

Highly Reflective Uranium Mirrors for Astrophysics Applications

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ABSTRACT

The reported optical constants of uranium differ from that of vacuum significantly more than other elements do over the range of about 150 to 350 eV. This suggests that uranium could be used to produce high reflectance imaging mirrors for many soft x-ray applications. Elemental uranium is too chemically active to be used as a front surface mirror without protection. We computed the expected reflectance of carbon-coated uranium films and of uranium-nickel alloys for low-angle reflectors. Carbon is mostly transparent below its K absorption edge at about 283 eV. The reflectance at 10 degrees from grazing is computed to be greater than 50% at 277 eV (C K_α). For comparison, about 5 degrees is the maximum grazing incidence angle for which conventional materials are computed to have comparable reflectance. We sputter deposited and measured the reflectance of carbon-coated uranium layers at 44.7 Å (C K_α). Sample reflectance was a factor of two greater than that of nickel, the material used for low-angle mirrors. The initial oxidation behavior of sputtered uranium-nickel alloys is similar to pure U so their reflectance was not determined. Coatings based on uranium should be considered for all applications where high-reflectance, broadband, low-angle soft x-ray mirrors are required.

Keyword list: soft x-ray astronomy, uranium thin films, broadband reflectors, oxides, oxidation, carbon overcoats, barriers, uranium compounds, uranium alloys.

1. INTRODUCTION

1.1 Why Uranium?

The optical properties of uranium are noteworthy over much of the EUV and soft x-ray portions of the spectrum. Uranium metal is very dense (bulk density about 19 gm/cc) and has 92 electrons that may interact with photons over many energy ranges. One of the unique properties of uranium is that at about 200 eV it has a very large index of refraction coupled with very modest absorption. As can be seen in the delta-beta scatter plots¹ (Fig. 1 and 2) for 220 eV (56.4 Å) and 277 eV (44.7 Å) respectively no other element has a delta as high, and its beta is remarkable low. The beta of uranium is less than one-quarter of Ni and other 3d elements and less than one-sixth the value of Ir and other 5d elements. There really is no other element even close to it on the scatter plots.

Several groups realized that these unique properties might make some impressive optical devices possible. A group at MOXTEK investigated the preparation of U/Sc multilayer mirrors in the water window in 1993-5.² This approach relies on the low absorption of uranium to allow many layers in a multilayer to participate in multilayer reflectivity and uses the high delta to produce high amplitude reflectance at each interface. The BYU members of our group used uranium in the late 1990's to prepare near-normal-incidence mirrors with high reflectance at 41 eV and low reflectance at 21 eV.³ Two years ago, March 25, 2000, the IMAGE satellite was launched carrying three uranium-coated mirrors we designed and prepared for the EUV instrument.⁴

A third potential application of uranium is for broadband, low-angle-of-incidence mirrors for the 100 to 300 eV range. It is this application which we are exploring here. Cash had performed calculations in the mid 1990s indicating that uranium might produce phenomenal single-surface mirrors for x-ray astronomy, perhaps twice as reflecting as the nickel, gold or iridium mirror traditionally used for low-angle reflectance.

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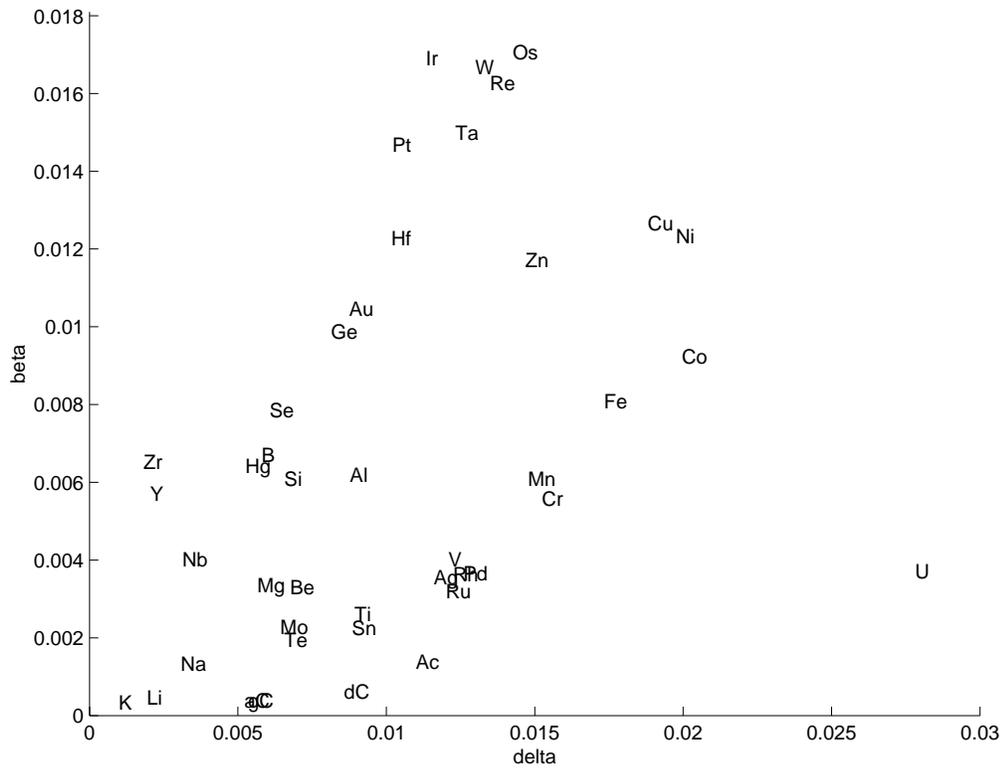


Figure 1: *delta*-beta scatter plot at 220 eV.

