Crack Patterns in Thin Films and X-ray Optics
Thermal Deformations

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Crack Patterns in Thin Films and X-ray Optics Thermal Deformations

by

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A thesis submitted in partial fulfillment of the requirements for the degree of Master of Mechanical Engineering
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Thin films and multilayers are widely used in many applications, ranging from X-ray optics to microelectronic devices. In service, the X-ray optics elements are exposed to the X-ray beam, which heats up the structure resulting in the thermal deformations, and consequently in distortions of the reflective surface. In addition, the excessive heating may activate interdiffusion in the multilayers coatings and result in degradation of their reflective performance and even film cracking. Therefore, analysis of the thermally-induced deformations and stresses in the X-ray optical elements is important.

The presented work is organized in two major parts. The first part examines formation of the peculiar periodic crack patterns observed in the thermally loaded Mo/Si multilayers. Film stress evolution during thermal cycling of the multilayers on Si substrate is analyzed. Results of the high-speed microscopic observations of crack propagation in the annealed Mo/Si multilayers are presented. The observations provide experimental evidence of the mechanism underlying formation of the periodic crack patterns.

In the second part, thermal deformations and the resulting surface curvature changes in the X-ray optics elements are analyzed. Finite element modeling is used to assess the potential to thermally control curvature in the X-ray mirrors consisting of the Mo/Si multilayers on a Si substrate. Influence of heating due to the X-ray beam irradiation on thermal deformations in the X-ray mirror bonded to a thick substrate is analyzed in-depth. The detailed consideration includes analysis of the thermal and structural mechanics simulations. Based on simulations of different model configurations, influence of structural composition on thermal distortions of the optics elements is addressed. Results of this analysis can be used to mitigate distortions of the X-ray optics caused by the X-ray
beam and provide basis for further studies of thermally controlling surface curvature in the optical elements.
Chapter One
Introduction

1.1 Fracture and crack patterns in thin films

Thin films are widely used in various applications, such as microelectronic devices and micro-electro-mechanical systems (MEMS); they serve as protective and thermal barrier coatings. Thin film multilayers are used as reflective coatings in optical applications. Often film deposition processes and/or service conditions lead to high levels of stress, which may cause film fracture and result in failure of the device. The industry has been facing many reliability issues connected to fracture of thin films during several past decades. Difficulties in the analysis involve complicated nature of mechanisms underlying evolution of the film stresses and fracture processes, complex geometry and poorly known materials properties. Although there has been a great progress in research of thin films fracture, reflected by thousands of scientific publications, prediction of thin films reliability still remains a challenging task.

Typically, thin films are deposited on substrates at elevated temperatures by chemical vapor deposition, physical vapor deposition, electro-plating and other methods. When cooled, thermal expansion mismatch between the film and the substrate results in the mismatch strains, and consequently in the film stress. In addition, the deposition processes may result in high intrinsic stresses in the film. On top of that, thermal loading in service, such as thermal cycling, may result in significant redistribution of the stresses caused by plastic flow in metallic films, change of the microstructure, interdiffusion processes [DN88].

Failure in thin films may occur in different modes depending on the loading conditions, material properties and geometry. Under tensile stresses thin films typically fail through cracking and delamination. Compressive stresses may result in thin film buckling delamination followed by cracking
of the film. In addition, film failure may result in fracture of the substrate through crack penetration across the film/substrate interface or due to the crack kinking [HS92b], [Suo03].

Simultaneous occurrence of the different failure modes may result in their interaction and significantly complicate analysis of the problem. Further complications arise when a structure is subjected to thermal shocking or dynamic impulse loading conditions. Crack propagation under these circumstances is not well understood.

The delamination of a film from a substrate [EDH88] due to a uniform tensile stress is illustrated in Figure 1. According to the strain energy release rate (SERR) criteria, the fracture occurs when the SERR of the interface crack $G_i$ due to the loading equals the critical value $\Gamma_i$ for a given materials combination:

$$G_i = \Gamma_i(\psi)$$

This critical value is also called the interface fracture toughness and it additionally depends on the ratio between the normal and shear stresses ahead of the propagating crack [MS65], which is defined through the mode mixity $\psi$ [RSW90].

![Figure 1. Delamination of a film from a substrate](image)

A typical failure mode for a brittle thin film under tension is the fracture by film cracking, where the crack path forms a channel [EDH88]. Illustration of this case is provided in Figure 2. The crack front advances in the film perpendicular to the interface without penetration so that the substrate remains intact. Utilizing the SERR criterion, the film fracture occurs when the crack driving force
equals the fracture toughness of the film:

\[ G_f = \Gamma_f(\psi) \]  \hspace{1cm} (2)

The depicted problem is three-dimensional, however for the straight cracks exceeding a few times the film thickness, the steady-state solution of the problem can be obtained in the plane-strain formulation [Gec79].

![Figure 2. Steady-state channel cracking due to a tensile film stress](image)

Analysis of the problem shows that the relatively thick substrate constrains the crack propagation, as opposed to a case of a free-standing film under tensile loading. The crack driving force increases for increasingly compliant substrates, since a compliant substrate provides less constraint on the crack [Beu92]. The plastic yielding in the substrate also facilitates the channeling crack growth by providing less constraint on the crack [BK96]. A fraction of results done in the field may also be found in [HE89], [SH90], [YSE92], [HS92a], [HHL95].

Growth of multiple channeling cracks across the film provide an effective way of releasing energy in a tensile strained film on a substrate (Figure 3). In case of the straight channeling cracks, the formed array of cracks was found approximately periodic. The cracks spacing depends on the elastic mismatch between the film and the substrate, magnitude of the tensile stress in the film and its fracture toughness [Tho90], [F:91], [TOG92], [SSF00].
In practical applications, such as MEMS and microelectronic devices on thin wafers, the sub-
strate is of a comparable thickness with the film such that stress in the film is allowed to relax
due to the substrate deformation. Analysis of the problem showed that a thinner substrate reduces
the crack driving force [Vla03]. Finite element modeling was utilized to study the channeling
cracks in thin films and multilayers including effects of plasticity [AB02], creep of the under-
layer material [LHP03], thermal cycling loading [BE01], [BA03], influence of the substrate thick-
ness [HPHS03], periodic interconnect structures [AJB02], and buffer layers [TMV05].

Stress concentration at the root of a channeling crack may cause decohesion along the film-
substrate interface [HE89]. Depending on fracture toughness of the substrate and the interface as
well as on the elastic mismatch between the bonded materials, a film crack may penetrate into
the substrate [YSE92]. Film decohesion around the root of a channeling crack (Figure 4) relaxes
constraint of the substrate increasing the crack driving force. Case of a symmetric film delamination
accompanying a channeling film crack was studied in [MPH07]. The authors analyzed delamination
conditions as a function of elastic mismatch, film stress and the interface fracture toughness using a
plane-strain FEM model.

However, as seen from the illustration (Figure 4), the delamination around the crack root may
not necessarily be symmetric. In the asymmetric configuration the film stress distribution ahead of
a propagating crack will strongly depend on the delamination front geometry and on the relative
position of the crack. Consequently, the crack deviates from the direct path attracted by higher intensities in the stress field.

Deviation of a crack from its original path is illustrated in Figure 5. Combination of the external normal and shear components acting on the crack are represented by the mode-I and mode-II stress intensity factors $K_1$ and $K_2$, respectively. The nonzero shearing component forces the crack to kink. Note, that the kink is assumed to be infinitesimally small – much smaller than the crack. Stress- and energy-based fracture mechanics criteria for prediction of the crack kinking direction may be found in the following papers: [ES63], [Sih73], [TP81], [Wu78], [TP82], [CR80], [HH89], [HBE91], [YX92], [Kan94].

Prediction of the crack path under general loading conditions at the crack tip is a much more involved task than those formulated under the steady-state conditions. Since the crack path is not known a priori, iterative approach must be undertaken for solution of the problem. Numerical
methods were successfully applied for many problems, such as prediction of asymmetric growth of delamination between two bonded wafers [Tur04] and modeling of evolving crack patterns in thin films [LHP03], [SC03], to name a few. Fracture mechanics-based analysis of a single and multiple channel cracks in thin elastic films [XH00] was used to predict the crack path tendency. It was also shown that a mode-I crack growth ($K_{II} = 0$) along a spiral may exist in a biaxially stressed film in case there are curved flaws to start the cracking. However, to the best knowledge of the author, no published attempts have been made so far to model interaction of a propagating film crack with the advancing delamination front.

