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X-ray-Induced Damage Observations on ZERODUR Mirrors

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ABSTRACT

Catastrophic damage has been observed in some ZERODUR mirrors used as first mirrors in two beam lines at the National Synchrotron Light Source (NSLS). Despite the high reflectivity of the coatings used on these mirrors, a significant flux of high energy photons penetrates below the coating and is absorbed in the substrate. Although model calculations indicate that the local temperature does not increase significantly, we suspect that over long time periods the absorbed flux produces structural changes in the material, leading to a build-up of surface stress, gross figure changes, and growth of fractures. These changes are probably related to the nature of the two-phase glass-ceramic composition of the ZERODUR material. Metal mirrors and single-phase materials do not exhibit such catastrophic damage under similar exposure conditions.

KEYWORDS: Radiation damage, x-ray damage, synchrotron mirrors, synchrotron beam lines, ZERODUR, fused silica, x-rays, x-ray optics

1. INTRODUCTION

The Optical Metrology Laboratory at Brookhaven National Laboratory routinely performs surface figure and finish tests on mirrors delivered for use at the National Synchrotron Light Source (NSLS). Over the past decade we have developed a large database of mirror surface quality measurements. Most of this data is for mirrors that are delivered prior to their installation in NSLS beamlines. Once a mirror is installed in a beam line, it usually remains in place for several years. Occasionally, old mirrors are replaced by newer mirrors to take advantage of recent improvements in fabrication technology that produce better and more robust mirrors. In some cases, mirror replacement is necessitated by severe degradation of the mirror surface which manifests itself by a steady decline in the performance of a beam line system over time. We now have measurements on a number of mirrors that have been removed from NSLS beam lines after various amounts of exposure to the SR beam. In most cases we have been able to compare the before and after measurements to see what changes have occurred during exposure to the x-ray beam. Unfortunately, the exposure conditions for each mirror are all different, so no systematic analysis of damage can be inferred from these observations. The observations can only serve as a guide as to what may happen under certain conditions of exposure to synchrotron radiation.

A rather small literature exists that deals with x-ray-induced radiation damage in optical materials. Most radiation damage studies on glass have been concerned with energetic particle effects: reactor neutrons and electrons, and ion beam bombardment¹. Most of the effort related to glasses has been on quantifying the color center formation process². Very little information is available on the effects produced by x-rays alone. Franks and Stedman discuss some experimental work on various materials to be used as SR monochromator grating substrates³. Zietz, et al., exposed several materials to the direct beam from the DORIS storage ring at high energy and power and saw damage to varying degrees on all except the SiC sample⁴. MacDowell, et al., also measured the performance of a number of materials at the Daresbury SRS exposed to the direct beam and observed severe damage to the glass materials⁵. In these tests the substrates were allowed to heat up to high temperatures, so it is difficult to separate the thermally-induced damage from purely radiation-induced effects. Our sample exposures are at the other end of the spectrum from those of Zietz and MacDowell -- relatively low doses over long time periods at, or near, room temperature.

Table I is a summary of a number of observations made to date on NSLS mirrors that exhibit some kind of change in surface quality. Roughness and surface figure measurement results are listed for both the "before exposure" and the "after exposure" conditions, where possible. The surface roughness measurements were made with a WYKO NCP 1000 Surface Profilometer.

which was converted to a Micromap Promap 512 system. Most of the roughness measurements were made with the 2.5X magnification objective, which samples a region about 3 mm long on the surface, although some measurements were made with a 10x objective, sampling an 800 μm long region. The figure measurements were made with a Long Trace Profiler (LTP). Only the overall radius of curvature measurement is reported in the Table, since it is a good indicator of the internal substrate stress level. In some cases measurements of surface roughness or figure were not available and are indicated as such.

One can see from the Table that most of the damage is seen in glass or fused silica-like materials. We have seen few examples of damage in metal substrates and none in single-crystal silicon or silicon carbide. The mirror that has shown the most severe damage is the ZERODUR sphere in beam line X19A. The remainder of this paper will discuss the damage observed on this mirror, the results of before and after measurements, and a possible damage model to explain the source of the observations. We should point out that an identical sister mirror to the X19A mirror which was used at beam line X7B shows nearly the same damage morphology.