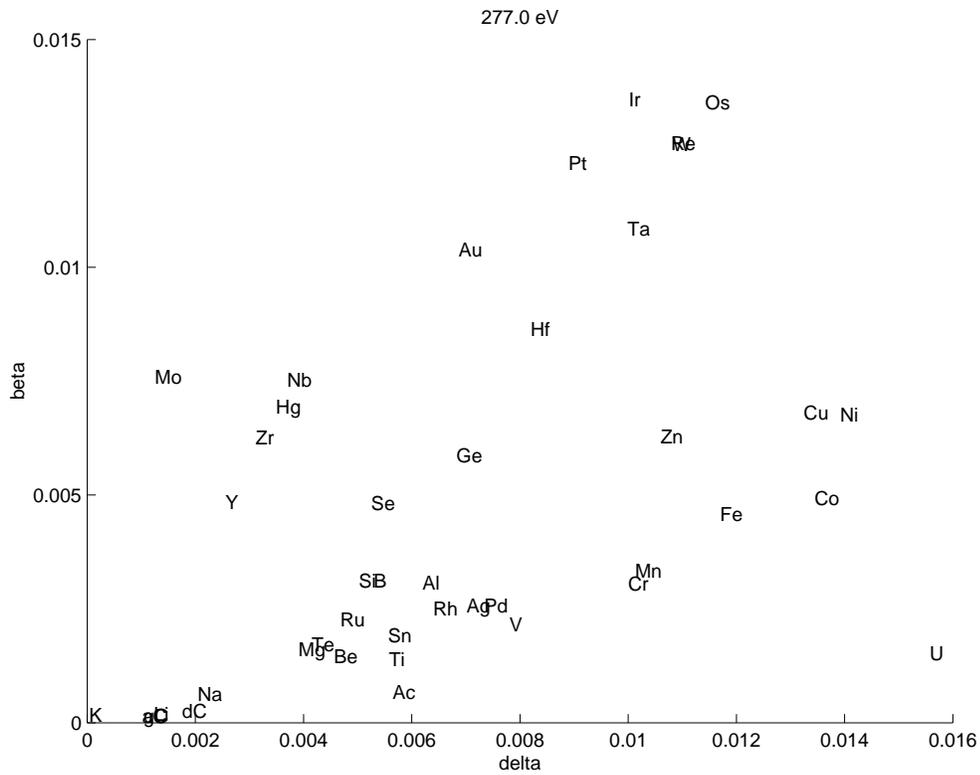


Figure 2: *delta*-beta scatter plot at 277 eV.

In Fig. 3 a and b we compare the computed reflectance of these materials at a grazing angle of 5 and 10 degrees from 30 to 530 eV. If the promise of uranium could be realized the reflectance of low angle mirrors could roughly double what is currently obtained for the range of about 150 to 300 eV. The curves for these plots were computed at the CXRO website http://www-cxro.lbl.gov/optical_constants/mirror2.html for the option “thick mirrors”, that is, single surface mirrors. Note that, since these curves are computed, actual thin films could be expected to show different reflectance.

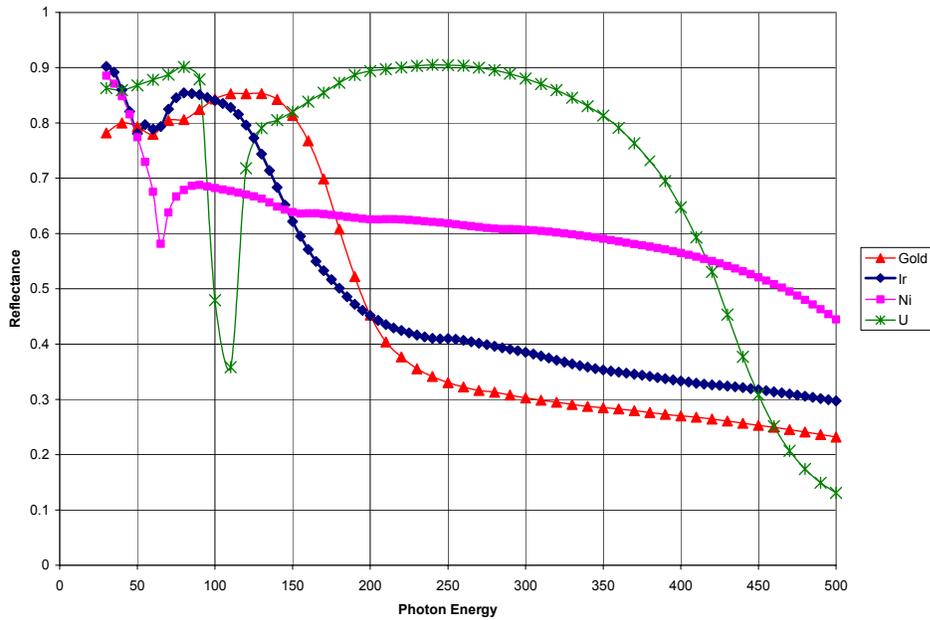


Figure 3a: Reflectance (unpolarized- computed from atomic scattering factors) versus photon energy at 5 degree from grazing.