While patterns containing straight cracks are very common to films in residual tension, combination of different effects such as temperature gradients, externally applied stress, influence of boundaries and asymmetry may produce, under favorable conditions, some peculiar crack patterns. First notion of helix-like crack patterns in residual tension in Pyrex glass plates was published in [Arg59]. Oscillating and brunched cracks along a large temperature gradient in a glass plate rapidly immersed into water were reported in [YS97]. Based on the experimental observations, the authors concluded that the oscillation arises from the two competing mechanisms: deviation of the crack from the straight path to release more strain energy, and restoration force which drives the crack away from the lateral edges towards the plate center line with the largest tensile stresses. From the experiments on drying precipitates on the substrates [NLJR02] it was found that the advancing delamination front forces the running tunnel film crack to turn inwards. The authors were able to model the spiral cracking with a spring-block model [LN00]. Experimental observations of the simultaneously running outwards spiral, sinusoidal, saw-tooth and crescent-like crack patterns in drying sol-gel silicate thin films on glass and steel substrates were reported in [SW03]. Based on the experimental data, it was suggested that the observed curving crack paths were attributable to film delamination. Observations of spiral crack paths and other interesting patterns can be found in other papers [DHJSC94], [CC95], [Gar90].

Recently, interesting sinusoidal and spiral crack patterns were observed in the Mo/Si multilayers deposited on the Si substrate. The samples were subjected to the 3-point bending, annealed at high temperature of about 500 °C and slowly cooled in a vacuum chamber, followed
by their microscopic observations [MLL+04], [VMM]. Similar to observations of other authors (e.g., see [NLJR02], [SW03]), the through-thickness cracks were accompanied by debonding of the adjacent areas. Based on the experimental findings the authors suggested that a combination of biaxial film stress, temperature and the externally applied stress possibly with asymmetric film debonding causes these periodic crack patterns.

The work presented in Chapter Two is the continuation of the study of the cracking behaviour in the annealed Mo/Si film aiming at finding the exact root cause of the observed crack patterns. This was accomplished by analyzing the stress state in the film as a function of temperature (section 2.1) and further results of microscopic observations (section 2.2). In particular, cracks propagation and crack patterns evolution captured using the high-speed photography are presented and discussed. It is believed that the present experimental work would contribute to a better understanding of the crack patterns formation in thin films and inspire development of the theoretical and numerical models capable of accurately predicting the observed cracking phenomena.
1.2 Thermal deformations in X-ray optics

Multilayer X-ray optics are used in the extreme ultra-violet lithography (EUVL), X-ray diffractometers (XRD), X-ray reflectometers (XRR), synchrotron sources and other X-ray devices [DBH+00]. Nowadays, the most widespread optics for XRD/XRR systems are the so-called "Göbel mirrors" [HOH+05]. The Göbel mirrors convert X-ray beam coming from an X-ray source into an intense parallel or focused monochromatic beam. Figure 6 shows a principle scheme of the Goebel mirror configuration in a X-ray diffractometer.

![Figure 6. Application of Göbel mirror in X-ray diffraction](image)

The mirror consists of a stack of typically 50-200 alternating nanometer-thick layers made from two different materials with a continuously varying curvature and thickness (Figure 7). To achieve high beam reflecting performance it is necessary to deposit ultra-precise multilayer stack onto an ultra-precise prepolished substrate of parabolic form [Bra02]. Common material combinations used in reflective X-ray optics are Mo/Si, W/B$_4$C, W/Si, Ni/C and others. In case of multilayers consisting of alternating Mo/Si nanolayers, heating above 110 °C causes interfacial chemical reaction that significantly degrades the mirror performance [Boe01], [BMP+03].

In the synchrotron diffraction optics typically the Si bulk single crystal is used to select a particular X-ray wavelength when the incoming beam diffracts from the single crystal. In this type of configuration large amount of heat is generated in the crystal, which has to be properly cooled, typically with water or liquid nitrogen [BJD+05], otherwise it may melt in the high power synchrotron beam. Such a case is shown in Figure 8, where the burn trace is seen on the surface of a Si
monochromator from excessive heating by the synchrotron beam [Sta]. Besides that, reduction of the beam quality is caused by thermal distortions of the crystal lattice that leads to a broadening of the rocking curve and reduces the peak intensity [BFKM00], [Beg97], [Kho91].

Although, the reflectivity optics of the in-house X-ray systems are usually not exposed to high incoming beam energies, even low temperature changes may lead to deviations from their initial ultra-precise geometry. This results in loss of the beam conditioning quality, as mentioned above. Geometrical distortions due to temperature changes are associated with the thermal expansion mismatch of different materials. Since Si substrate and the multilayer stack shown in Figure 7 have different coefficients of thermal expansion, upon heating/cooling the structure inevitably deforms. If temperature change is small (on the order of 10–20 °C), the deformation is small too and does not cause substantial reduction of beam conditioning, since thickness of the Si substrate is typically 2000 times larger than thickness of the multilayer stack. However, the wafer with deposited layers is, in turn, bonded to a thicker substrate (Figure 9) to increase its structural stability and improve
handling when the mirror is mounted in a device. When heated or cooled, deformation of this structure may become considerable since thickness of the Si wafer and the substrate is comparable.

Geometrical distortions of the X-ray optics may also arise due to the temperature gradients existing in the structure. For the plate-like structures (thickness is much less than the other dimensions), influence of the through-thickness temperature gradient on the surface curvature would be much higher than influence of the in-plane temperature gradients. This behaviour may be utilized to control surface curvature of the optical elements by applying necessary heating/cooling conditions.

Another source of geometrical distortions that should be noted here arises from high residual stresses due to the nature of film deposition processes [DN88]. This issue is addressed in section 2.1.2 in a greater detail.

In view of the above, discussion in Chapter Three will be concentrated on the analysis of deformed state of the X-ray mirror caused by thermal loading. Potential of thermal curvature control in X-ray optics is addressed in section 3.1. This is accomplished by investigating influence of the through-thickness temperature gradient on curvature of the mirror surface using the finite element modeling. Problem of thermal distortions in X-ray optics exposed to the X-ray beam irradiation is discussed in section 3.2. Numerical analysis contains results of thermal and mechanical finite element simulations of different X-ray mirror configurations on a thick substrate. Impact of boundary conditions, material properties and geometry is assessed. Ways of reducing mirror distortions are proposed based on these studies.
2.1 Residual film stress

2.1.1 Curvature method

Curvature measurement is the traditional experimental technique for determining stress in thin films deposited on substrates. The method is based on the observation made by Stoney [Sto09] that stress in the film strains the substrate so as it bends (Figure 10).

\[ \sigma = k \frac{M_s h_f^2}{6 h_f}, \]  

(3)

Figure 10. Substrate curvature caused by stress in the film

The biaxial stress in the film can be calculated from the substrate curvature using the Stoney equation:
where \( k \) is the substrate curvature, \( h_s \) and \( h_f \) are the substrate and the film thicknesses. The biaxial modulus of the substrate \( M_s \) is given by

\[
M_s = \frac{E_s}{1 - \nu_s},
\]

(4)

where \( E_s \) and \( \nu_s \) are the elastic modulus and the Poisson’s ratio of the substrate material, respectively.

Note, that the Stoney formula in equation (3) does not contain material properties of the film, only those of the substrate. Effects of film thickness on substrate curvature in bimaterials was first analyzed in detail in [Tim25]. The analysis shows that the thin film approximation gives error of 15\% for the film/substrate thickness ratio of \( h_f/h_s =0.05 \) in case the elastic mismatch between the materials is neglected [FS04].

