Recent Developments in Profile-Coated Elliptical KB Mirrors

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Abstract

Profile coating is a precision deposition technique developed to modify the surface figure of a sample. The technique uses a mask with a calculated aperture together with a substrate motion to produce the desired coated surface profile along the direction perpendicular to the substrate motion. The mask contour is determined by the desired profile and the thickness distribution at the substrate position without a mask. Profile coating has been successfully applied to produce elliptical Kirkpatrick-Baez (KB) mirrors using both cylindrical and flat super-polished Si substrates. Previously, focusing widths of 70 nm with 15-keV monochromatic beams, and 80 nm with white beams were achieved using an Au-profile-coated KB mirror with a flat Si substrate. Now precision elliptical KB mirrors with sub-nm figure errors can be routinely produced with both Au and Pt coatings on flat substrates. In this paper we present recent results and discuss the stability of profile-coated mirrors. Comparison studies of bare Si substrates and Au- and Pt-coated KB mirrors under extended synchrotron x-ray radiation and low-temperature vacuum annealing will be discussed in terms of film-stress relaxation and Si plastic deformation.

Keywords: x-ray optics, KB mirrors, profile coating, sputter deposition, film stress, Si plastic deformation

PACS: 41.50.+h; 42.82.Cr; 81.15.Cd; 68.60.Bs

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1. Introduction

It has long been recognized that mirror figure can be modified by controlled thin-film deposition [1]. Thin-film deposition with masking techniques has also been used to control coating thickness [2, 3]. Differential deposition through a narrow slit and controlled sputtering power was used to make elliptical KB mirrors from cylindrical substrates [4]. We have developed a profile-coating technique to achieve the same goal of making ultra-precise elliptical mirrors for x-ray microfocusing [5, 6]. With the help of advanced metrology measurements, precision elliptical KB mirrors with sub-nm figure errors can now be routinely produced using flat Si substrates with only two profile coatings [7]. Sub-100-nm focusing has been demonstrated in a synchrotron x-ray beamline using an Au-profile-coated KB mirror with a flat Si substrate [8].

Although there are many advantages to profile-coated mirror optics, one serious concern is in long-term figure stability. We have noticed significant changes in mirror figure during extended x-ray irradiation. The observed changes are believed to result from plastic deformation of profile-coated KB mirrors, even though the mirrors were

operated in a total reflection mode under critical angles. This plastic deformation is a surprise since in our applications there is very little heat load on each KB mirror and the crystalline Si substrates that we used is brittle at room temperature: the brittle-ductile transition is over 847 ˚C [9].

The observed plastic deformation may be related to film stress introduced during coatings. It is known that residual film stresses can be relaxed at low temperatures on the order of a few hundreds of degrees C [10-12]. We found that low-temperature vacuum annealing can cause plastic deformation similar to that introduced by extended x-ray radiation in the profile-coated KB mirror. To overcome this plastic deformation we need to optimize the mirror dimensions to improve mechanical stability. We found also that the Pt-profile-coated KB mirrors have smaller deformation than the Au one does.

2. Recent results of Au- and Pt- coated KB mirrors using flat Si substrates

Precision elliptical KB mirrors with sub-nm figure errors are now routinely produced with profile coatings on flat substrates. Shown in Fig. 1 are a height profile measured by microstitching interferometry, and the best-fit ellipse with parameters of S_1 = 98 m, S_2 = 74.88 mm, and θ = 3.63 mrad, as well as the residual height profile for a Au-profile-coated KB mirror on a 45-mm L × 20-mm W × 4.5-mm H super-polished Si substrate. S_1 is the source-to-mirror distance, S_2 the mirror-to-focus distance, and θ the mirror glancing angle. The small rms height error of 0.85 nm between the best-fit ellipse and the measured data predicts a figure-limited focal spot of 38.9 nm (FWHM) for the mirror. This predicted focusing is close to the diffraction limit of 37.8 nm for a perfect

ellipse, according to wave-optical simulations [13]. The mirror has not yet been tested on an x-ray beamline.

Another Au-coated KB mirror on a 20-mm L × 10-mm W × 8-mm H flat Si substrate with parameters of S_1 = 36 m, S_2 = 30 mm, and θ = 3mrad has been used on the 34-ID beamline at the Advanced Photon Source. This mirror has achieved focusing widths of 70 nm with 15-keV monochromatic beams and 80 nm with white beams. It was used in conjunction with a computer-aided, differentially polished mirror to make a KB pair in a nanofocusing setup [8]. Separately, in a microfocusing setup on the same beamline, both mirrors in the KB pair were Au-profile-coated on flat substrates, with dimensions of 70-mm L × 9-mm W × 4.5-mm H and 40-mm L × 10-mm W × 20-mm H. This KB pair has been successfully operated and produced ~0.60 × 0.40 μm^2 focused white beams for several years under normal conditions until it was recently subjected to extended x-ray radiation with the slits fully open continuously for several days. After this exposure the KB pair could no longer be used for microfocusing.