2. X19A ZERODUR DAMAGE OBSERVATIONS

The mirror in the X19A beam line consists of a 700 mm long by 100 mm wide by 50 mm thick rectangular piece of ZERODUR, polished into a spherical shape with an extremely long radius of curvature, $R = 5 \text{ km}$. The reflecting surface is

Table I - Summary of mirror damage observations on NSLS mirrors. The "before exposure" and "after exposure" roughness and figure measurement values are listed where available. Measured radius of curvature is used as the figure parameter. Large changes in radius indicate significant surface stresses.

Beam line	Material/coating	Exposure	Roughness range (RMS) [Date - Before] [Date - After]	Figure: (Radius of curv) [Before] [After]	Physical Damage
X3	Fused silica cylinder Au coated	White beam = 4 yrs	4/88 - 33-80Å 4/92 - 64-84Å	N/A $R = 2.2\text{km}$	Burn marks, coating intact
X16A	Fused silica cylinder Pt coated	White beam = 7 yrs	1/86 - 18-40Å 5/93 - 9-1400Å	N/A LTP $\Rightarrow 7 \text{ km}$	Cracks into surface
U10A	Fused silica flat Au coated	Monochromatic beam	Before N/A 10/91 - 3-70Å (coated) 4-20Å (stripped)		Coating crazed 'upstream end', dark bands
U10A	Fused silica cylinder Au coated	White beam = 6 yrs	Before N/A 10/91 - 16-46Å	N/A $R = -5\text{km}$	Black marks on surface, discoloration on back
X19A	Zerodur sphere Rhodium coated	White beam = 3 yrs & 6 mos.	8/93 - 4Å 5/96 - 20-60Å	$R = 5 \text{ km}$ $R = 1\text{km}$	Material color change, deep subsurface cracks, 'figure' change, wrinkles in surface
X19A	Glass (BK7?) flat Al coated	White beam = 6 mos	1/97 - 8Å 6/97 - 9-1000Å	$R = 30\text{km}$ $R = 38\text{km}$	Coating crazed down central stripe, brown color centers
X19A	Pyrex uncoated flat	Direct undulator light = 3 mos.	4/96 - 4Å 10/96 - 54-300Å		Brown burn marks (raised above surrounding area?)
X7B	Zerodur sphere Rhodium coated	White beam = 3 yrs	SAME AS X19A	SAME AS X19A	SAME AS X19A
X13	Float glass uncoated	EP Wiggler direct beam	8/94 - 4Å 10/96 - 13-70Å		Brown burn marks
X15B	ENP/Al cylinder	Monochromatic beam	2/87 - 14-36Å 5/94 - 28-96Å	N/A $R = -10 \text{ km}$	No visible damage but finish degradation

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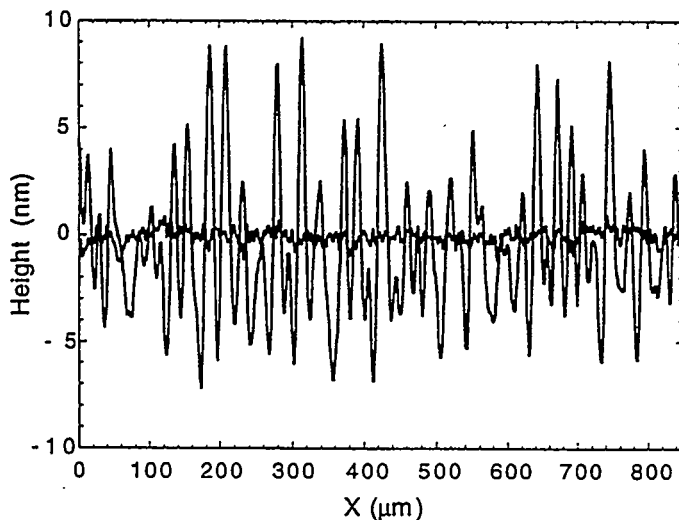


Fig 1 - Surface roughness profiles from the damaged (large amplitude) and undamaged (small amplitude) regions of the X19A ZERODUR mirror. The periodic structure in the damaged region has a period of about 25 μm .

this sample of the exposed region is on the order of 3.5 nm RMS, while the roughness in the unexposed region remains at the level of the "before exposure" value of about 0.4 nm RMS. The roughness in the damaged region appears as regular corrugations with a period of about 25 μm . This is more easily seen in a 3-dimensional contour plot where the periodicity of the corrugations is distinctly along the length of the surface. The beam from a HeNe laser reflected off of the center of the damaged region produced one well-defined diffraction spot off to each side of the specular reflection, exactly at the angle predicted by the measure periodicity.

