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IN NORMAL INCIDENCE COPPER MIRRORS*

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ABSTRACT

Water-cooled copper mirrors used at near-normal incidence on two beam lines at the NSLS are observed to undergo severe degradation upon exposure to the direct SR beam. These mirrors are used on beam lines designed to utilize radiation in the wavelength regions longer than 100 nm and are coated with a UV reflection-enhancing coating, consisting of one or more bilayers of aluminum with a MgF_2 overcoat. Beamline performance degrades very rapidly following installation of a new set of mirrors. Analysis of the mirror surfaces by various non-destructive techniques indicates severe degradation of the coating and surface along the central strip where most of the x-ray power is absorbed from the beam. In one case where the mirror had three bilayer coatings, the outer coating layer has disappeared along the central strip. Rutherford backscatter measurements indicate compositional changes between layers and confirm the existence of a carbon deposit on the surface. Thermal modeling suggests that most of the damage is caused by direct photon interaction, since the temperature rise in the energy deposition region is small.

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Introduction

The U9 beamline at the NSLS VUV storage ring is unique among the other beam lines in that it utilizes metal mirrors exposed to the direct SR beam at near-normal incidence. Subsequent to installation of the first set of copper mirrors on U9A and U9B in April 1982, the performance of each mirror was noted to degrade very rapidly with time upon exposure to the SR beam. Several improvements were made in the design of the substrate and coating on later versions of each mirror based on the experience gained from the earlier mirrors. An analysis of the damage observed on the first set of mirrors is presented here, and the subsequent changes in the mirror design are discussed.

Mirror Design Parameters

The U9 beamline consists of two branches - U9A and U9B - each with its own first mirror designed to collect about 30 mrad of incident beam direct from the source. The U9A mirror is used at a 7° angle of incidence; the U9B mirror is used at a 45° angle of incidence. The damage observations discussed below will concern mainly the U9A mirror because it exhibited much more catastrophic damage than the U9B mirror. Speculation on the reasons for this difference between the two mirrors will be discussed later.

The U9A mirror, shown in the damaged condition in Fig. 1, consists of an OFHC copper substrate with a reflecting surface that was prepared by diamond flycutting and post-polishing. The blank size is 100 mm x 50 mm x 25 mm with a length of copper tubing brazed onto the rear surface to allow for water cooling. During the period of operation discussed in the present paper, the maximum heat load on the mirror never exceeded more than a few watts, so the

mirror temperature was expected to always remain near ambient. The U9A beamline is designed to operate in the windowed ultraviolet at wavelengths longer than 105 nm. For this reason the copper mirror was coated with a standard UV-enhancing reflection coating, which consists of an aluminum thin film overcoated with a MgF_2 protective layer[1]. Following an initial exposure period, the mirror was cleaned of carbon contamination and recoated with two additional sets of Al/MgF_2 bilayers over the original Al/MgF_2 coating.

Exposure History

The U9 mirrors were the first to be exposed to beam from the VUV storage ring. They were installed in April 1982 during the commissioning of the storage ring and were removed in March 1983 during a major shutdown period. At this time they were cleaned by an oxygen plasma technique [2] and recoated. Uncertainty in the coating parameters for the U9A mirror resulted in application of a third Al/MgF_2 bilayer to that mirror alone. One serious difficulty in the coating process was that the actual coating thickness parameters were not well documented. Both mirrors were reinstalled in the U9 beamline in May 1983 and were in use through February 1984, when they were removed and replaced with a new set.

During the initial operating period of the storage ring, detailed records of stored beam current were not kept as they are today. It is difficult to assess the precise total x-ray fluence received by the mirrors over the early time period. Our best estimate is that the original coating was exposed to an average 20 ma beam for 522 hours, resulting in an integrated exposure of 10.4 amp-hours. Record-keeping was much improved during the second operating period, and we estimate a total integrated

exposure of 118.0 amp-hours for that period. In terms of total time integrated energy density at the center of the U9A mirror, the two time periods give the following:

- (1) April 1982 - March 1983: $0.67 \times 10^6 \text{ J/cm}^2$
- (2) May 1983 - February 1984: $7.62 \times 10^6 \text{ J/cm}^2$

One observes that over the first time period the total peak fluence was a little less than one megajoule per cm^2 , while over the second time period the total fluence was a bit more than a factor of 10 greater, i.e., 7.62 megajoules per cm^2 . The resultant damage was observed to be significantly different between the two periods. The initial exposure resulted in a noticeable dark streak down the center of the mirror where the peak intensity was located [3] which could not be completely removed by plasma cleaning. The second exposure resulted in catastrophic damage to the U9A mirror as in Fig. 1, but only in cosmetic damage to U9B. The surface in U9B appears to have been altered by the exposure, but it remains attached to the substrate.