The nature of film stresses falls into two major categories: growth or intrinsic stresses and induced or extrinsic stresses. The growth stresses depend on the conditions of film deposition processes and are connected to various complex physical phenomena occurring in the film material as well as at the materials interfaces [DN88]. The extrinsic film stresses in semiconductor applications are typically caused by the temperature change between the film deposition processes and the in service conditions, since the film and the substrate materials have different coefficients of thermal expansion. Then the elastic thermal mismatch strain in a film with respect to the substrate is

\[
\varepsilon_{th} = (\alpha_s - \alpha_f)\Delta T,
\]

(5)

where \( \alpha_s \) and \( \alpha_f \) are the substrate and film coefficients of linear thermal expansion, \( \Delta T \) is the temperature change. The corresponding mismatch stress is

\[
\sigma_{th} = \varepsilon_{th} M_f,
\]

(6)
where $M_f$ is the biaxial modulus of the film material. Rearranging equations (5) and (6), the coefficient of thermal expansion of the film material can be estimated by

$$\alpha_f = \alpha_s - \frac{\sigma(T)}{M_f(T - T_{ref})},$$

where $\sigma(T)$ is the film stress at the temperature $T$ with respect to the reference stress-free temperature $T_{ref}$.

Similarly to the Stoney formula for stress, the elastic mismatch strain in a film $\varepsilon_m$ with respect to the substrate can also be determined from the curvature measurements:

$$\varepsilon_m = k \frac{M_s h_s^2}{6M_fh_f^3}.$$  

Note, that the physical origin of the mismatch strain is immaterial.

2.1.2 Residual stresses in the Mo/Si multilayers

This section presents results of the curvature stress measurements during thermal cycling of the Mo/Si multilayers on Si substrate. The Mo/Si multilayers were deposited on a (100) Si substrate using magnetron DC sputter deposition at the Fraunhofer Institute for Material and Beam Technology in Dresden, Germany [Boe01]. The sputtered Mo/Si film consisted of 60 alternating layers with the corresponding thicknesses of 2.7 and 4.2 nm each, producing a total thickness of 353.4 nm. The Si substrate was 525 $\mu$m thick with the diameter of 100 mm. It should be noted, that the presented stress data was calculated based on the thin film approximation and the small deformation assumption.

Stress evolution in the Mo/Si multilayers stack as a function of temperature is presented in Figure 11. Three heating cycles with the peak temperature of 500 $^\circ$C were applied to the system, as shown in the figure. In the initial state at room temperature the total stress in the Mo/Si multilayers (further referred as the film stress) is compressive and equals about -360 MPa. The first heating cycle contains different stages of the film stress evolution. Without considering the mechanisms underlying kinetics of the film stress evolution, one could allocate the following four stages:
• 23 < T < 100 °C: linear increase of the compressive film stress with temperature. It can be suggested that in this temperature interval the change in stress is entirely attributed to the thermal expansion mismatch between the Si substrate and the Mo/Si multilayers, as seen from the structure of equations (5) and (6). Since the coefficient of thermal expansion of the Si/Mo multilayers stack is higher than that of the Si (due to the Mo layers), increase in temperature results in the proportional increase of compressive stress in the Mo/Si multilayers.

• 100 < T < 375 °C: relaxation of the compressive film stress due to interdiffusion between the Mo and Si layers. Results of the X-ray diffractometry investigations carried out by Böttger [Boe01] show that the Mo/Si multilayers are thermally stable for temperatures below 100 °C. Heating above this temperature stimulates interdiffusion between the Mo and Si layers. According to [HDS89], the hexagonal MoSi$_2$-phase (h-MoSi$_2$) nucleates at the temperature as low as 275 °C. However, the crystallization does not take place until the temperature is raised to 375 °C.

• 375 < T < 475 °C: rapid increases of compressive stress in the Mo/Si multilayers during formation of the h-MoSi$_2$-phase.

• 475 < T and T = 500 °C for 20 minutes: stress increase in the Mo/Si multilayers changing from compressive to tensile behaviour after 10 minutes. In the crystallization process the more
dense h-MoSi$_2$-phase consumes Mo and Si material from the multilayers. This densification process results in volume contraction of the multilayers, and consequently in a rapid increase of the tensile film stress.

Initial stage of cooling from 500 °C reveals that the crystallization process continues until the temperature falls below 475 °C. This short portion can also be characterized by a rapid increase of tensile stress in the forming h-MoSi$_2$-phase due to the reasons discussed above. The subsequent cooling results in a linear increase of the film stress up to about 800 MPa caused by the elastic thermal mismatch strain between the Si substrate and the multilayers.

Heating during the second thermal cycle follows the curve path of the cooling stage of the first cycle up to the temperature of 475 °C, where the stress is seen to increase rapidly, which similar to the first cycle. Formation of the h-MoSi$_2$-phase and the thermal elastic mismatch strain are the two competing mechanisms influencing stress in the multilayers in opposite ways, as described above. When temperature equals 475 °C the two competing mechanisms equilibrate each other. The rate of the crystallization process depends on the amount of Mo and Si phases left in the multilayers, and consequently it decreases in time (assuming constant temperature conditions).

Since much of the Mo and Si materials were consumed in the crystallization process occurred in the first cycle, the increase of the film stress is now much smaller compared to that of the first one, although the annealing times were equal. The cooling portion of the curve is linear apart from the temperatures close to 500 °C where the crystallization process still plays a significant role in the film stress evolution. At room temperature the stress in the multilayers stack reaches the value of about 1000 MPa. The third cycle is very similar to the second one in terms of the stress vs. temperature behaviour. The small increase in the tensile film stress caused by formation of the h-MoSi$_2$-phase indicates that almost all Mo and Si elements were consumed in the crystallization process. The film stress reaches 1100 MPa when the structure is cooled to the room temperature.

Based on the conclusion that change of the film stress below 475 °C is governed by the thermal expansion mismatch, the coefficient of thermal expansion of the Mo/Si multilayers can be estimated.
Consider the following values of the film and substrate material properties [ME]:

\[
\alpha_s = 2.6 \times 10^{-6} \text{ K}^{-1}, \quad E_f = 250 \text{ GPa}, \quad \nu_f = 0.3, \quad M_f = 357 \text{ GPa}.
\]

Now, for the material constants given above and the stress values measured at the corresponding temperatures of 23 °C and 400 °C (see Figure 11):

\[
\sigma(23 ^\circ C) = 1100 \text{ MPa}, \quad \sigma(400 ^\circ C) = 640 \text{ MPa},
\]

the coefficient of thermal expansion of the Mo/Si multilayer after the first cycle is calculated from (7):

\[
\alpha_f = 5.2 \times 10^{-6} \text{ K}^{-1}.
\]

From the parallel run of the stress vs. temperature curves for the second and third thermal cycles below 475 °C it is reasonable to assume that the CTE of the Mo/Si film does not change significantly and may be taken, as calculated above.

2.2 Microscopy analysis of the crack patterns

2.2.1 High-speed camera setup and samples

The Mo/Si multilayers used in this study were manufactured by the magnetron DC sputter deposition at the Fraunhofer Institute for Material and Beam Technology in Dresden, Germany. The stack consisted of 40 Mo and Si alternating layers with 2.7 and 4.2 nm in thickness, respectively. The multilayers were deposited onto a 525 μm thick (100)-Si substrate with a diameter of 100 mm. The two tested wafers had designations PS221 and PS227, according to the manufacturer.

Annealing was performed using a high temperature oven capable of heating up to 1100 °C. Prior to annealing, the investigated samples were cut out from the wafer into pieces about 2×2 mm² large using a diamond scriber. The cutting procedure induced defects and flaws on the edges of the (100)-Si wafer. The cutout samples were placed onto a thick ceramic plate and annealed in the oven at 500 °C for 20 minutes. After annealing the samples batch was taken out of the hot oven and
exposed to the ambient environment with temperature of about 23 °C. The samples were allowed to cool down slowly under the natural air convection while resting on the ceramic plate 1–5 minutes. For crack growth observations the samples were placed onto a steel stage of the microscope covered with a 1 mm thick ceramic plate. The thin ceramic plate was used to prevent immediate sample cooling during optical observations. Temperature of the samples was not measured during testing.

Figure 12 shows the experimental setup used to investigate the cracking behaviour in the annealed Mo/Si multilayers. To record the crack propagation in time a high-speed digital camera was attached to a long-range optical microscope, as shown in the figure.

