Si mirrors. The B_4C/Si mirrors have lower peak reflectivity R, but much narrower bandpass $\delta\lambda$.

	B_4C/Si		Mo/Si	
Design	R	$\delta\lambda$ (Å)	R	$\delta\lambda$ (Å)
133 Å, normal incidence	0.275	3.1	0.61	5.3
182 Å, 45°	0.33	11	0.53	26.5
236 Å, 45°	0.30	20	0.39	46

From this work we conclude that when high reflectivity under moderate heat loads is the only concern, Mo/Si will continue to be the material pair of choice for λ>124 Å. However, B_4C/Si multilayers are clearly superior when narrow bandpass, good off-peak rejection, or thermal stability is important. The comparison is particularly favorable for B_4C/Si at the longer wavelengths, where the difference in the reflectivity decreases, and the filtering characteristics of B_4C/Si are far superior. Many applications involving synchrotron radiation or laser-produced plasmas will benefit from both the optical characteristics and excellent stability of these EUV reflectors.

To summarize this section, our group's very recent successes with sputter-deposited Y- and B_4C-based multilayers show that there is still much to be gained by studying and optimizing

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R. Watts C. Tarrio, and T. Lucatorto of NIST made the reflectivity measurements on the B_4C/Si mirrors at the SURF-II beamline. The Mo/Y multilayers were made and their reflectivity measured in collaboration with C. Montcalm and B. Sullivan of the National Research Council of Canada. J. A. Leavitt and L.C. McIntyre of the University of Arizona Physics Department did the ion beam analysis.

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1. **Epitaxial Growth and Surface Structure of (0001) Be on (111) Si.** Judith A. Ruffner, J.M. Slaughter, James Eickmann, and Charles M. Falco, Appl. Phys. Lett., **64**, 31 (1994).

2. **Roughness Correlations in Si/ Mo Multilayers.** J. M. Slaughter, D. G. Stearns, and Charles M. Falco, *Physics of X-Ray Multilayer Structures*, 1994 Technical Digest Series, vol. 6 (Optical Society of America, Washington, D.C., 1994) pp. 19–22.

3. **Epitaxial Growth of Be(0001) on Ge(111) and Ge(111) on Be(0001).** James Eickmann, J.M. Slaughter, and Charles M. Falco, *Physics of X-Ray Multilayer Structures*, 1994 Technical Digest Series, vol. 6 (Optical Society of America, Washington, D.C., 1994) pp. 178–181.

4. **Si/ B_4C Soft X-Ray Multilayer Mirrors.** Charles M. Falco, Brian S. Medower, and J. M. Slaughter, *Physics of X-Ray Multilayer Structures*, 1994 Technical Digest Series, vol. 6 (Optical Society of America, Washington, D.C., 1994) pp. 166–169.

5. **Preliminary Survey of Material Pairs for XUV Multilayer Mirrors for Wavelengths Below 130 Å.** Claude Montcalm, Patrick A. Kearney, J.M. Slaughter, M. Chaker, and Charles M. Falco, *Physics of X-Ray Multilayer Structures*, 1994 Technical Digest Series, vol. 6 (Optical Society of America, Washington, D.C., 1994) pp. 3–6.

6. **Structure and Performance of Si/ Mo Multilayer Mirrors for the Extreme Ultraviolet.** J.M. Slaughter, Dean W. Schulze, C.R. Hills, A. Mirone, R. Stalio, R.N. Watts, C. Tarrio, T.B. Lucatorto, M. Krumrey, P. Mueller, and Charles M. Falco, J. Applied Physics, **76**, 2144 (1994).

7. **Y/ Mo Multilayer Mirrors for XUV Wavelengths.** Claude Montcalm, Brian T. Sullivan, M. Ranger, J.M. Slaughter, P.A. Kearney, Charles M. Falco, and M. Chaker, Optics Letters, **19**, 1173 (1994).

8. **Si/ B_4C Narrow-Bandpass Mirrors for the Extreme Ultraviolet.** J.M. Slaughter, Brian S. Medower, R.N. Watts, C. Tarrio, T.B. Lucatorto, and Charles M. Falco, Optics Letters, accepted.

9. **Growth Modes of Pd, Ag, and Si Thin Films on B.** Patrick A. Kearney, J.M. Slaughter, Dian Hong Shen, and Charles M. Falco, J. Vac. Sci Tech. B, accepted.

10. **Growth and Surface Structure of Epitaxial Be Thin Films.** Charles M. Falco, James Eickmann, Judith A. Ruffner, and J.M. Slaughter, Proc. 2nd Conference on Thin Film Physics and Applications, Shanghi, China, April 15–17, 1994, submitted.

11. **On Developing a Table Top Soft X-Ray Laser.** A. Morozov, K. Krushelnick, L. Polonsky, C.H. Skinner, S. Suckewer, C.M. Falco, and J.M. Slaughter, Proc. IV Inter. Colloq. on X-Ray Lasers, Williamsburg, May 1994, submitted.

Degrees Awarded

Patrick A. Kearney	Ph.D., passed oral exam 3 May, 1994.