Damage Morphology

The catastrophic damage on the U9A mirror is shown in greater detail in Figs. 2 and 3. Figure 2 is a x25 magnification of one edge of the damage strip. Quite evident is the fact that some of the coating layers are peeling away from the surface in the region of maximum power density. There is a transition region surrounding the "burned" region in which the coating appears to be blistered and filled with subsurface bubbles. At higher magnification (x80) using differential interference contrast, the large

blisters appear quite regular, while the surface exposed by the peeling coating appears quite rough. Optical profilometer measurements of the surface roughness inside and outside the burned area confirm the rougher nature of the burned region. Representative profiles of each region are shown in Fig. 4. Far from the central burned area the surface has a roughness on the order of $24\text{\AA}$ RMS, while within the burned area the roughness is on the order of $63\text{\AA}$ RMS. An increase in surface graininess is evident in the profile of the burned region by a disproportionate number of spikes at heights below the zero mean level.

Rutherford Backscatter Analysis

The one question that arose immediately upon visual inspection of the damaged region was: How much of the original coating remains on the surface inside the burned area? But corollary to that is the question: What is the actual composition of the original surface? To answer these questions, we employed the technique of Rutherford Backscatter Analysis (RBS), using 2 MeV alpha particles to probe the elemental composition as a function of depth below the surface in the damaged and undamaged regions. Backscatter spectra from two regions on the surface, one outside the burned area, the other inside of it, are superimposed in Fig. 5. The general features evident in each spectrum are the weak individual peaks produced by the Al, Mg, and F elements riding on top of the strong signal from the Cu substrate. The high energy edge of the Cu signal is shifted to higher energy in the damaged region, indicating a loss of material from the burned area. Figure 6 shows the residual signal for each region after a best-fit curve to the Cu background is removed. The three Al layers dominate the undamaged spectrum, while only two Al layers are evident in the damaged region. The F signal

has disappeared completely from the damaged region. Also evident in the undamaged region is a peak due to backscatter from carbon on top of the outermost layer. No evidence for a corresponding oxygen signal is seen. Also, the carbon signature is not evident in the damaged region.

A modeling effort was employed to analyze the RBS spectra as a series of thin film layers of various compositions on top of the copper[4]. Owing to the complexity of the layer structure, the compositions indicated in Fig. 7 can only be considered the best-fit approximation to the actual structure, based on the limitations of the model and the measurement statistics. One limitation in the model is the lack of sensitivity to the outermost MgF_2 layer. The RBS signature from this layer is buried underneath the strong Al signal. Consequently, we indicate that the outermost layer in the undamaged region is Al combined with a thin C contamination layer. The damaged region model does not fit as well to the measurements as does the undamaged region. This is most evident in the long tail of the lower Al peak toward low energies, and the corresponding roll-off at the top of the Cu edge. The nature of the Al tail and Cu edge indicates that the Al has diffused down into the Cu on the order of $1000\text{\AA}$. However, a simple model of a Cu-Al interface layer with a smoothly varying Cu-to-Al ratio is insufficient to fit the observed data. We surmise that the Al is diffusing preferentially down grain boundaries in the Cu substrate, resulting in a laterally inhomogeneous surface. The present model is not capable of handling such inhomogeneous surfaces, and we cannot expect a detailed fit to the data. One also notes that the model confirms the disappearance of the fluorine from the damaged region. The outermost Al layer is gone, as well as the two outermost MgF_2 layers (assuming the original design composition was correct). The bottom Mg layer is still within the damaged region, because it is required to provide

the separation between the two Al peaks. The exact nature of the Mg/Al transition region structure is not known from these measurements.