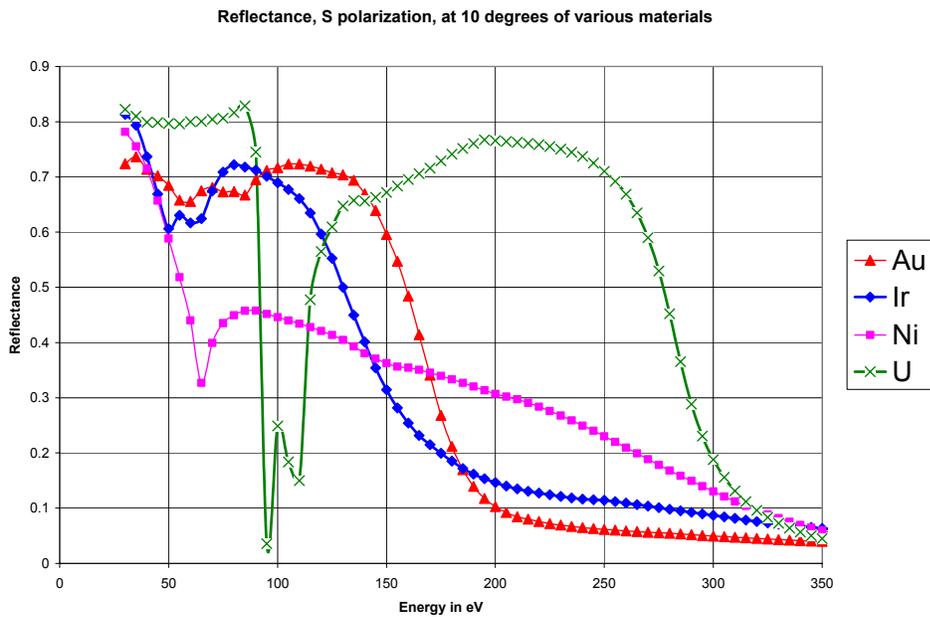


Figure 3b: Reflectance (computed from atomic scattering factors) at 10 degree from grazing.

This spectral band has been ignored somewhat by x-ray astronomers, because many sources are cut off by interstellar absorption. However, one can see out of the galaxy in this band at the poles, and it is rich in astrophysical diagnostics. The band will become even more important as x-ray observatories start chasing high redshift targets. Thus the improvement from the coatings will be of major significance for some future x-ray observatories.

Our two groups began an effort about three years ago to see if the promise of uranium for low-angle mirrors could be realized. We present here computational analyses and our first experimental results.

1.2 A challenge to realizing uranium's promise: oxidation

Uranium may have outstanding optical properties, but several of its material properties caused us to pause. As many investigations over the past several decades have shown, the best materials for stable, high-reflectance multilayers are traditionally refractories, like tungsten, molybdenum and carbon, or elements with strong covalent network structures, like silicon, which inherently possess very low diffusion rates. Uranium's melting point is 1405 K. This is just a little higher than copper (1376) or gold (1337). These were found to be unsuitable when used x-ray multilayers together because of their high tendency for mutual interdiffusion. Pew, et al.² have shown that U/Sc ML are stable, however.

In addition, uranium 238 is mildly radioactive with a 4.5×10^9 year half-life. Radioactivity of the films will not a real issue for their use in soft x-rays. It is not easy to measure radioactive decay from our samples. We use depleted uranium which has less than 0.2% U-235. In the IMAGE mission the total thickness of the U layers were about 500 angstroms and counts were less than background (~ 1 decay/s/cm²). Single surface low-angle mirrors will use less than this. ThF₄, in thicknesses which produce many more decays, has used for decades in many optical multilayers.

More detrimental for mirrors, uranium is chemically active. Uranium is subject to extensive oxidation in laboratory air, about 60 angstroms in 13 min, and more than 130 angstroms in a day.⁵ The density of uranium atoms in its oxides is less than in the untarnished metal and we anticipated that such layers would be less reflecting. Calculation of the effect of UO₂ tarnish layers of various thicknesses from 0 to 100 Å on the low-angle reflectance of an infinitely thick uranium thin film is shown in Fig. 4.

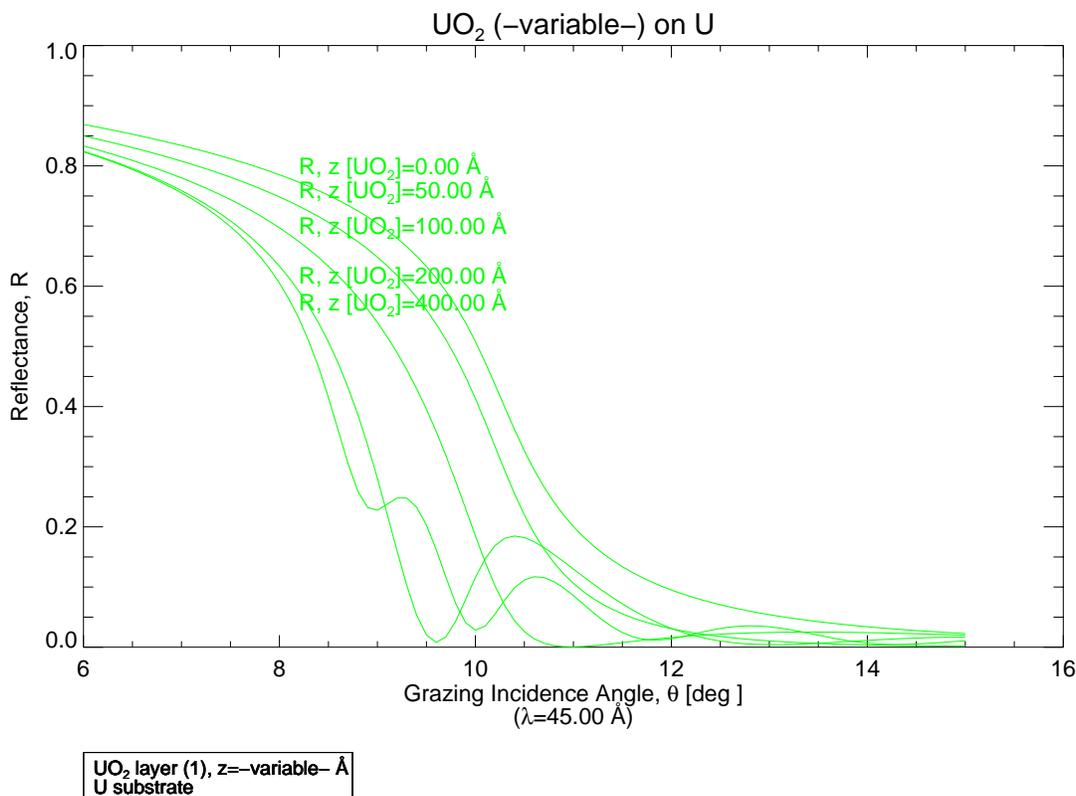


Figure. 4: Computed reflectance (0 to 1) of oxidized uranium for various thicknesses of the uranium dioxide.