Following the roughness measurement, surface figure measurements were made with the LTP in the damaged and undamaged areas. Before and after profiles of the mirror surface are shown in Fig. 2. The original surface had a radius of curvature of $R = 4.960 \text{ km}$. After exposure to the SR beam for about 3 years, the radius changed to $R = 1.067 \text{ km}$. This is a change in the total sag of the surface of about 40 μm , from 10 μm to 50 μm . We noticed that the rear surface of the mirror no longer made uniform contact with the granite surface plate of the LTP - the entire mirror substrate was bent like a stressed beam and rested upon a small area on the bottom. We had to prop it up to keep it from rocking during the measurement.

Suspecting that the surface region exposed to the beam may have contracted, as has been seen in the past by others^{3, 6}, we made several transverse scans with the LTP across the width of the surface at various positions along the length. Two such scans are shown in Fig 3, made at different locations on the surface. In the region of maximum damage the surface is indeed slumped by about 3 μm relative to the undamaged edges of the mirror. A trench of varying depth extends down the length of the mirror surface exactly in the region of SR beam exposure. Color center formation is seen in the substrate directly under this region, extending far down into the substrate.

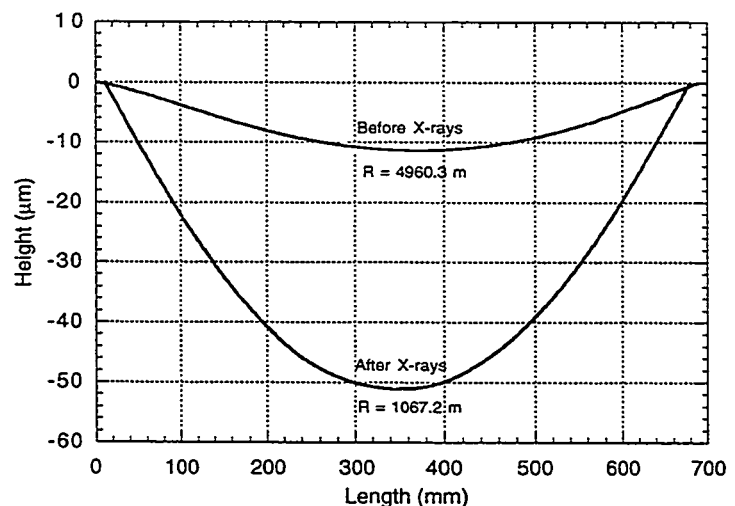


Fig 2 - Figure profile measurements made on the X19A mirror before and after 3 years of x-ray beam exposure.

coated with a 200 $\text{\AA}$ thick coating of Rhodium, and the mirror is used as a collimator with the SR beam incident at a 3 mrad grazing incidence angle. The critical energy for this material and angle of incidence is about 20 keV, which is well beyond the critical energy of the NSLS x-ray bending magnets of 5.5 keV. The mirror was held in a three-point mount and was exposed to the white beam with no active or passive cooling provided. Finite element thermal analysis indicates that the temperature rise of the substrate is only a few degrees Celsius during a typical operation cycle.

Immediately after removing the mirror from the X19A beam line, it was noted that the region down the center of the surface which was exposed directly to the white beam appeared hazy to the eye. This is indicative of a rough surface. Measurements were immediately made of the roughness at various points on the surface with the Micromap system. Figure 1 shows the results from the exposed region and from the unexposed region. The roughness in

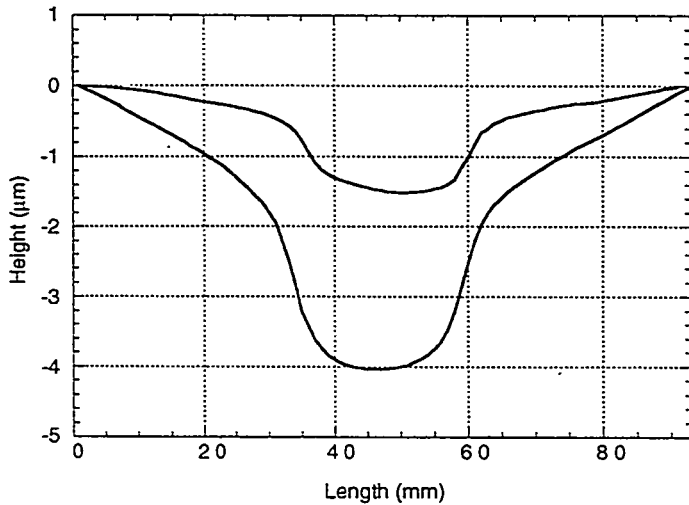


Fig 3 - Transverse LTP scans made across the width of the damaged surface at two locations on the surface, showing the channel produced by the x-ray beam. The original spherical surface sag was 0.25 μm in this direction.