![Figure 12. High-speed camera experimental setup](image)

One set of high-speed photography results presented in this study was obtained using a digital consumer camera ”Philips SPC/1300NC” capable of recording 90 frames per second (fps) with the resolution of up to 320×240 pixels. In the course of experiments it became evident, that even for the slower propagating cracks the rate of 90 fps was insufficient to capture important stages of the crack growth. Based on this experience, the high-speed photography results at a later stage of the work were obtained using a high-end professional fast-motion camera “Photron FASTCAM-Ultima 1024”. Propagation of the film cracks using this equipment was recorded at a rate of 1000 fps with the resolution of 512×512 pixels.
The section presents results of optical observations of the crack propagation in the annealed Mo/Si multilayers using the high-speed photography. In most cases cooling of the tested samples led to formation and growth of cracks in the Mo/Si film. However, a few samples did not reveal any cracking even when cooled to the room temperature, although all the samples were cutout from the same PS221 wafer. Although not verified in depth, absence of a critical starter defect in the Mo/Si film may explain this rare behaviour. This argument can be supported by the fact that no cracks are usually observed in the Mo/Si wafers annealed at 500 °C. No cracks were observed in the Si substrate.

Optical observations showed that the cooling rate of the samples correlated with the speed of cracks propagation in the annealed Mo/Si film. Only the slow propagating cracks revealed formation of the peculiar crack patterns. Obviously, the highest cooling rates are achieved when a hot sample is placed onto the metal stage of the microscope under the room temperature conditions. The faster propagating cracks were observed immediately after a sample was brought in contact with the microscope stage. As the sample cools down the cooling rate decreases. Accordingly, the cracks were observed to propagate slower after the sample temperature decreased. At this point it is also important to note, that the limited field of view of the microscope prevented from simultaneously observing the whole surface of a sample.

In most cases the fast propagating cracks preceded the slow growing cracks. However, in some rare cases this succession was observed to be broken. An example of a typical pattern formed by the fast growing cracks is presented in Figure 13. In both images the crack pattern has a tree leaf-like geometry, which was formed by the cracks branching and looping. The darker portions of the images correspond to the film delaminated areas that lie out-of-plane with respect to the original horizontal plane of the sample. The microscope image shown in Figure 13(a) contains both the tree leaf-like cracks and the cracks having a square-sinusoidal geometry in the heavily delaminated area seen in the upper right portion. The right part of Figure 13(b) shows a completely delaminated part of the film which is displaced with respect to the presumably non-delaminated portion seen on the left side. The black area at the upper left corner of this image represents a piece of a broken
delaminated film which was displaced by a large distance as a result of its sudden delamination and cracking.

![Figure 13. Tree leaf-like branching channeling cracks](image)

(a)  
(b)

Figure 13. Tree leaf-like branching channeling cracks

The fast propagating cracks were observed to form a grass-like pattern geometry presented in Figure 14. This oblong pattern was formed as a result of growth and branching of the channeling cracks accompanied by their intersection and termination. In this case a radial structure of the crack pattern can be recognized. The figure also shows that the cracks from the grass-like pattern turn

![Figure 14. Burst channeling cracks transforming into the sinusoidal and crescent patterns](image)

Figure 14. Burst channeling cracks transforming into the sinusoidal and crescent patterns

into the sinusoidal- and crescent-like shapes. One may also observe, that the film is delaminated to a large part.

Another example of the fast channeling parallel cracks that turn simultaneously into the sinusoidal and crescent types of cracks is shown in Figure 15. The parallel straight cracks emanated
from a mother crack by branching. It is interesting to note, that the amplitude of the crescent cracks increases as they propagate. As in the figures above, the film delamination is clearly seen.

![Image of cracks](image)

**Figure 15.** Channeling cracks transforming into a sinusoidal followed by a crescent form

Experimental studies of cracking behaviour in the annealed Mo/Si film discovered a new type of a crack pattern that was not previously reported in the literature, to the best knowledge of the author. This crack pattern may be best described as a square-sinusoidal or the “Chinese Wall” pattern, which is presented in Figure 16. It can be observed, that the period and the amplitude of the pattern remained constant as the crack propagated. Figure 16(b) shows that after the path of the square-sinusoidal crack was disturbed by the branched daughter crack, the mother crack could restore its initial periodic square-sinusoidal form. One can also observe the small delaminated parts around the considered crack. In the lower part of Figure 16(b) one may also notice presence of a straight channeling crack terminating at the square-sinusoidal crack.

Results of optical observations presented so far do not give the answer why such peculiar periodical crack patterns form in the annealed Mo/Si film. Remarkably enough, discovery of the mechanism explaining the observed crack behaviour was made by chance during adjusting the microscope focus. It was noticed, that a sinusoidal-like crack was closely surrounded by an area of the film lying out of the focus plane of the initially adjusted microscope. This area can only be attributed to a delaminated area of the film.

Figures 17(a) and 17(b) present the sinusoidal-like film cracks accompanied by delamination. In both images the delamination can be recognized as a slightly raised area around the crack relative
Figure 16. Square-sinusoidal or the "Chinese Wall" type channeling cracks accompanied by delamination

Figure 17. Sinusoidal channeling cracks accompanied by delamination
to the rest of the sample surface. The delamination follows and completely encloses the channeling crack, which terminated by looping on itself. Since stress in the film is tensile (see section 2.1.2) such closed delamination cannot take place alone in the film, as opposed to the case of buckling delamination under compressive film stresses [HHE00], [MCL02]. In the present case, the channeling crack releases the film edges necessary for the delamination to take place.

![Figure 18. Saw tooth-type channeling crack following a straight path accompanied by delamination](image)

During crack propagation the direction, amplitude and period of the pattern may suddenly change, as also seen from the microscope images (Figure 17). However, the undisturbed crack propagation under the uniform conditions results in a constant geometry of the crack pattern, as presented in Figure 18 showing a saw tooth-like crack pattern. One can also observe that the delamination front repeats the major direction of the crack propagation and remains at a constant distance from the crack centerline.

A close interaction between a channeling crack and film delamination producing the periodic crack patterns can be evidenced from the microscope images in Figure 19. It is interesting to note, that direction of the crack propagation changes as soon as it reaches the delamination front, although the periodical pattern is pertained.

Spiral crack patterns were also found in the annealed Mo/Si film. Two examples representing such cracks are shown in Figures 20(a) and 20(b). Close to the imaginary center the channel cracks show some similarity with an Archimedes spiral, which is more distinct in the right image. After propagating approximately three spiral cycles the crack formed a crescent pattern followed by
Figure 19. Sinusoidal channeling cracks following a curved path accompanied by delamination

Figure 20. Spiral channel cracking in the annealed Mo/Si film accompanied by delamination
termination and looping on itself. As in the cases of sinusoidal-like cracks, the delaminated area is present around the spiral cracks. It should be noted, that the spiral cracks were rarely observed in the rapidly cooled annealed Mo/Si film. One may suggest, that these patterns form under much slower cooling rates which provide uniform thermal loading conditions [MLL⁺04], [VMM].

Figure 21. Burst channeling cracks transforming into a sinusoidal in the annealed Mo/Si film

Experimental observations showed that the periodic crack patterns in the annealed Mo/Si film can also be triggered by impulse dynamical loading. Figure 21 presents the crescent-type crack pattern formed as a result of the drop test. The present case reveals a much larger ratio of the amplitude to the period length of the crescent pattern compared with the purely thermally loaded samples, shown above. However, formation of the crack patterns caused by the impulse dynamic loading was not pursued in the current work.

2.2.3 High-speed photography results

So far the discussion was concentrated on the post mortem microscope images of the formed crack patterns. Based on these observations it was concluded that interaction of a channeling crack with the film delamination front leads to formation of the peculiar periodic patterns. To shed light on the process of the crack patterns formation results of high-speed photography are presented and discussed in the following section.
Figure 22. Propagation of a straight channeling crack accompanied by delamination at selected instants of time

Figure 22 presents selected frames captured during propagation of a straight channeling crack in the annealed Mo/Si film. The cracking event was recorded using the Philips digital camera at a rate of 90 fps. The emboss digital filter was applied to the originally recorded frames. It is seen that as the channel runs through the film it is accompanied by a symmetric film delamination at all stages of the crack growth. The image comparison shows that the delamination front continues to advance in time and is much slower than the film crack. The average speed of the channeling crack is approximately 0.4 mm/s, which is relatively slow when compared to the cracks forming the leaf- or grass-like patterns (see below). Unfortunately, magnification and resolution of the used optical equipment does not allow to unambiguously determine whether the delamination runs ahead of the
channeling crack or vice versa. However, it should be noted that the frames f-40 and f-55 indicate that the channeling crack runs behind the advancing delamination.