In these calculations the uranium oxide which forms is assumed to be smooth. In fact, the surface might be expected to roughen somewhat as a tarnish film forms and if there is a volume expansion associated with oxidation. The expected ratio of the thickness of a uranium dioxide thin film to the uranium thin film from which forms is 2 to 1, assuming bulk densities.⁶ The optical constants for UO_2 were estimated using the CXRO website. They are based on the atomic scattering factors for U and O, the CRC handbook⁷ density of uranium dioxide of 10.96 and the ratio of 1 to 2 for U to O (proper stoichiometry) in the compound. Note that UO_2 can tolerate substantial nonstoichiometry.⁸

We decided to investigate the possibility of trying to block the oxidation of uranium in either of two ways, first, by overcoating the uranium with an inert, protective coating, and second, by alloying the thin film with more inert elements.

Consider the first approach, using overcoats to block uranium oxidation. Notice in the lower left corners of Fig. 1 and 2 (delta-beta scatter plots) the low absorbing elements at 220 and 277 eV, respectively. Of them carbon stands out as most suitable (transparent and inert) and that is what we chose to investigate. Carbon has been used as a spacer layer in EUV and soft x-ray optics. Carbon-based and polymeric windows are used in this region.^{9 10} It may be that carbon can retard the oxidation of metals it is deposited on. A knowledgeable observer may note that it may also be possible to make a compound of some of the other elements which have low beta and one or more of these might be suitable protective overcoats. This is true but beyond the scope of our investigation. Also, we note that, below its K edge at about 185 eV, boron or B_4C could be used. The reason that carbon is so transparent at 277 eV is that its K absorption edge is only a few eV higher.⁷

While a carbon film, of the correct structure and sufficient thickness, deposited on top of the uranium might retard the formation of uranium oxide, the carbon layer will unfortunately reduce the reflectance of the uranium. This is true even just below the absorption edge of carbon. Since we anticipated that the thickness of the carbon must be at least one hundred to several hundreds of Å to prevent the diffusion of oxygen into the uranium, we expected that thin film interference could be important. We employed IMD¹¹ to compute the expected reflectance of such a composite for various thicknesses of the carbon. This thickness was optimized by making contour plot of the computed reflectance as a function of angle and carbon thickness. See figure 5. We thus had a target thickness for the overcoat of 100-200 Å.

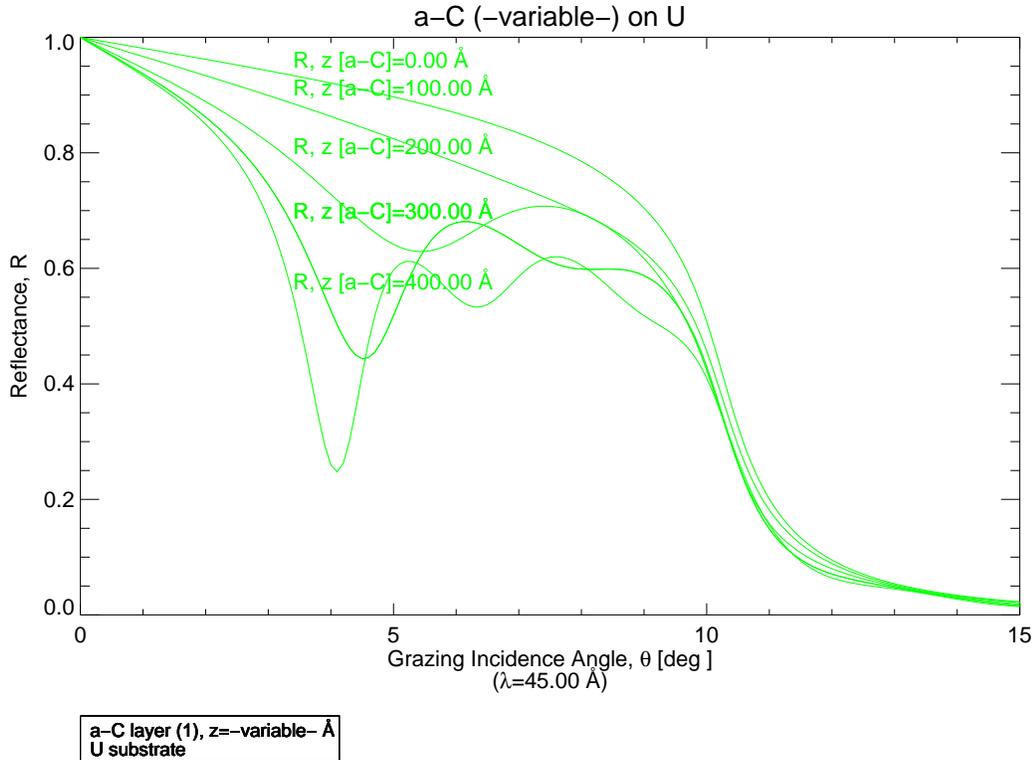


Figure 5: Computed effect of a-carbon overcoat on the reflectivity of a thick uranium layer. (IMD)

We also investigated under what circumstances a multilayer would be, in principle, superior to a bilayer. We could find no multilayer designs which are superior to a single layer if the goal is high broadband (from 125 to 350 eV) reflectivity. However, over narrower ranges they could have a modest roll to play.