exposed for about 3 years, with the region of lesser damage off to the side that was exposed for 6 months. The maximum depth in the central region is about 2 μm below the top rim of the trench, while the maximum depth in the offset region is about 1 μm below its rim.

The SR beam illumination was not uniform down the center of the mirror. A part of the mirror nearer to the upstream end of the beam line received more flux and consequently more damage. We have positioned the mirror so that this end is always located to the left in all the measurements. We use the terms "left end" and "right end" of the mirror to indicate the more damaged and less damaged regions, respectively.

3. CATASTROPHIC FAILURE OBSERVATIONS

After the mirror was removed from the beam line the second time, it was immediately measured again in the metrology laboratory, as indicated above. Over a period of several days, we noticed a series of cracks developing in the surface near the left end where maximum roughness and slumping occurred. Measurements show that the edges of the cracks are raised about

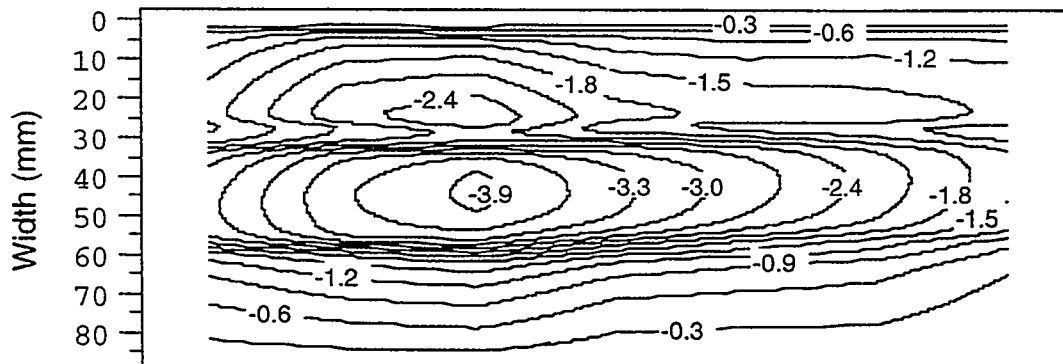


Fig 4 - Contour map of the damaged surface after the second exposure period. Depth contours are in μm . The locations of the two damaged regions are clearly visible as strips along the length direction. The total length of the mirror is 700 mm. Maximum exposure and damage is to the left of center.

Since a replacement mirror was not available at the time of these measurements, it was decided to reinstall the mirror in the beam line, but shift it over to one side to use an undamaged region of the surface. Although the radius of curvature was no longer correct for this beam line, the smoother surface area would allow for continued beam line operation at a reasonable flux level. The mirror was in place for an additional 6 months and then it was removed again for installation of an undamaged temporary mirror. Another series of tests were again performed on the mirror. Again, the region that was exposed off to the side showed the same signs of damage: microroughness corrugations and slumping. The roughness levels were less than the previous damaged area levels, which was to be expected, since the exposure time was much less. The depth of the slumping was also correspondingly smaller than before. A interpolated contour map of the final surface generated from the transverse scans is shown in Fig. 4. This clearly shows the region of maximum damage occurring in a trench down the center of the mirror that was

1 μm above the surrounding area in a cusp-like shape extending for several millimeters away from the crack. The fractures extend several millimeters down into the surface. After several days the growth of the fractures appeared to stabilize and stop. Fig. 5 is a top view of the end of the mirror back lighted in such a way as to make the cracks visible through the thin Rhodium coating. The two main fractures start at the edge of the mirror (top edge in the photo), proceed transversely across the secondary damage trench, turn abruptly parallel to the edge of the trench for about 1 cm and then turn again to run transversely across the maximum damage region. They stop immediately at the lower edge of the maximum damage region.