3. Extended synchrotron radiation and low-temperature annealing tests

One of the damaged mirrors, the 70-mm-long one, was re-measured using microstitching interferometry. The original coating of this mirror a uniform 15-nm-thick Cr layer and then a Au-profiled layer with thicknesses varying from 100 nm in the center to over 6 μm at the ends, as shown in the surface height plot in Fig. 2. The surface profiles before and after the extended x-ray radiation are shown in Fig. 2(a). There is a clear upward, concaved profile change, as can be seen from the height difference data. The maximum deflection at the center of the mirror is ~140 nm.

To understand the observed deformation, we have carried out low-temperature annealing tests on separated profile-coated KB mirrors and bare substrates with the same dimensions of 70-mm L × 9-mm W × 4.5-mm H. Shown in Fig. 2(b) is a comparison of the surface height profiles for a Au-profile-coated mirror before and after annealing. The sample was heated in vacuum using a quartz halogen lamp [14] at a rate of ~10˚C/min up to a maximum temperature of ~350 ˚C and then held at this maximum temperature for 15 min. We noticed that the annealing temperature could not be much higher than ~350 ˚C. Samples annealed at ~400 ˚C show small bumps on the edges where the coatings were thick. For samples annealed at ~350˚C the surfaces were still smooth, as seen under a scanning electron microscope.

For annealing, the sample was placed on an 8-inch × 10-inch Cu plate, which was sitting above the lamp on a substrate carrier using four small stainless steel screws as spacers. The heating temperature was monitored using a thermocouple attached to the holder of the lamp, where the temperature on the Cu block was calibrated using a pyrometer through a ZnSe window. The samples shown in Figs. 2(a) and 2(b) have the same coating profiles, and a similar upward profile change is evident after the annealing in both cases. The maximum deflection at the center is ~175 nm. Prior to annealing, this test KB mirror was stored at room temperature for over a year with no measurable change in the surface profile.

To further study the trend of the annealing effect, we have repeated the same annealing procedure for a Au-profile-coated, a Pt-profile-coated, and a bare Si substrate. All three substrates are from the same purchased batch, with the same dimensions of 70-mm L × 9-mm W × 4.5-mm H. All samples were annealed three times with their surface

profiles measured after each annealing and compared to that before annealing. The maximum deflection results were plotted as shown in Fig. 3. All samples had an upward concaved profile change after the first annealing, with the Au-coated one displaying the most change. After the second annealing, the changes are all smaller. For Au-coated and bare Si, the profile change turned to a downward convex, represented by a negative maximum deflection in the figure. The third annealing had very little effect in the surface profile for all three samples.

4. Discussions and summary

Precision elliptical KB mirrors can be produced using either cylindrical or flat substrates. When flat substrates are used the film stress becomes a significant problem since the coating thickness can be quite large in order to transform the original flat substrate to an elliptical profile. For mirror applications, an ultra-smooth, highly reflective coating is needed, which is often achieved using growth conditions with a low Ar pressure and a low substrate temperature. A large film stress is expected under these conditions [15] and it will bend the substrate. For a wafer substrate the curvature change due to film stress is proportional to t_s^2/t_f, where t_s and t_f are the substrate and the film thickness, respectively [16]. When the substrate is relatively thin and narrow, such as in our case with dimensions of 70-mm L × 9-mm W × 4.5-mm H, a change in film stress may introduce a measurable change in the surface profile from a previous balanced equilibrium state. For nanofocusing x-ray KB mirrors a drift of tens of nm from an ideal ellipse will severely aberrate the focus, rendering the mirror useless. The film stress relaxation [10-12] may introduce the detected surface profile change. Additionally, the

film stress may generate supersaturations of point defects and impurity atoms in the underlying Si crystal [17]. At very high stresses, dislocation movements in Si may also occur at lower activation energies than normal, leading to plastic deformation in the substrate [18].