Turn now to the second approach, alloying. Alloys can be more inert than the parent metals. Stainless steel, for example, is mostly iron but the addition of small amounts of chromium and nickel greatly changes the gross oxidation behavior of the composite. Nickel and platinum are two elements which have high deltas in the range of investigation (see fig. 1 and 2) and are considerably nobler than uranium. Nickel oxide, NiO, forms only slowly on nickel and is only a few tens of angstroms thick in a period of many months. Platinum stops with a single layer of oxygen on its surface. It might be possible to introduce enough nickel or platinum atoms in a uranium layer to slow substantially or to stop the oxidation of the uranium beyond a certain point by obstructing the access of uranium and oxygen to one another. In addition, electron transfer occurs in many intermetallic compounds, particularly when one is more electropositive than the other, as uranium is to nickel or platinum. This electron transfer might retard the activity of uranium. In fact, a number of elements have been considered for improving the high temperature properties of uranium, including its corrosion resistance. The most studied are γ -uranium miscible systems, including Zr¹², Mo, Ti, Nb, and Ru. Cordfunke (ref. 8) states that the alloying of U with small amounts of Pt has been studied, not so much as an anticorrosion aid, but as an alpha-alloy stabilizer, preventing the deformation of uranium as it is heated through the α to β transformation temperature. There is some hope that small amounts of alloying can help. Mulberry is the name of a U-7.5 at. % Nb 2.5 at. % Zr alloy, which is labeled the "stainless" uranium alloy.¹³ It is thus named because of its low oxidation rate, including in moist air. The question is whether such behavior is sufficient for EUV and soft x-ray applications. Passivating films a few hundreds or thousands of angstroms could be completely satisfactory for bulk sample protection and completely inadequate for EUV and soft x rays. As Fig. 4 suggests tarnish films which are as thin as a few hundreds of angstroms have a dramatic effect on reflectance in this range.

Concerning nickel, the nickel-uranium compound U₆Ni is reported to be exceedingly brittle.¹⁴ This is a sign of significant electron transfer in compound formation, which could aid in reining-in uranium's propensity to oxidize. As further evidence of reduced activity, U₆X (where X is some metal) compounds are reported in ref. 14 to be generally inert in both dilute and concentrated nitric acid. This is indicative of their resistance to bulk oxidation. It can, thus, be hoped that alloying may slow uranium oxidation to the point that the alloys may be used without a passivating overlayer or with only an ultrathin one. We did calculations using IMD which showed that reflectance could be expected to remain high for uranium films alloyed with 10 to 20% of nickel if there is no oxidation. The investigation we conducted on alloys was brief, only preliminary, and more work needs to be done. The three samples were prepared by cosputtering nickel and uranium and their oxidation studied by ellipsometry as is described below in sections 2.2 and 3.2.

2. EXPERIMENTAL: SAMPLE PREPARATION

2.1. A carbon (barrier layer) uranium bilayer.

Uranium and carbon were deposited sequentially without breaking vacuum using a two-gun, DC-magnetron sputtering system at BYU.¹⁵ The substrates were held in the same apparatus which had been used to sputter the EUV mirrors for the IMAGE mission. See figure 6. Sputtering is upward. The samples are attached to the aluminum block via small adjustable tabs. The block is placed in the spinning holder using screws and small washers. More details are available in ref. 3. The holder can be spun rapidly (many times per second) using a motor internal to the vacuum system to more evenly expose the samples to the atoms emerging from the sputter gun to build up a spatially uniform thickness coat.

The base pressure of the chamber was $1-2 \times 10^{-6}$ torr, and was achieved by cryopumping. For sputtering a blocking shutter was swung over the port leading to the cryopump. We backfilled with 99.999% pure Ar to a pressure of approximately 1×10^{-3} torr. The pressure of the system was measured using an Edwards Model 655 Barocel™. This is a 100-millitorr full-scale vacuum/pressure transducer, or capacitance manometer. The composition of the gas atmosphere during the entire process was monitored using a Ferran Scientific Millipole (quadrupole mass) Analyzer (MPA). The system also measured the total pressure of the system. We recorded this pressure but used the Barocel to measure the absolute pressure of the chamber. The MPA reading was about a factor of 2 higher than the manometer and tracked consistently with it as the chamber pressure varied.

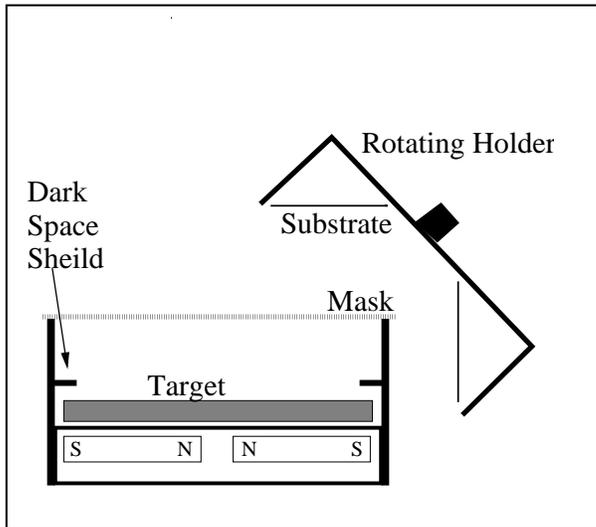


Figure 6: Sample holder on revolving carousel rotates above the sputtering gun.

Spatial uniformity of the coating on the sample was achieved by masking the area of the sputter guns. The shape of the mask had been determined previously by an iterative process of measuring the uniformity across the sample and then adjusting the shape of the mask and then repeating the process during the preparation of the EUV mirrors.^{3,4} The uniformity had been optimized by measuring the peak position of bilayer U/Si multilayer samples at several places along their length using low-angle x-ray diffraction (XRD) using a Scintag 2200. We used the same measurements to determine the bilayer period thickness by iteratively fitting the measured diffraction peaks to those computed using the IMD program. Previous to sputtering the samples for this study the sputter rate of the uranium had been determined several times over a couple of years. The thickness of the uranium was determined by the time the substrate was over the uranium gun. In the case of sample U9C01 and U9C06 this was 280 and 310 seconds, respectively. With a sputter rate of 0.716 \AA/s for uranium the target thicknesses of uranium were 200 and 220 \AA , respectively. The thickness of the carbon was also determined by the time the substrates were over the carbon gun, 667 and 560 s, respectively. The target thickness of carbon was 100 \AA based on an assumed sputter rate for carbon of 0.15 \AA/s . The thickness carbon was only approximate since the sputter rate of carbon had not been measured for over a year.