This catastrophic failure of the mirror substrate is indicative of severe internal surface and bulk stresses in the material. We set up a crude polariscope to look for stress-induced birefringence in the substrate. Viewing the right side of the mirror through the width of the substrate, we saw a series of bands approximately parallel to the top and bottom surfaces. The dark color center region immediately below the top surface obscured the observation of the bands in the upper half of the substrate, but the stress-induced birefringence was evident all the way down to the bottom surface. The orientation of the parallel bands was characteristic of those expected in a uniformly bent beam⁷. This confirmed the observation that the entire substrate was bent into a smaller radius of curvature by the surface damage.

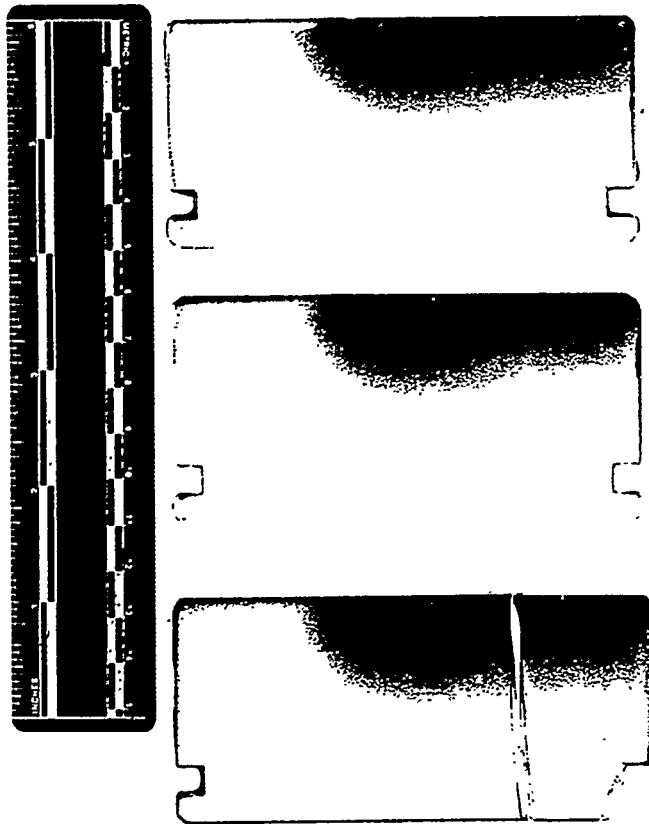


Fig 5 - Cross sections cut from the damaged mirror about 15 cm in from the left end. Final thickness after polishing is 9 mm. The two exposure regions are clearly visible, as is the extent of color center formation below the top surface. Fracture of the bottom piece occurred after the polishing.

The polariscope also revealed that the left end of the mirror was now nearly stress-free. No banding was observed, only a single change from light to dark in the vicinity of the cracks. The fractures had relieved most of the stress on the left side of the mirror.

Since the mirror was now damaged beyond repair, it was decided to cut several cross section slabs from the region of maximum damage to get a better idea of the extent of the damage below the surface. The slabs were cut and polished and are shown in Fig. 5. The slabs were cut from the region just to the right of the fracture zone, starting about 15 cm from the end of the mirror. Each slab is about 9 mm thick after polishing. One can clearly see the extent of the subsurface color center formation. The actual SR beam footprint is about 20 mm wide on the surface in each of the two exposure regions. One can see the qualitative difference between the 3 year exposure in the center and the 6 month exposure off to the side. What may not be evident in the reproduction of the original photos are the small cracks propagating a few millimeters down into the substrate from the top surface. The bottom slab shows a large crack propagating about 6 mm down to the bottom edge of the darkest color center region. The fracture of the bottom slab occurred after the polishing operation and may be a result of the initial stress concentration in the material.

4. DAMAGE MODEL

NSLS x-ray ring bending magnets provide a continuous range of photon energies with equal amounts of power both above and below the critical energy of 5.5 keV. There is still a significant flux above the rhodium absorption edge at 20 keV. Using the thin film and absorption cross section information available at Eric Gullikson's CXRO web site, based on the tables by Henke, et al.⁸ and the SR spectral distribution curves computed by Green⁹, we estimate that about 10% of the SR beam power lies above the 20 keV absorption edge. A significant flux can penetrate deep into the Rh and even pass through into the ZERODUR substrate. Over a long period of time the damage effects accumulate and produce changes in the ZERODUR material.