The film stress relaxation at low temperatures is believed to be due to crystallization and diffusive relief of compressive stresses initially generated by atomic peening during deposition [12]. Elimination of voids and buried residual gas atoms also leads to a densification of the film. A densification in the film leads to an extra tensile stress in the film and an upward concaved change of the mirror surface profile, as seen in Figs. 2 and 3. We have found that thick Au films have a large thermal stress due to a substantially larger thermal expansion coefficient of Au compared to that of Si [20]. Pt has a relatively smaller thermal expansion coefficient than Au and thus a smaller thermal stress, which may attribute to the observed differences in a smaller stress relaxation (Fig. 3). The bare Si substrate also has a surface stress due to a compression of the surface layer during polishing [21, 22]. For chemical-mechanical polished substrates, the surface stress is relatively small and decreases rapidly with annealing. This smaller stress explains the smaller deflection for the bare Si substrate as shown in Fig. 3.

The observed plastic deformation is mostly due to bending of the whole mirror, as demonstrated by metrology measurements performed on the polished backside of a similar KB mirror. The deformation was not limited to the footprint of the x-ray beam and in general the mirror could not be reused by shifting sideways to a new location.

The similarity between low-temperature annealing and the extended x-ray radiation effects, as seen in Fig. 2, indicates that plastic deformation of the profile-coated

KB mirrors under extended x-ray irradiation may also be due to the film-stress relaxation in the coated layer. this comes as a surprise since these KB mirrors are not supposed to experience much heat load. The maximum x-ray power at 34-ID with slits fully opened to 200 × 200 μm^2 is ~114 mW, as measured by a thin thermocouple wrapped inside a 0.57 g Cu foil. The temperature of the Cu foil raised about 20 °C from room temperature in less than 1 min when the foil was directly hit by the white beam of x-rays. Considering the fact that for KB mirrors most x-rays are reflected, it is unlikely that the mirror temperature will increase much at all. For a full white beam incident at 3 mrad on an Au-coated KB mirror, the observed temperature rise was ~4 °C, as measured by a thin thermocouple spring-loaded on the surface of the Au layer. However, it is difficult to estimate the x-ray radiation effect on the surface of the Au film. It is known that the dominant absorption mechanism of the x-ray energy is photoionization in a thin surface layer [19]. This surface effect may be sufficient to cause a stress relaxation in the Au film without a noticeable increase in the mirror temperature. The surface photoionization under extended x-ray radiation may cause the same densification effect in the coated film as observed in low-temperature annealing. While it is difficult to pinpoint the exact mechanism of radiation damage to our KB mirrors, it makes sense to use thicker substrates and coating materials with a smaller film stress. Low-temperature annealing before the final corrective coating should be helpful too, even though the annealing temperature is limited by crystallization in the thick film. One may also use cylindrical substrates so that less coating is needed. However, x-ray-quality cylindrical substrates are more difficult to obtain and are expensive. Less expensive spherical substrates may be an alternative solution. But in this case, only a small region close to the center line

can be used, since only that region is measured by metrology and coated precisely according to the design parameters; away from the center line, the profile is affected by the spherical shape of the substrate.

In summary, we have achieved sub-nanometer figure-error accuracy for elliptical KB mirrors using profile-coating on flat Si substrates. These mirrors have been used to produce sub-100-nm focusing of hard x-rays. We have found that extended x-ray radiation may deform these mirrors if they are too thin and too narrow. This kind of deformation can be reproduced using low-temperature annealing cycles. It is likely that both extended x-ray radiation and low-temperature annealing may cause the total stress in the coated film to relax. This stress relaxation may introduce a plastic deformation of the mirror and affect the mirror performance. We observed that the magnitude of mirror deformation is proportional to the film stress. For applications where extended x-ray radiation is needed, one should use substrates with dimensions optimized to improve mechanical stability, pretreat the mirror with low-temperature-annealing prior to the final corrective coating, and select coating materials with smaller film stresses.

Acknowledgements

We thank R. Khachatryan, M. Wieczorek, and J. Attig from Argonne National Laboratory for technical assistance. This work is supported by the U. S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. DE-AC02-06CH11357 between UChicago Argonne, LLC and the Department of Energy.