The two samples prepared at BYU in the fall of 1999 were sent to CASA. The preparation of the first sample, U9C01, was flawed; the sample was not spun in the substrate holder. This was an oversight but the sample was studied nevertheless. The sample clearly had a uranium coating and a carbon overcoat, but the thickness of each was uncertain. The second sample, U9C06, was deposited without problems. Both samples were stored in laboratory air in Provo and shipped in plastic (Fluoroware) containers. They were stored in a dry box at the University of Colorado and measured at the same time, about 5 weeks after the first was prepared and within one week after the second.

2.2 Uranium alloy samples

For the second approach, the uranium target was compositionally modified with nickel. To do this, small pieces of nickel were placed on the uranium target covering part of racetrack and making electrical contact with the target. The racetrack is the part of the target that is most heavily eroded in sputtering. It is known that when the plasma is ignited that atoms will be sputtered both from the target and conducting pieces of modifier. Both these sputtered atoms will be incorporated into the deposited film. For the initial sample sputtered about 20% of the surface of the racetrack was covered with nickel. For later samples 60% of the target was covered. The nickel content of the films was not measured. When, and if, the Ni work is repeated, we will use XPS which is now available to us to determine the extent to which Ni is incorporated into the films.

3. CHARACTERIZATION

3.1 Ellipsometry

After the alloy samples were prepared they were stored in air. The thickness of the oxide which formed on them was determined as a function of their time in air using spectroscopic ellipsometry (John A. Woollam model M44). The oxidation rate was observed to not differ significantly from pure uranium films within the accuracy of our measurements. We needed optical constants for the oxide layer to model the samples' ellipsometry measurements. We used the values for UO_2 which David Oliphant had determined earlier for the tarnish layer that forms on uranium films.⁵ We obtained reasonable fits for the oxide using UO_2 constants. These suggest that the initial layer which forms on the alloy films is predominantly UO_2 and that, either the sputter rate for nickel was much smaller than uranium, or forming a nickel-uranium alloy does not significantly slow the oxidation of ultrathin alloy films. That is, while Ni definitely alters the large-scale oxidation of bulk U-Ni, for example in U_6Ni (see the discussion above), its blocking effect may not become active until a large barrier oxide film builds up. If this oxide film proves to be too thick, then reflectance could be lower. As we noted previously, even ultrathin oxide films can have an important effect on changing EUV and soft x-ray reflectance.

3.2 X-ray diffraction

The thickness of relatively thin films on a smooth substrate such as a silicon wafer can be determined by matching x-ray diffraction (XRD) peaks to a model. A Scintag x-ray diffractometer measured the relative intensity of peaks being diffracted from two U/C multilayer samples (U9C04 and U9C05) at Cu K_α . The data was fit using IMD to determine the thickness of the carbon and the uranium in the two. Thus, for the first sample analyzed at 44.7 \AA , U9C01, the approximate thickness of the uranium was calculated from the previously determined deposition rates and the carbon rate was only known approximately at the time of deposition. By the time we did the second sample, U9C06, the rates were much better known.

3.3 At-wavelength reflectance

The reflectance at 277 eV (C K_α , 44.7 \AA) was measured using a grazing incidence monochromator equipped with a Manson x-ray source with a carbon anode at CASA (University of Colorado, Boulder). A flowing, gas-proportional counter detector was used to measure the counts coming from the x-ray source. The samples were measured at angles ranging from 1 to 12° from grazing. These measurements are shown in Fig. 7, curves a and b. The reflectance curves are not accurate below about 5° because the beam over-illuminates the sample at low angles. That is, at low angles the beam spills off one of the edges of the sample. Therefore, the apparent decline of reflection at low angles is, at least in part, an artifact of measurement.

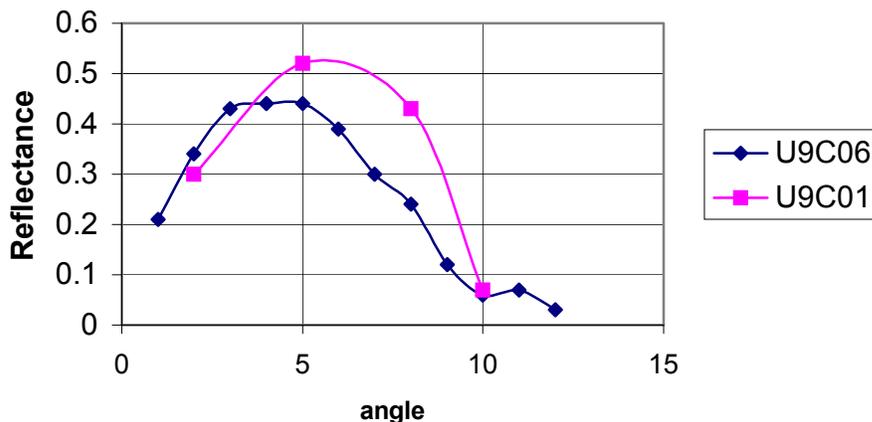


Figure 7: Measured reflectance at 44.7 \AA (277 eV) of two uranium samples coated with carbon as a function of angle. The lines are drawn only as an aid to the eye.

4. DISCUSSION OF MEASUREMENTS

The first thing to note is that the reflectance from the two mirrors is very high. Sample U9C01 is about twice as high as nickel mirrors previously prepared and measured at CASA. This is a significant increase. Particularly since two reflections from mirrors are required for low angle imaging. Nickel is the material with the previous record for highest low angle reflectance at 10° over this portion of the soft x-rays. In designing future broadband, low-angle mirrors a coating based on uranium as the reflecting surface should be considered.

The second thing to note is that the measured reflectance of samples U9C01 and U9C06 are somewhat less than the computed reflectance for 100 to 200 Å of carbon on uranium. This may be due to a variety of measurement- or sample-related reasons. Sample-related reasons will be discussed below. The important point is this there is a need for further development with the strong possibility of even better mirrors being produced.