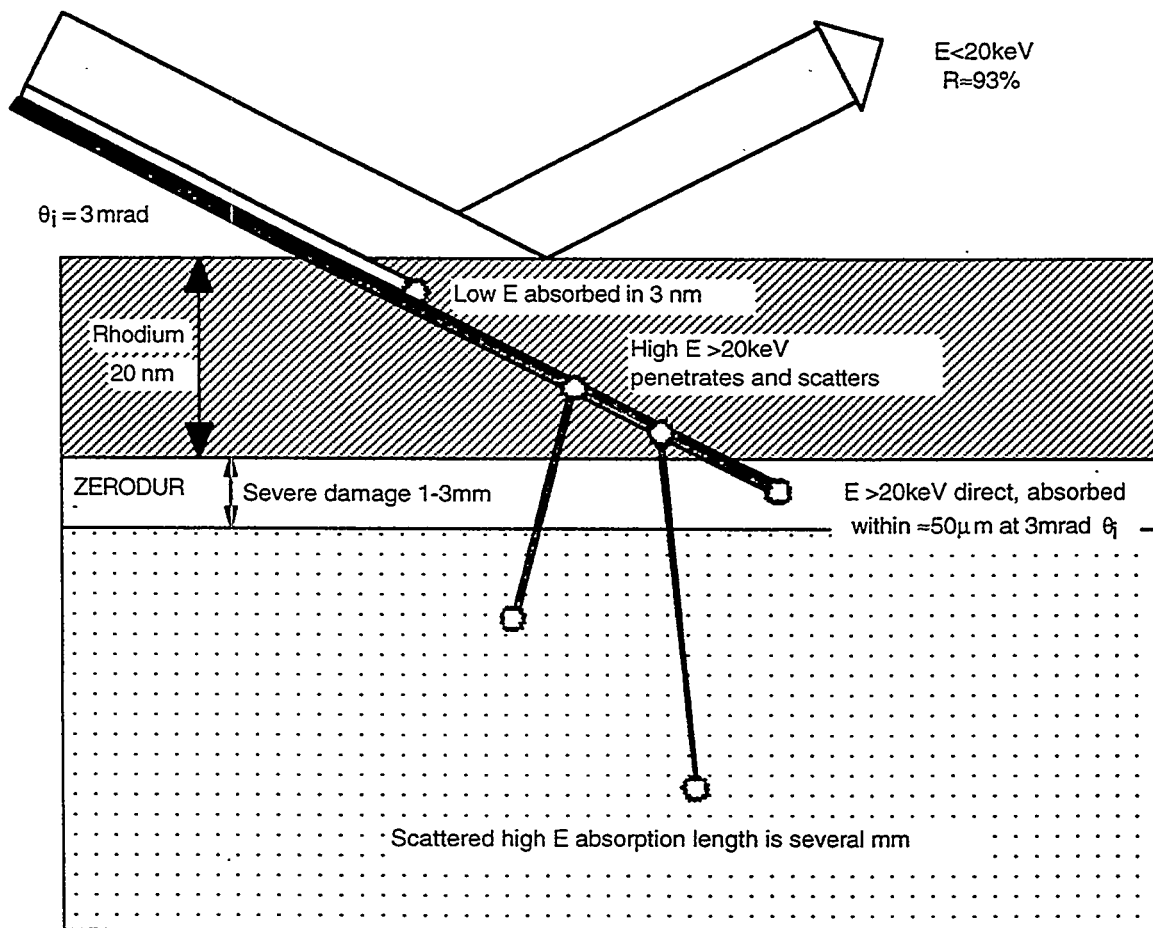


Fig 6 - Damage model (not to scale) for the X19A ZERODUR model exposed to NSLS bending magnet radiation. The mirror is coated with a 20 nm layer of rhodium with a critical reflection energy of 20 keV at the 3 mrad grazing incidence angle. Most of the 10% of the incident flux above the critical energy is absorbed in the 50 μm layer under the coating. Severe damage is observed in the top 3 mm of the surface.