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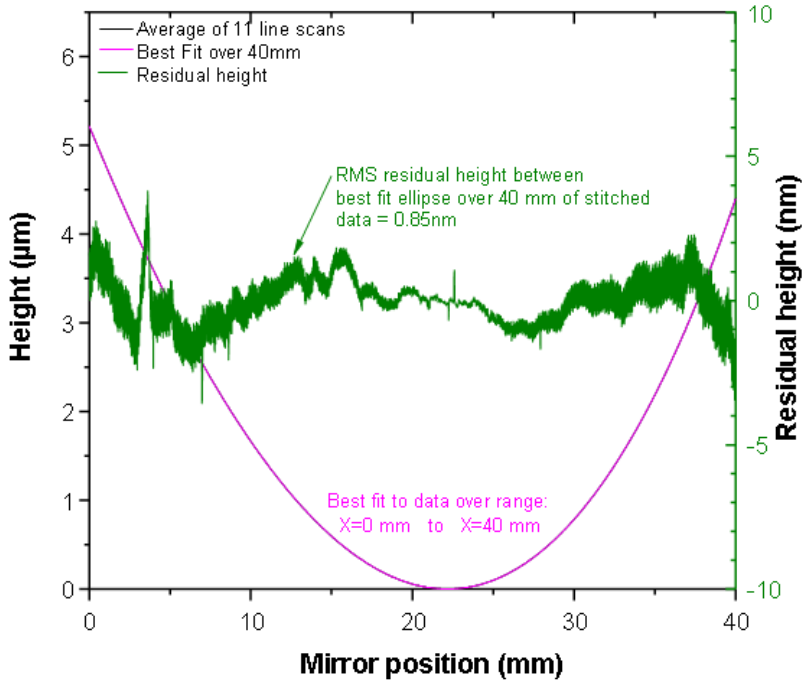
FIGURE CAPTIONS

Fig. 1. Surface height profile measured by microstitching interferometry, and the best-fit ellipse, as well as the residual height profile for an Au-profile-coated KB mirror. Note that with a sub-nm rms height error, the measured and the best-fit ellipse overlap with each other in the figure.

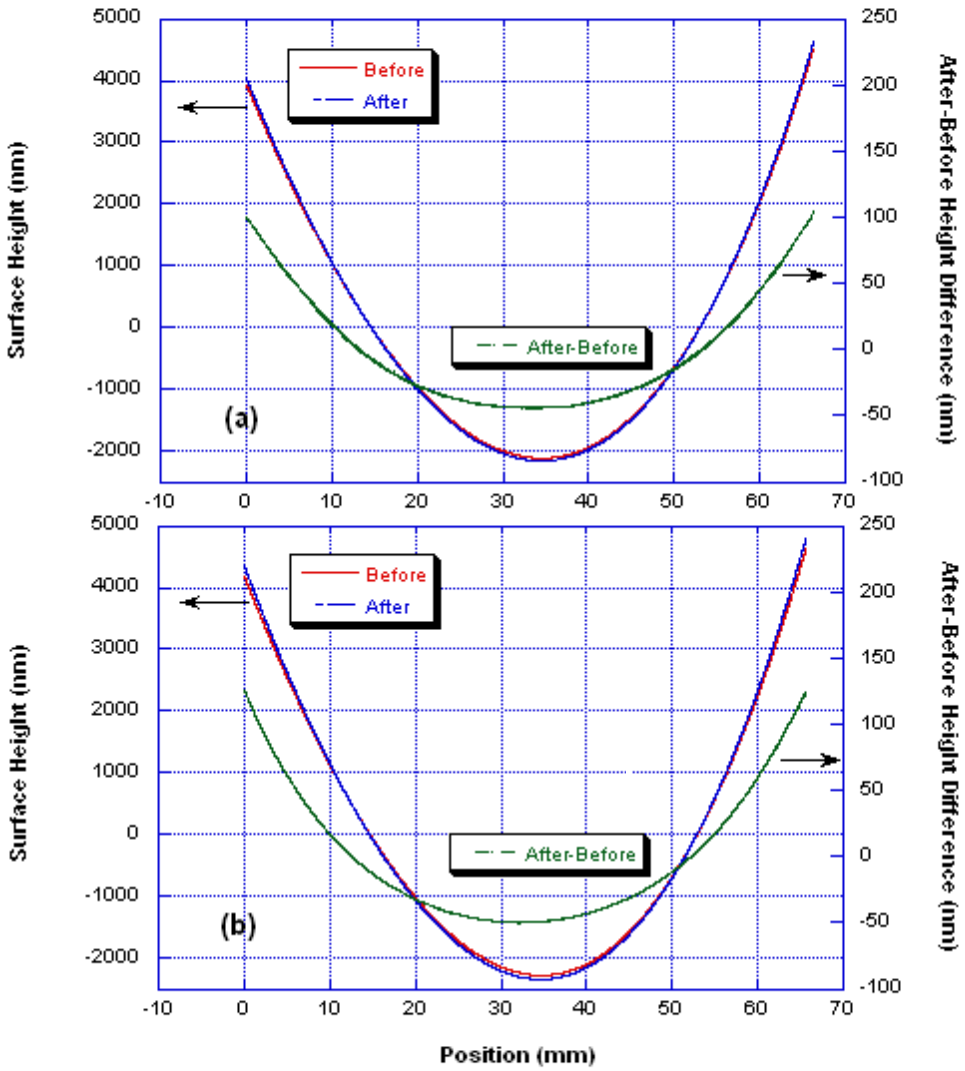
Fig. 2. Surface profiles before and after: (a) the extended x-ray radiation and (b) low-temperature annealing. A clear upward, concaved profile-change can be seen in both (a) and (b) from the height difference data. The maximum deflection at the center is ~140 nm in (a) and ~175 nm in (b).