A third thing to note is that sample with the higher reflectance was U9C01. This was the “defective” sample, the one prepared without the sample holder spinner being on. It can be higher than the more carefully prepared sample if: 1. It has thicker (or thinner) uranium and that helps. 2. It has thicker (or thinner) carbon on top and that helps. 3. The uranium is (partially) oxidized and that is, nevertheless, at least partially adequate.

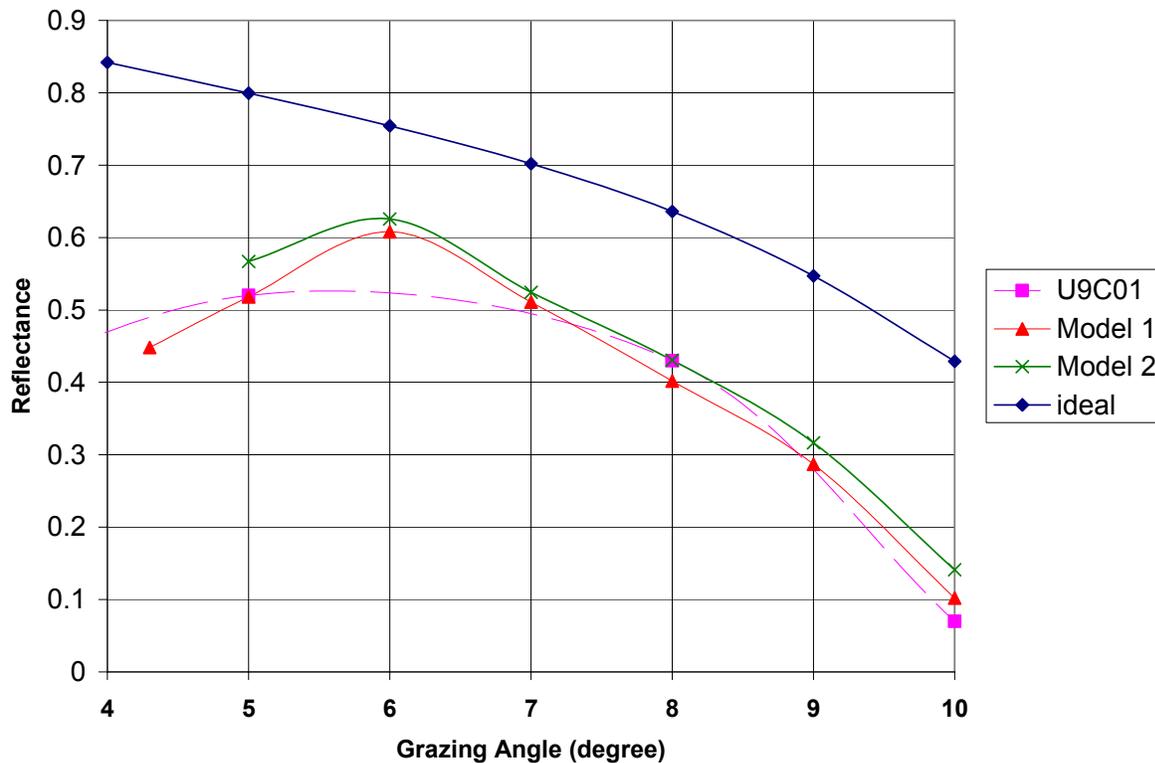


Figure 8: Reflectance of U9C01 and various models (unpolarized, computed from atomic scattering factors-IMD) versus grazing angle. Ideal is 100 Å a-C/20 Å UO₂/ 200 Å U on 18 Å SiO₂ / Si substrate and 12 Å of roughness. Model 1 has 300 Å a-C/ 160 Å UO₂/ 75 Å U on 18 Å SiO₂ / Si substrate with 37.5 Å roughness. Model 2 has 300 Å a-C/ 100 Å UO₂/ 25 Å U on 18 Å SiO₂ / Si substrate with 12.5 Å roughness.

In Fig. 8 we show the expected reflectance for U9C01. Its reflectance is higher than the measured sample. As was mentioned above the beam size being larger than the sample can be a problem at low angles. In addition, surface roughness can also contribute to the nonspecular scatter of light away from the detector.

There are several material issues which can contribute to the difference. These include the effect of differing uranium layer thicknesses, oxidation, surface roughness, or discrepancies in optical constants. The formation of an oxide though the carbon capping layer may be possible. The oxidation of uranium to UO₂ should not significantly decrease the

reflectance of the sample below about 8°. See figure 4. The calculations show that at low angles the reflectance of UO_2 is comparable to that of U.

Comparing figures 4 and 5 reveals another interesting fact. The computed reflectance of UO_2 is greater than the computed reflectance of U with a barrier layer of $>200 \text{ \AA}$ of sputtered carbon. The carbon layer was deposited on the U to prevent UO_2 from forming, but after modeling the effects of oxide on the reflectance we see that it may be acceptable to allow the surface of the uranium to form a native oxide of any thickness if a thin carbon barrier is not adequate to stop oxidation. This would only be beneficial below 8° grazing. Above 8° grazing a capping layer to prevent the oxidation of the U should increase the reflectance of the sample.

We varied the thickness of layers and roughness to obtain the model curves in Fig. 8. To bring the reflectance down to the measured values at 5° as show in the figure it was necessary to increase the carbon thickness in the model to about 300 Å and to add a layer of UO_2 under it. That is, we had to assume that the carbon was much thicker than the target and a portion of the uranium oxidized. The precise values are shown in the figure caption. We do not assume that the carbon layer was as thick as the model would indicate, but because the sample was not rotated this is possible. To bring the thickness down at 10° as far as shown in the figure the roughness had to be $>35 \text{ \AA}$. This may be unphysical. The roughness of UO_2 in the 200 Å range is about 10 Å rms as measured by AFM. Thin film optical interference effects due to UO_2 may be present. Compare the lower curves in Fig. 4 with U9C06 in Fig. 7. More samples need to be prepared, studied and their compositions and structures determined via XRD, sputter XPS etc.