A sketch of the proposed damage model is presented in Fig. 6. The white beam from the bending magnet is incident on the mirror surface at a grazing angle of 3 mrad. The nominal operating parameters for the x-ray ring provide a total power of 23 watts/mrad (horizontal) at 250 ma of operating current. The mirror accepts about 3 horizontal mrad of flux, so the nominal total power incident on the surface is about 69 watts. The reflectivity for the low energy photons is about 93%, so most of the radiation below 20 keV is reflected. The low energy photons that are not reflected are entirely absorbed in the Rh coating. At 3 mrad incidence angle, the typical absorption depth for these energies is about 3 nm, while the coating thickness is 20 nm. The energy deposited by the absorbed low energy photons is dispersed mainly by non-radiative processes and quickly

turns to heat. The actual heat load is very small, since the absorbed power is spread over a large surface area in the beam footprint.

The story for the 10% of the power contained in the high energy photons is quite different. The absorption length in Rh for photon energies above 20 keV is greater than 20 nm, so a significant fraction of the high energy photons are both absorbed in the Rh and penetrate into the substrate. The photons that penetrate into the ZERODUR at a 3 mrad angle then can travel several millimeters before being absorbed by the light elements that comprise the substrate material. This linear distance corresponds to a vertical depth of between 20 and 50 μm below the surface. In other words, the unscattered photons that penetrate through the Rh are absorbed in a 50 μm layer in the ZERODUR immediately below the surface. The photons that are absorbed in the Rh layer have a high probability of producing another photon by fluorescence decay. These photons are emitted into all directions, with a significant probability that it will travel down into the substrate at near normal incidence. If it does, it will travel several millimeters before being absorbed. This explains the observed color center formation deep into the substrate, well below the surface layer.

5. ABSORBED DOSE

In order to compare these observations of radiation damage to other studies, it is necessary to estimate the total absorbed dose in the damaged region. For the sake of simplicity, we calculate the dose for the incident flux in a unit horizontal milliradian acceptance angle. For the present X19A case the calculated numbers shown should be scaled by a factor of 3.

If we assume that 10% of the original 23 watts of power makes its way down into the substrate and that all of the photons are at an energy of 20 keV, the total absorbed power is

$$P_{\text{abs}} = 2.3\text{W} \div 1.6 \cdot 10^{-19} \text{J/eV} = 1.44 \cdot 10^{19} \text{eV/sec.} \quad (1)$$

The dose rate is the power absorbed per unit mass. The density of ZERODUR is 2.53 g/cm^3 and the volume over which the power is absorbed is given by

$$V = 20\text{mm} \times 600\text{mm} \times 50\mu\text{m} = 600\text{mm}^3. \quad (2)$$

The dose rate is thus $9.45 \cdot 10^{21} \text{ev/kg} \cdot \text{sec}$. The conversion to Rads is $1 \text{Rad} = 6.24 \cdot 10^{10} \text{MeV/kg}$, so the dose rate becomes

$$\text{Dose Rate} = 0.151 \text{MRad/sec} = 1513 \text{Gy/sec.} \quad (3)$$

This is an extremely high dose rate for conventional radiation damage studies using Co^{60} or high energy electron sources. Estimating the total absorbed dose for a typical NSLS operating schedule, the total absorbed dose would be 543 MRad in one hour, $6.52 \cdot 10^3 \text{MRad}$ over a 12 hour day, $0.130 \cdot 10^3 \text{MRad}$ per 20 day month, and $1.56 \cdot 10^6 \text{MRad}$ per year. In a recent study on radiation damage in ZERODUR, Pannhorst⁶ saw significant color center darkening after an exposure of $1.3 \cdot 10^6 \text{Gy}$. At our dose rate, this exposure would be achieved in only 1000 seconds! The SR beam provides about 6 orders of magnitude greater exposure than is found in typical radiation damage experiments.

6. SURFACE STRESS ESTIMATE

One of the first observations of damage in this ZERODUR mirror was the bending of the entire substrate. From our model, we infer that most of the damage occurs in a thin layer at the top surface of the substrate. With this assumption, we can calculate the amount of stress in the surface layer using beam-bending theory and Stoney's formula^{10, 11}.

In the simple case of beam-bending due to a uniform bending moment applied to the substrate by an external system, the magnitude of the maximum stress at either surface is given by the equation⁷

$$\sigma_x = \frac{Eh}{2R} \quad (4)$$

where E is Young's modulus ($90 \cdot 10^9 \text{Pa}$ for ZERODUR), h is the total thickness, and R is the radius of curvature that the originally flat beam is bent into. For a 50 mm thick bar bent into a 1 km radius, we get that the maximum surface stress is about 2.1 MPa or 300 psi. This is a significant stress, but it is far from the 50 to 100 MPa bending strength limit¹² to explain the spontaneous formation of large surface fractures in the mirror.