Fig. 3. The maximum deflection at the center of the sample after each annealing for Au- and Pt-profile-coated KB mirrors, and a bare Si substrate. All three samples have the same physical dimensions. A positive number indicates an upward concaved profile-change and a negative one a downward convex change. Very little profile change was observed after the third annealing.

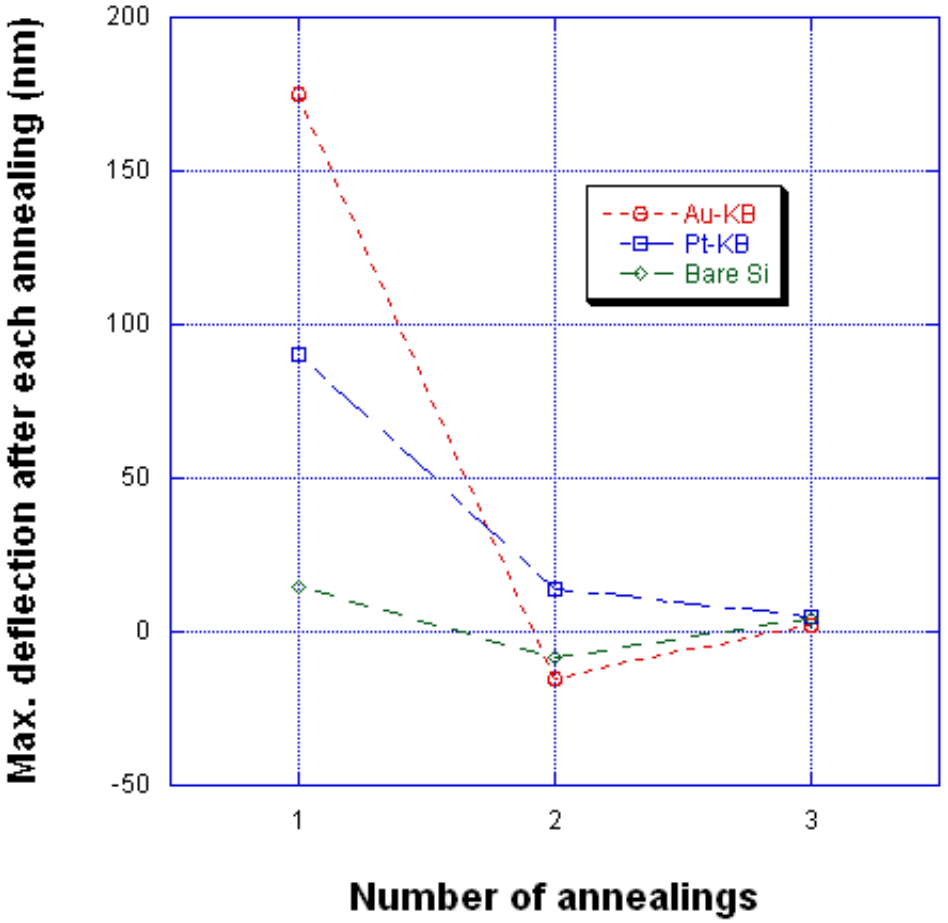
C. Liu Fig. 1


Average of 11 line scans
Best Fit over 40mm
Residual height
RMS residual height between best fit ellipse over 40 mm of stitched data = 0.85nm
Best fit to data over range: X=0 mm to X=40 mm
Height (µm)
Residual height (nm)
Mirror position (mm)
0
1
2
3
4
5
6
10
5
0
-5
-10
0
10
20
30
40

C. Liu Fig. 2


Before
After
After-Before
(a)
(b)
Surface Height (nm)
After-Before Height Difference (nm)
Position (mm)
5000
4000
3000
2000
1000
0
-1000
-2000
250
200
150
100
50
0
-50
-100
-10
0
10
20
30
40
50
60
70

C. Liu Fig. 3


Max. deflection after each annealing (nm)
200
150
100
50
0
-50
1
2
3
Number of annealings
Au-KB
Pt-KB
Bare Si