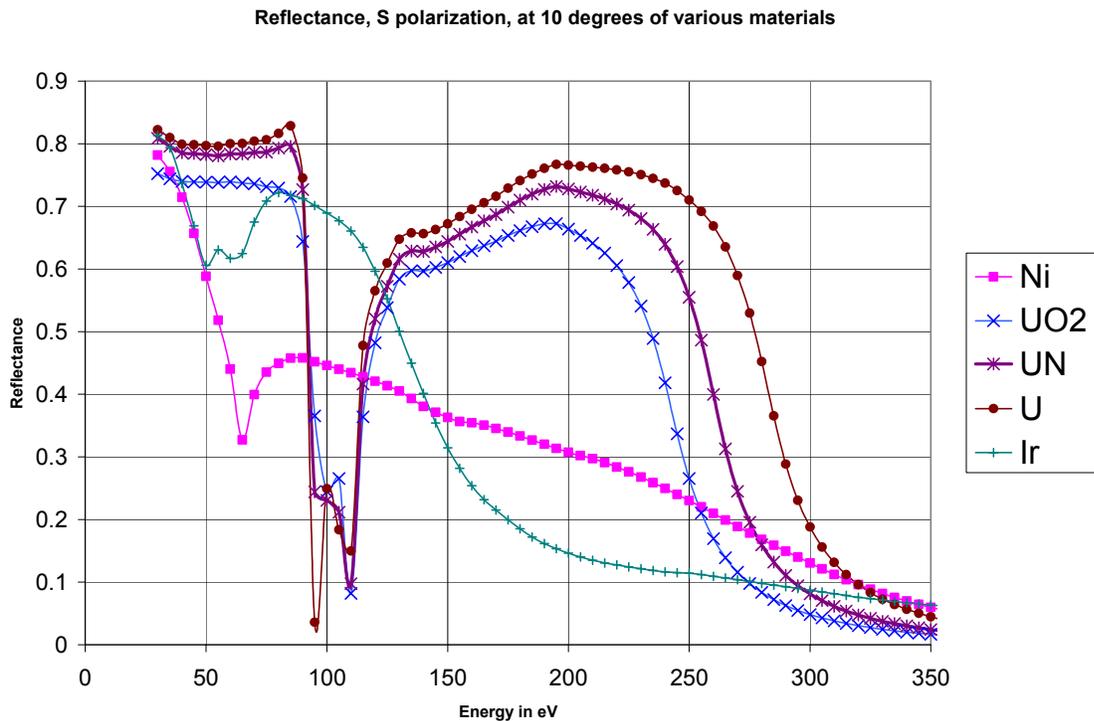


Figure 9: Computed reflectances (CXRO website¹) versus photon energy at 10 degree from grazing. Reflectances computed from atomic scattering factors and the atomic densities in each compound as determined from handbook densities and formulas (stoichiometries).

4.2 Alloy films:

There is insufficient data to conclude that alloying will not stabilize uranium surface against more oxidation than is tolerable for soft x-ray applications. Certainly thin films of mulberry and other alloys shown to have good resistance to oxidation in bulk form could be prepared and their oxidation behavior investigated. If the nickel work is repeated the composition of the deposited films needs to be ascertained by XPS.

4.3 Compounds.

Adding metals with high delta to uranium may be a way of stabilizing it. In addition, it may be that adding lightweight elements such as nitrogen, oxygen or carbon to uranium could be useful in preparing surfaces with high uranium density for soft x-ray reflectors. See figure 9.

The properties of many bulk materials have been reported but few thin film studies have been reported. UN has a bulk density of >14 and it is report to have better resistance to oxidation than uranium metal. Specifically, it is reported that UN powder or single crystal) is stable in moist air at room temperature and that UN powder is stable in boiling water for days in the absence of oxidizers.¹⁶ Adam Fennimore sputtered and studied TEM cross sections of ultrathin UN films for EUV applications. UN was deposited by reactively sputtering uranium in a nitrogen-containing atmosphere.¹⁷ This investigation could profitably be renewed. In contrast with nitride uranium carbides are reported to be much less stable.¹⁸

Oxygen naturally combines with uranium surfaces exposed to the environment. The uranium dioxide formed has been used for the top layer for EUV mirrors.^{3,4} Its optical constants have recently been determined through the 21 to 270 eV range and its use for low-angle soft x-ray mirrors is being considered.¹⁹

5. CONCLUSIONS

Carbon coated uranium layers were found to be a better low angle reflector at 44.7 \AA than any material currently used. Developing coatings based on uranium should be a priority for all future missions where broadband, low-angle, soft x-ray mirrors are required. Even though the measured reflectance was lower than predicted the analysis of the data has exposed another alternative. The calculated reflectance of UO_2 is comparable to U below 8° grazing angle. Three courses of research are available; prepare and measure the reflective properties of UO_2 or UN thin films, prepare and measure the reflective properties of U alloy thin films or better understand why the original C/U samples were not as reflective as the theory predicted. Despite the somewhat lower than expected reflectance the measured results provide a promising alternative to current mirrors, particularly because their reflectance can be expected to be even higher with optimization and exploring the properties of other uranium compounds and alloys. Uranium radioactivity is not an issue for mirror applications in soft x-rays.²⁰

ACKNOWLEDGEMENTS

We are grateful to James Reno for sputtering the first carbon/uranium film studied and to David Oliphant who measured the thickness of the oxide on the alloy samples as a function of time. We also acknowledge grateful the financial contributions of V. Dean and Alice J. Allred and Marathon Oil Company (US Steel) of gifts to Brigham Young University for thin film research.

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Citation style for this publication:

David D. Allred, Matthew B. Squires, R. Steven Turley, Webster Cash, and Ann Shipley, "Highly Reflective Uranium Mirrors for Astrophysics Applications," in *X-ray Mirrors, Crystals and Multilayers*, Andreas K. Freund, Albert T. Macrander, Tetsuya Ishikawa, and James. T. Wood, Editors, Proc. SPIE **4782**, pp. 212-223, SPIE, Bellingham, WA, 2002.