![Image of cracks](image)

Figure 23. Propagation of wavy channeling cracks at different instants of time. One of the channels turns into a Chinese wall-type pattern.

Figure 23 shows simultaneous propagation of two parallel wavy channeling cracks. It can be seen that both cracks make a 90° left turn followed by formation of the Chinese Wall pattern in case of the left crack. Propagation of the left wavy channel is disturbed (frame f-60) by a scratch on the sample surface left by the diamond scriber, which can be seen at the bottom of the images. Obviously, this deviates the channeling crack from its original direction. Although the accompanying delamination is hardly seen in the presented images, a close observation reveals its presence. The initially small delaminated areas around the channeling cracks continue to grow causing delamination of the gross film areas.

Formation of the periodical crack patterns from the bursting grass-like pattern cracks, discussed above, was captured using the fast-motion Photron camera. The samples were prepared from the PS227 wafer. Figure 24 presents different stages of the crack pattern formation recorded at a rate of 1000 fps. Comparing the two first frames (frame-1546 and frame-1545) the average crack propagation speed can be estimated at least 200 mm/s. The third frame (frame-1544) reveals that after 0.002 s growth of the cracks slows down substantially. The fast crack growth was always observed
Figure 24. Selected frames showing growth of the burst-like channeling cracks followed by formation of the periodic crack patterns
to be accompanied by large displacement of the dust particles initially resting on the film surface. This flying particles event suggests that the fast growth of the channeling cracks is also accompanied by the film delamination, which suddenly creates the out-of-plane displacement of the film. However, due to the short period of time when these cracks propagate and limited optics resolution, delamination during the fast crack growth was not directly observed. After 0.007 s (frame-1539) the delamination is clearly seen to spread around the almost motionless channel cracks. The following frames show that the slow crack propagation phase begins where formation of the periodic crack patterns is accompanied by film delamination.

Figure 25 presents frames showing formation of the periodic crack patterns selected every 0.005 s. At all stages the channeling cracks interact with the advancing delamination front, although it is impossible to determine whether the cracks run ahead or behind the delamination judging by these microscope images. It can be observed that the delamination growth is faster in the vicinity of the channeling cracks as the latter provide the necessary free film edges. However, it is seen that after delamination is emitted by a channeling crack the former also continues to grow, although at a slower rate.

The interaction between a channeling crack and the advancing delamination front is the key point in the formation of the observed periodic crack patterns. In the immediate vicinity behind the delamination front the stored strain energy due to the tensile film stress is not completely released. Because of constraints provided by the substrate, the strain energy release rate necessary to crack a film attached to a substrate is much higher than the strain energy release rate necessary to crack the unattached film. Based on the optical observations it may be suggested, that during the slow crack growth phase the strain energy stored in the film is lower than that necessary for a channeling crack to occur in the non-delaminated film. On the other hand, the still stored strain energy in the film behind the delamination front is enough for the film cracking to occur. This explanation is supported by the fact that the channeling crack always propagates in close vicinity of the delamination front.

Formation of the crescent-like cracks presented in Figure 26 illustrates propagation of the channeling cracks along the advancing delamination front. In the first image (frame-2911) the long channeling crack on the right has just grew behind the delamination front and stopped, although it
Figure 25. Selected frames showing formation of the sinusoidal and crescent-like cracks
could continue growing along the available delamination seen in the image. After 0.008 s (frame-2903) the crack changed its path into the direction of the self-emitted delamination. One may notice that a smaller channeling crack seen in the lower right part of the image grows along the delamination front too. In the third and fourth images (frame-2860 and frame-2795) both channeling cracks grow behind the delamination front simultaneously stimulating debonding of the film. The later stages of the crack pattern formation essentially repeat the described cracking behaviour.

It is known from the fracture mechanics, that a crack propagates in the direction so as to maximize the strain energy release rate. Applied to the considered case, it may be suggested, that a
channeling crack changes its propagation direction when it is more advantageous to turn in the
direction of the self-emitted delamination, in terms of the strain energy release rate maximization.

2.3 Conclusions for Chapter Two

The Mo/Si film must be annealed up to 500 °C in order to form the h-MoSi$_2$. As a result of this
phase transformation and the thermal mismatch between the film and the substrate, high tensile
film stress arises when the structure is cooled. At a certain temperature, the strain energy stored
in the system is released by film cracking and delamination. It was observed that the cooling rate
influences the speed at which the film cracks propagate. The decreasing cooling rate resulted in the
slower propagating cracks. The periodic crack patterns were observed to form by the quasi-statically
propagating cracks.

Microscopic observations using the high-speed camera revealed that the peculiar crack patterns
form as a result of the interaction between the propagating channeling cracks and the advancing
delamination front. Periodicity of the patterns can be explained by a higher speed of channeling
cracks relative to the delamination and by the periodical process when a channeling crack turns
into the direction of the self-emitted delamination occurring at certain favorable conditions. These
successive crack turns produce the observed periodic crack patterns. However, a quantitative de-
scription of the conditions, which force a channeling crack to turn would help to better understand
the observed behaviour.
Chapter Three
Thermal Deformation of X-ray Optics

3.1 Wafer curvature due to the through-thickness temperature gradient

X-ray mirrors typically have a parabolic reflective surface along the beam application line and zero curvature in the perpendicular direction (refer to section 1.2). The average curvature of the parabolic surface is about $1/10 \text{ m}^{-1}$ [Bra] and cannot be changed after production. For such fixed systems different measures are undertaken to mitigate any parasite mirror surface distortions (e.g., thermal deformations caused by heating), which reduce the reflected beam quality. Some aspects connected to this problem are addressed later in section 3.2. At this point, the opposite problem formulation presents a great interest: what are the conditions to achieve the necessary variable curvature of the mirror surface which could additionally be varying in time? This approach has long been utilized in adaptive optics such as image stabilization systems in consumer photo equipment or image correction due to atmospheric and other distortions in powerful astronomy telescopes.

In context of the X-ray mirrors one could imagine numerous ways to control curvature of the reflective surface. Probably, the most conventional one would be bending of the mirror. This can be achieved by directly applying mechanical loads to the structure, by utilization of the piezoelectric effect, etc. A more unconventional way, at least from the author’s point of view, is utilization of thermal loads. It is obvious, that mentioned above parasite thermal distortions could also be utilized to control shape of the X-ray optics and thus curvature of the reflective surface. Thermal loads can be applied, for instance, by attaching heating or cooling elements to the mirror. To identify the necessary thermal loading, effect of temperature distribution on thermal deformations in the mirror needs to be analyzed first.
This section intends to provide insight for thermally inducing the target curvature in a Si wafer with a deposited reflective multilayer. For the sake of simplicity, the discussion is narrowed to axisymmetric case of uniform wafer deformations. Given the actual geometry, materials and temperature constraints, results of this study help to identify the temperature loading necessary to produce the required surface curvature of the X-ray optical element.

3.1.1 Model description

Consider axisymmetric wafer geometry shown in Figure 27. For convenience, cylindrical coordinate system is defined at the centre of the wafer with directions \((\rho, \theta, z)\). The wafer is 20 mm in diameter and it is composed of a 525 \(\mu\)m thick Si substrate, as well as the front- and backside layers. The wafer front side represents X-ray reflective coating consisting of 40x(Mo/Si) alternating layers with a uniform thickness of 2.7/4.2 nm each, as shown on the blow out. The back side of the wafer is sputtered with a 3 \(\mu\)m thick tungsten film. The choice in favour of the tungsten film is based on its ability to be deposited either with compressive or tensile stresses [Wat08], enabling one to magnify or compensate wafer curvature which may be caused by the front side layers. The wafer is taken to be initially flat, as well as stress free.