If we assume that most of the stress arises from the compaction of the thin damaged layer within 50 μm of the surface, we can use Stoney's equation to estimate the stress in the layer. For a thin layer of thickness t on top of a thick substrate of thickness b , where $b \gg t$, we have

$$\sigma_x = \frac{1}{6R} \frac{Eb^2}{(1-\nu)t} \quad (5)$$

where ν is Poisson's ratio for the material. If we assume that the damage layer is 0.5 mm thick, the layer stress is about 93 MPa or $13 \cdot 10^3$ psi. If we assume the damage layer is only 50 μm thick, this number goes up a factor of ten to 936 MPa or $136 \cdot 10^3$ psi. Either one of these stress levels is sufficient to exceed the bending strength limit of the material and can account for the spontaneous generation of cracks in the material.

The fact that the large cracks were observed to form in the surface only after the second removal from the beam line is probably related to static fatigue effects accelerated by atmospheric water vapor absorbed onto the surface^{12, 13}. Water vapor accelerates the breaking of bonds in the silica material and, as long as the stressed material is under ultra high vacuum in the beam line, static fatigue is minimized. If the material is brought out of the vacuum and exposed to air, the threshold for static fatigue is more likely to be reached in a finite time.

7. DISCUSSION

ZERODUR belongs to a class of materials called glass-ceramics¹⁴. It is not a true glass, but consists of a combination of a vitreous phase and a quartz crystalline phase. The quartz crystals have a negative coefficient of thermal expansion and the vitreous phase has a positive coefficient. By tailoring the proportions of crystalline to vitreous phases, a material with an extremely low thermal expansion can be formed. ZERODUR contains about 75% crystalline material and 25% vitreous. The observed surface damage after exposure to the SR beam indicates that the surface layer has undergone some modification, resulting in a volume contraction, or compaction. This phenomenon has been seen in other radiation damage studies on ZERODUR, but the nature of the process is not understood⁶.

Most of the radiation damage studies on glass and fused silica have been done with neutron and electron sources. Very little is known about the effects of long-term exposure to softer x-rays. Pure crystal quartz becomes more disordered under exposure to radiation from neutrons and electrons with a subsequent decrease in density. Pure fused silica glass, however, becomes slightly more ordered with a slight increase in density¹. Since ZERODUR is predominantly crystalline, one would expect that the damaged surface layer would expand after irradiation. We observe just the opposite effect: the thin damaged region contracts with a large internal tensile stress which puts the upper half of the substrate into a state of compressive stress. One possible explanation for this effect is that in the thin damaged region, the crystalline phase has grown at the expense of the vitreous phase. We plan to perform further analysis on the material to confirm this conjecture. Preliminary results from atomic force microscope measurements of the surface morphology in the damaged and undamaged regions seems to indicate that the microcrystals in the damaged region are significantly different and probably larger than in the undamaged region.

8. CONCLUSIONS

It is obvious that ZERODUR is not a good mirror substrate choice for the conditions of operation at the X19A beam line. This is not to say that it is not a suitable material for all SR applications. To the contrary, it is quite suitable for applications where exposure to hard x-rays will be minimal. ZERODUR was chosen primarily for its low thermal expansion coefficient and its ready availability. The thinking was that even if the uncooled substrate were to heat up with a few degrees of thermal gradient, the surface figure would not be significantly affected. It is now clear that other factors are more important in influencing the performance of the material in this SR beam line. Despite the extreme grazing angle of incidence of the SR beam on the X19A mirror and its sister mirror in beam line X7B, a significant high energy flux penetrated below the coating and into the substrate. Over long time periods, this energy source was able to affect the chemistry of the material and change its structure. Since ZERODUR is a two-phase material, and glass is an inherently thermodynamically unstable material, we can expect changes to occur in the balance between the phases. The evidence suggests that single phase materials, such as single crystal silicon, CVD silicon carbide, and pure fused silica, do not suffer from the same catastrophic damage mechanism

as does ZERODUR. Single phase, or metal, materials are a better choice for use as mirror substrates when used as uncooled first mirrors in moderate power SR beam lines, such as those on bending magnets at the NSLS.

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