Discussion

To assess the possibility of temperature as the driving mechanism behind the damage observations, the temperature rise in the thin film layers was estimated from a knowledge of the x-ray absorption lengths based on the data contained in the Henke tables[5]. At the critical wavelength of 30Å for the UV ring, the absorption length for Al is 2850Å, while for MgF_2 it is 6000Å. Based on an estimate of the average power density in the center of the mirror, the temperature rise in the layers is estimated to be only 0.01°C, so temperature alone is insufficient to drive the damage mechanism. We suspect that the real mechanism is a photon-assisted diffusion process which is dependent upon the large, local, non-equilibrium temperature distribution in the lattice surrounding an atom that has been photoionized by the incident beam. Such an energy deposition process over long time periods could lead to the generation and growth of lattice defects and grain boundaries in the Cu, with subsequent enhanced diffusion of the Al down the grain boundary interfaces[6]. A similar energy deposition process may also be responsible for the liberation of fluorine from the MgF_2 material. Decomposition of bulk alkali halides upon exposure to UV and x-ray beams has been noted in the past[7].

Based on the experience and limited analysis done upon the first removal of the U9 mirrors, a second set was fabricated with a different surface treatment. The second set consisted of a copper substrate with an electroless nickel plating (ENP) several thousandths of an inch thick.

The surface of the ENP was coated again with the UV-enhanced Al/MgF₂ bilayer. Upon exposure to the SR beam, this surface was also observed to degrade very rapidly. This mirror set has not yet been analyzed, but visual observations indicate compositional changes within the ENP - Al/MgF₂ interfaces. A third set of mirrors was then fabricated, one with ENP over the Cu substrate, the other with electroplated Ni over the copper. Both mirrors were then coated only with a thin gold layer. No MgF₂ overcoat was used on this third set. The performance of this third set is far superior to that of the previous two. It appears that local chemistry associated with the breakdown of the Al/MgF₂ bilayer is responsible for the severe degradation in the performance of the first two sets of mirrors.

Conclusions

Results from the RBS analysis of the first set of damaged mirrors indicate the unsuitability of Al/MgF₂ bilayer coatings exposed directly to the SR beam. In addition to the decomposition of the MgF₂ material exposed to the x-ray component in the center of the UV ring beam, a roughening of the surface is noted and enhanced grain boundary diffusion of the Al down into the Cu is suspected. The non-thermally driven changes in the surface morphology and composition from long-term exposure to soft x-ray indicate that potential problems may exist for other coatings planned for use on normal incidence SR mirrors. The need exists for a systematic study of material damage caused by the high fluxes of x-rays that will be produced on normal incidence optics by next-generation insertion devices.

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Figure Captions

- Fig. 1. Photograph of the U9A-M1 mirror after exposure to SR beam showing location of the region of catastrophic damage on the reflecting surface. Copper tubing is brazed to the rear surface for water cooling. The abrupt termination of the damaged region in the lower left of the photo is the result of the shadow cast by the edge of the U9B mirror.
- Fig. 2. Region of catastrophic damage near the shadowed left edge in previous figure at 25x magnification. The coating is blistered and peeling in the region exposed to the highest energy x-rays.
- Fig. 3. Nomarski micrograph at 80x of a region along the damaged edge in Fig. 2. Surface appears rougher where the coating has peeled away.
- Fig. 4. Surface profile measurements made with an optical profiler of two typical regions on the U9A-M1 mirror. Upper = "pristine" region well outside the damaged area; lower = inside the region of catastrophic damage. Roughness of the undamaged region profile is 24Å RMS; the damaged region profile is 63Å RMS.
- Fig. 5. Rutherford backscatter spectra from the U9A-M1 surface. Solid line is the undamaged region; dotted line is the damaged region. Energy scale is roughly 1.4 keV per channel. Undamaged region shows three peaks characteristic of the three aluminum layers superimposed upon the strong background from the copper substrate. Damaged region shows only two aluminum peaks with the copper edge shifted to higher energy, which is indicative of the material missing from the damaged region.

Fig. 6. Subtraction of the copper background shows more clearly the differences between the damaged and undamaged areas. The fluorine and carbon peaks are prominent in the undamaged region, but are absent in the damaged region.

Fig. 7. Models used to fit the measured RBS spectra. Numbers are the layer thicknesses in Angstroms. The Mg is combined with the Al in the damaged region in the proportion Al_4Mg . Also, the Al/Cu interface is modeled as a gradual change from mostly Al to mostly Cu over a depth of about 1000Å (not to scale in the diagram).

Fig. 1

Fig. 2

Fig. 3

Fig. 4

Fig. 5

Fig. 6

U9A-M1 RBS MODEL

OUTSIDE DAMAGED AREA

INSIDE DAMAGED AREA

Fig. 7

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