![Figure 27. Geometry of X-ray mirror indicating structural composition with front (reflective) and back sides](image)

Material properties of the mirror constituents are listed in Table 1. The materials are assumed to be isotropic elastic. The Poisson’s ratio and the coefficient of thermal expansion for the Mo/Si multilayer were estimated using the composite mixture rules [AZP84] and are input in terms of the effective values.

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Table 1. Material properties of X-ray mirror components at 27°C

<table>
<thead>
<tr>
<th>Material</th>
<th>Young’s modulus, $E$, GPa</th>
<th>Poisson’s ratio, $\nu$</th>
<th>CTE, $\alpha$, $10^{-6}$ 1/K</th>
</tr>
</thead>
<tbody>
<tr>
<td>Silicon</td>
<td>149.0</td>
<td>0.28</td>
<td>2.6</td>
</tr>
<tr>
<td>Tungsten</td>
<td>400.0</td>
<td>0.28</td>
<td>4.4</td>
</tr>
<tr>
<td>40x(Mo/Si)</td>
<td>170.0$^2$</td>
<td>0.30</td>
<td>3.5</td>
</tr>
</tbody>
</table>

$^1$material data taken from [cI]
$^2$Measured using the indentation test [Vol]

Analysis of the mirror geometry and the thermal expansion mismatch between the materials suggests that under uniform heating conditions the midplane of the structure will take a concave form. This can readily be shown, since thickness of the tungsten layer is about one order of magnitude higher than that of the Mo/Si multilayer (tungsten has a higher CTE too). However, for the sputtered Mo/Si multilayer the upper limit of operation temperature is about 100 °C [Boe01], [BMP+03]. Exceeding this temperature leads to degradation of the multilayers in terms of its abilities to reflect the X-rays due to a reaction between the individual layers. Preliminary simulations showed that the target radius of curvature of 10 m cannot be achieved by a uniform heating of the X-ray mirror in the given temperature range.

Now, let us explore the nonuniform temperature loading of the structure. For simplicity, assume the temperature gradient is constant. In this case the temperature varies linearly with the wafer thickness and is uniform in other directions. Under these conditions, a corresponding uniform strain field is generated resulting in the curved deformation shape. As a result of symmetry and translational invariance the deformed shape of the midplane is spherical, provided the deformation is small. It is now to be answered, whether such thermal loading is able to produce the required radius of curvature, mentioned above, in the prescribed temperature range. The next section focuses on analysis of this problem using the finite element modeling.

3.1.2 Finite element model

Commercially available finite element package ANSYS [Ans] was used to analyze thermal deformations of the X-ray mirror. Taking advantage of the axisymmetric mirror geometry, a corresponding axisymmetric model was created. The model dimensions and the finite element mesh together with
the boundary conditions are shown in Figure 28. As indicated, the model includes the Mo/Si multilayer on the top and the tungsten film on the bottom surfaces, respectively. The model was meshed with the 8-noded two-dimensional PLANE183 elements with the axisymmetric key option turned on. The Mo/Si multilayer was modeled with one layer of elements in thickness direction with the effective material properties, as mentioned above. This simplification does not significantly influence the deformed shape but drastically reduces number of elements in the model. The tungsten film was also modeled via on layer of elements in the thickness direction. Material properties used in the model are listed in Table 1.

Figure 28. Finite element model of X-ray mirror illustrating the applied temperature loading

The model was allowed to deform freely by constraining the axis of symmetry with the symmetry boundary conditions. In addition, the bottom node on the symmetry axis was constrained to prevent translational movement in the vertical direction.

Figure 28 also shows application of heating and cooling loads to the bottom and top surfaces of the model. This designation should rather be treated conditionally in the sense that the temperature at the bottom surface ($T_{bot} = 42 \, ^\circC$) is higher than the temperature at the top surface ($T_{top} = 23 \, ^\circC$) to produce the varying temperature distribution in the thickness direction. In fact, the through-thickness temperature gradient $dT/dz$ was applied directly to the model, as shown at the left portion of the figure.

Convergence study (see Appendix B has shown that the used finite element mesh is adequate to accurately reproduce thermal deformations of the model.
3.1.3 Results

The expanded axisymmetric simulation plot (Figure 29) shows distribution of radial strains \( \varepsilon_{\rho \rho} \) in the X-ray mirror. It can be observed, that the uniform through-thickness temperature gradient produces the corresponding uniform strain field with a linear distribution across the thickness, as expected for small deformations. Higher temperature at the bottom surface results in larger elongation of the material below the neutral surface of bending which produces the concave spherical shape of the mirror. Note, that the free-edge singularities [Bog71] are not reproduced in the plot which was achieved by excluding elements at the corresponding locations.

![Figure 29. Total radial strains in the X-ray mirror due to the through-thickness temperature gradient](image)

Figure 30 illustrates the out-of-plane displacement field of the X-ray mirror. Due to the axisymmetry, the circumferential curvature \( k_{\theta \theta} \) and the interaction term (the twist) \( k_{\rho \theta} \) are zero. Consequently, the only curvature term associated with the radial direction is nonzero:

\[
k_{\rho \rho} = k.
\]

As seen from the displacements plot, the bow does not exceed 5 \( \mu \text{m} \) for the 20 mm large wafer with total thickness of about 528 \( \mu \text{m} \) under the 19.1 \( ^\circ \text{C} \) temperature gradient. This result can be utilized to calculate the wafer curvature presented below. Note that because the solution is linear elastic, the calculated result can be scaled with the value of the temperature gradient.
The out-of-plane displacement $u_z$ can be written in terms of the radial position $\rho$ and its curvature $k$, as follows:

$$u_z(\rho) = \frac{1}{2} k \rho^2.$$ \hfill (9)

From equation (9) one can determine the uniform curvature as:

$$k = \frac{2u_z(\rho)}{\rho^2} = 9.8 \times 10^{-5} \text{ mm}^{-1},$$ \hfill (10)

which is the sought curvature of $1/10$ m$^{-1}$, as stated above.

The result from equation (10) can readily be verified by directly calculating the second derivative with respect to the radial position, as known from the analytic geometry. Again, assuming the curvature is small, equation of the neutral line of bending [PAK+85] is

$$k_{\rho\rho} = \frac{d^2 u_z}{d\rho^2}.$$ \hfill (11)

To find slope and curvature of the wafer surface, the first and second numerical derivatives were applied to the displacement field $u_z(\rho)$. The results for a Si substrate and the X-ray mirror consisting

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Figure 30. Out-of-plane displacements of the X-ray mirror due to the through-thickness temperature gradient
of the Mo/Si and W layers are shown in figures 31(a) and 31(b). The slope varies linearly with the distance from the wafer centre. The two lines representing the slope for Si wafer and X-ray mirror (Figure 31(a)) almost coincide on the plot suggesting the deformed shape of both structures is essentially the same. Analogous behaviour can be observed on the curvatures plot (Figure 31(b)).

![Graphs of slope and curvature](image)

Figure 31. Slope and curvature of the wafer reflective surface as a function of the radial position

It is seen, that the curvature is independent on the position for the most part of the wafer and the result from equation (10) is reproduced. However, two remarks should be made. Splash-like behaviour on the right portion of the curve representing curvature of the X-ray mirror is attributed to singular behaviour of the strain field at intersection of the free edges with the interface between Mo/Si stack and Si. The Si substrate does not have material interfaces and the curvature plot in this case is flat. The abrupt fall off seen at the left- and the rightmost parts of the curves should be disregarded. The author assumes that this erroneous behaviour is caused by application of the central difference scheme for the edge points instead of the forward and backward ones. This issue needs to be clarified with the ANSYS development team.

Essentially the same result for the Si substrate and the X-ray mirror in terms of the slope and curvature values shows that the influence of the relatively thin layers is insignificant. In other words, for a given geometry and materials set, influence of the temperature gradient is much higher than the influence of the thermal mismatch between the materials. The elastic mismatch in this case can be neglected at all because its impact is even much smaller than thermal mismatch influence [Tim25].
3.2 Thermal deformations of X-ray mirror due to beam irradiation

3.2.1 Model description

Beam irradiation of G"obel mirrors and Si monochromators usually takes place along a narrow stripe of the reflective surface (see Figures 6 and 8). In addition, the mirror is composed of different materials that also results in a complicated model geometry. Along with the nonhomogeneous thermal boundary conditions and nonlinear convective and radiation heat transfer, it is virtually impossible to construct an adequate analytical solution able to accurately predict thermomechanical behaviour of the mirror. To circumvent these problems, the finite element modeling was utilized in the course of this work with the simulation strategy described below.

The commercially available finite element package ANSYS [Ans] was utilized for computation of thermal deformations in the X-ray mirror. The analysis was carried out in two sequential steps: thermal and structural mechanics simulations. In the thermal analysis natural cooling to air convection and heat transfer by radiation from the model surface were assumed as the only boundary conditions applied to the model. The assumption of natural air convection is valid since no special devices such as air fans, radiators or similar are usually utilized. The Stefan-Boltzmann law for heat flow by radiation was used in the thermal analysis. However, in view of a small temperature difference between the X-ray mirror with the ambient and the low absolute temperature range overall, the heat flow by radiation is small compared with that of convection. Bearing this in mind, no radiation reflecting surface interacting with the X-ray mirror were modeled to simplify the analysis.

The calculated temperature distribution from the thermal analysis has been used as input for the structural mechanics model which was assumed to deform freely. The sequential (uncoupled) thermal-mechanical finite element simulation was utilized as the influence of the structural mechanics variables on the thermal state of the mirror was assumed to be negligibly small.

3.2.2 Geometry and material properties

Model geometry of the X-ray optical element is presented in Figure 32. The 0.525 mm thick Si wafer is bonded without slipping to a 5 mm thick substrate. The top surface of the Si wafer contains
40x(Mo/Si) multilayer stack with the overall uniform thickness of about 240 nm and a 3 \( \mu \)m thick tungsten film on its backside (see the blow out in Figure 9). The in-plane dimensions of the mirror are \( 60 \times 20 \) mm\( \times \)mm. The X-ray beam exposed area has dimensions of \( 60 \times 1 \) mm\( \times \)mm and is located at the center of the reflective surface, as illustrated in the figure.

![Figure 32. Geometry of an X-ray optical element subjected to the heat flux from an X-ray source](image)

Composition of the X-ray optical element can be changed by varying material and/or thickness of the substrate, bonding of additional structures, etc. As discussed in section 3.1, the major factors influencing geometry distortions of the X-ray optical element under thermal loading are relative thickness of its constituents, thermal expansion mismatch and presence of the through-thickness temperature gradient. To analyze influence of these factors for the considered here X-ray optical element, four different model configurations were analyzed, as presented in Table 2.

### Table 2. Model configurations of the X-ray optics element

<table>
<thead>
<tr>
<th>Model variation</th>
<th>Geometry configuration</th>
<th>Heat flux, W/m(^2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Model 1</td>
<td>Si wafer(^1) on glass substrate</td>
<td>( 1 \times 10^4 )</td>
</tr>
<tr>
<td>Model 2</td>
<td>Si wafer(^1) on Si substrate</td>
<td>( 1 \times 10^4 )</td>
</tr>
<tr>
<td>Model 3</td>
<td>Si wafer(^1) on invar substrate</td>
<td>( 1 \times 10^4 )</td>
</tr>
<tr>
<td>Model 4</td>
<td>Si wafer(^1) on both sides of glass substrate</td>
<td>( 1 \times 10^4 )</td>
</tr>
<tr>
<td>Model 5</td>
<td>Si monochromator(^2)</td>
<td>( 1.33 \times 10^6 )</td>
</tr>
</tbody>
</table>

\(^1\)contains 40x(Mo/Si)=60(2.7/4.2) nm [Bra]

\(^2\)Deutsche Elektronen Synchrotron [Sta]
Model 1 consists of the X-ray mirror bonded to a glass substrate. Combination of the X-ray mirror bonded to a Si substrate is designated as Model 2. Influence of the thermal expansion mismatch in this model is very small due to the relatively thin Mo/Si and W films.

**Table 3. Mechanical properties of materials of the X-ray optical element at 27°C**

<table>
<thead>
<tr>
<th>Material</th>
<th>Young’s modulus, $E$, GPa</th>
<th>Poisson’s ratio, $\nu$</th>
<th>CTE, $\alpha$, $10^{-6}$ 1/K</th>
</tr>
</thead>
<tbody>
<tr>
<td>Silicon</td>
<td>149.0</td>
<td>0.28</td>
<td>2.6</td>
</tr>
<tr>
<td>Glass</td>
<td>70.0</td>
<td>0.17</td>
<td>0.5</td>
</tr>
<tr>
<td>Invar</td>
<td>148.0</td>
<td>0.30</td>
<td>1.3</td>
</tr>
<tr>
<td>Molybdenum</td>
<td>330.0</td>
<td>0.30</td>
<td>5.4</td>
</tr>
<tr>
<td>Mo/Si stack</td>
<td>170.0$^1$</td>
<td>0.30</td>
<td>3.5</td>
</tr>
</tbody>
</table>

$^1$Measured using the indentation test [Vol]

In Model 3 the X-ray mirror is bonded to invar substrate. As seen from the Table 3, the thermal mismatch between Si and invar is smaller than that between Si and glass. Owing to this fact, invar is the material of choice for substrates of the modern X-ray optics, such as Göbel mirrors [Bra]. This configuration serves as the reference.

Model 4 presents a structure composed of the glass substrate bonded between the X-ray mirror (contains the Mo/Si and W films) and a blanket Si wafer. Thickness of the latter is equal 0.525 mm. Neglecting the small relative thickness of the Mo/Si and W films, it can be said that the structure is essentially symmetrical with respect to its thickness direction.

All models 1–4 are subject to a heat flux from a low power X-ray source of $1 \times 10^4$ W/m$^2$. The Model 5 represents a bulk Si monochromator exposed to a high power X-ray synchrotron source of 800 W utilized at the Deutsche Elektronen Synchrotron (DESY) site [Sta]. It was assumed that only 10% of the beam power is lost due to the material heating.

Thermal properties of the materials used in simulations are presented in Table 4. Due to the small relative thickness of the Mo/Si and W films compared to the Si wafer and rather close thermal properties, these layers were not explicitly modeled in the thermal simulations and were included in the Si wafer. Material interfaces were modeled as ideal.

As seen from the table, the glass material has the lowest thermal conductivity at 27°C temperature, whereas Si thermal conductivity is about 100 times higher.
Table 4. Thermal properties of materials of the X-ray optical element at 27°C

<table>
<thead>
<tr>
<th>Material</th>
<th>Density, ρ, kg/m$^3$</th>
<th>Thermal conductivity, $k$, W/m-K</th>
<th>Specific heat, $c_p$, J/kg-K</th>
<th>Emissivity, $e$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Silicon</td>
<td>2330</td>
<td>130.0</td>
<td>714</td>
<td>0.6</td>
</tr>
<tr>
<td>Glass</td>
<td>2200</td>
<td>1.3</td>
<td>750</td>
<td>0.9</td>
</tr>
<tr>
<td>Invar</td>
<td>8050</td>
<td>10.2</td>
<td>515</td>
<td>1.0</td>
</tr>
</tbody>
</table>

material data taken from [cI]

3.2.3 Computation of the heat transfer coefficient

The nature of the convection heat flow heavily depends on the flow conditions near the surface, and it is nonlinear. In its simplest form, the convection heat flow can be accounted for by utilizing the heat transfer coefficient. Using the Newton’s law of cooling for convection heat flow, the heat transfer between a moving fluid and a surface can be determined [LL06]:

$$Q_c = h_c A_s (T_s - T_a),$$

(12)

where $h_c$ is the average convective heat transfer coefficient;

$A_s$ is the cross-sectional area for heat flow through the surface;

$T_s$ is the temperature of the surface;

$T_a$ is the temperature of the ambient.

The natural cooling conditions to air convection are assumed with the ambient temperature of 27°C.

The procedure for estimation of the average heat transfer coefficient $h_c$ follows the recommendations described in [Bla00]. Assuming the natural cooling conditions to air convection with the ambient temperature of 27°C, the constants and expressions necessary for computation of $h_c$ are presented in Table 5. It must be noted that the average heat transfer coefficient depends on the surface orientation. For simplicity, position of the X-ray mirror reflective surface was assumed horizontal located side up.

Figure 33 shows the plot of the average heat transfer coefficient $h_c$ as a function of temperature difference between the surface with natural convection to air and the ambient temperature. The curves represent the three orientations of the mirror surfaces with geometry dimensions shown in
Table 5. Constants and expressions used for calculation of the average heat transfer coefficient

<table>
<thead>
<tr>
<th>Configuration</th>
<th>Characteristic length, $L_{ch}$</th>
<th>Constant, $C'(27^\circ C)$</th>
<th>Heat transfer coefficient, $h_c, \text{W/m}^2\text{-K}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Vertical plate</td>
<td>$\frac{H}{2}$</td>
<td>1.51</td>
<td>$C'(\Delta T_{L_{ch}})^{0.25}$</td>
</tr>
<tr>
<td>Horizontal plate</td>
<td>$WL/[2(W + L)]$</td>
<td>1.38</td>
<td>$C'(\Delta T_{L_{ch}})^{0.25}$</td>
</tr>
<tr>
<td>(heated side up)</td>
<td></td>
<td>0.69</td>
<td>$C'(\Delta T_{L_{ch}})^{0.25}$</td>
</tr>
<tr>
<td>(heated side down)</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Figure 32. According to the calculations, the vertical wall and the horizontal side down have the highest and the lowest $h_c$, respectively. The saturation-like behaviour of the $h_c(\Delta T)$ curves suggests

Figure 33. Average heat transfer coefficient as a function of temperature for three different plate surface orientations

that active types of cooling with higher heat transfer coefficient (e.g., forced air flow, water cooling, etc.) need to be applied for a more effective heat removal, if necessary.

3.2.4 Boundary conditions

Heat generated in the optical element due to the X-ray beam irradiation is applied at the corresponding area (Figure 32) as a heat flux into the system, which causes increase in temperature. The heat removal is modeled by the surface convection to air, as discussed in section 3.2.3, and by the radiation heat flow based on the Stefan-Boltzmann law [LL06]. This law postulates that the radiation heat flow between a surface and its surroundings is governed by the highly nonlinear equation with
respect to the temperature:

\[ Q_r = \sigma A F_T \left( T_1^4 - T_2^4 \right), \]  

where \( \sigma = 5.67 \times 10^{-8} \text{ W/m}^2\text{-K}^4 \) is the Stefan-Boltzmann constant;

\( A \) is the effective area of the emitting surface;

\( F_T \) is the exchange radiation factor. In absence of other reflecting surfaces \( F_T = e; \)

\( T_1 \) is the absolute temperatures of the emitting surface;

\( T_2 \) is the absolute temperatures of the ambient.

Analysis of the equation 13 suggests that the heat transfer by radiation is comparably small for low absolute temperatures and low temperature differences between a radiating surface and its surroundings. This case applies to the X-ray reflective optics exposed to a low X-ray beam power density. Large contribution of the heat flow by radiation may be anticipated for the X-ray diffractive optics such as monochromators, since their temperature may rise up to 500 °C and more.

3.2.5 Finite element model

Figure 34 shows mesh of the finite element model used in simulations. Owing to the two planes of symmetry (xz and yz), a quarter-symmetric model was used, which resulted in reduction of the model size by a factor of four. For thermal simulations the model was meshed with the three-dimensional eight-noded solid elements SOLID70 [Ans]. The heat transfer coefficient \( h_c \) was evaluated at differential temperature between the surface and the ambient by setting the key option KEYOPT(2)=3. Overall number of SOLID70 elements in the finite element mesh is \( 60 \times 20 \times 10 = 1200 \). Three-dimensional thermal surface effect elements SURF152 were overlaid at the corresponding areas of the model for generating heat flux due to the X-ray beam exposure and radiation heat exchange with the ambient.

The structural mechanics simulation utilized the same model discretization as the thermal analysis. However, different types of elements were used. The Si wafer consisting of the Mo/Si multilayer and W film were modeled using three-dimensional 8-noded solid-like-shell elements SOLSH190.
Figure 34. Mesh of the quarter-symmetric finite element model of the X-ray optical element

This materials composition was included in the model as a single layer of elements in the thickness direction, which allowed to drastically reduce the model size. The substrate was meshed with the three-dimensional 8-noded SOLID185 elements.

Convergence study has shown that the used finite element mesh is adequate to accurately reproduce thermal deformation of the model (see Appendix B).

3.2.6 Thermomechanical simulation results

As mentioned above (see section 3.2.2), the Mo/Si and W film constituents were not explicitly modeled in thermal simulations. Due to this reason it is more convenient and appropriate to discuss the corresponding thermal results referring to the blanket Si wafer instead of the X-ray mirror.

Steady-state results of thermal simulations are presented in the text below. Figures 35(a) and 35(b) show expanded half-symmetric plots of temperature distribution and the through-thickness temperature gradient in Model 1. As seen from the figures, the region of the highest temperature of about 314.5 °K is located in the vicinity of the X-ray beam exposed area. The coldest points lie farthest from the beam irradiated area with the temperature of about 312.2 °K. The highest temperature gradient arises in the glass substrate at the intersections of its outer edges with the Si substrate/glass interface. Overall, the temperature gradient in the Si is much lower than in glass, which can be explained by the higher thermal conductivity of the former.
Simulation plot of the average heat transfer coefficient $h_c$ shown for the Si wafer of Model 1 is presented in Figure 36. This result exactly corresponds to the previously calculated values of $h_c(\Delta T)$ (see Figure 33) given the temperature distribution shown in Figure 35(a).

Si wafer on Si substrate combination (Model 2) reveals a different behaviour from Model 1 in terms of the temperature distribution in the structure. The temperature is essentially uniform (Figure 37(a)) with a value of approximately $313.7 \, ^\circ\text{K}$, which lies between the minimum and maximum temperature values of Model 1. Simulation results show a very small temperature gradient in the structure (Figure 37(b)) which, as already stated above, can be explained by the high thermal conductivity of Si. The highest temperature gradient is observed in the beam exposed area.

Simulation results showed that Model 3 (Si wafer on invar) and Model 4 (Si wafer/glass/Si wafer) demonstrated similar temperature distribution as was calculated for Model 1. These results are not presented.

The Si monochromator is exposed to a 133 times higher heat flux than the X-ray mirror. Temperature distribution in this case is presented in Figure 38(a). Obviously, the natural convection to air fails to effectively remove such high amount of heat, which leads to the temperature rise to
Figure 37. Thermal simulation results for Model 2 (Si wafer on Si)

about 500 °C. The hottest points lie in the vicinity of the beam exposed area whereas, the coldest ones are located at the side-wall edges, as expected. The through-thickness temperature gradient (Figure 38(b)) reaches maximum values of about 7600 K/m along the beam heated area.

Figure 38. Thermal simulation results for Si monochromator (Model 5)

In the structural mechanics simulations, deformation of the X-ray optical element was modeled as linear elastic. It was assumed that the structure is free from stresses and strains at the ambient (initial) temperature. In what follows, simulation results demonstrate deformation behaviour of the optical element corresponding to the calculated temperature distributions presented above.

Figures 39(a)–39(d) show equally scaled (magnification factor 2000) deformed shapes of the Models 1–4. The dashed lines show the model contour outlines in the undeformed state. This side-by-side comparison reveals that the X-ray mirror on glass substrate (Model 1) and the X-ray mirror on Si substrate (Model 2) have the highest and the smallest geometry distortions among the four cases. Similarly, it can be observed, that the X-ray mirror on invar substrate (Model 3) gets more distorted, than the [X-ray mirror/glass substrate/Si wafer] structure (Model 4). Here, the geometry distortion is understood as deviation from the original shape. Since Model 2 has
negligible influence of the thermal expansion mismatch between, as discussed above, the only way geometry distortions may occur here is via the through-thickness temperature gradient. However, by comparing the initial model contour outline (dashed lines) with the deformed shape (Figure 39(b)) it is evident that the structure experiences only linear volumetric expansion without change in shape. Thus, the through-thickness temperature gradient (Figure 35(b)) is too small to cause any observable mirror distortions. Now, recalling that the through-thickness temperature gradient for Model 1 (Figure 35(b)) and in much the same way for Models 3 and 4 is close to the results of Model 2 (away from materials corners), it can be inferred that impact of the temperature gradient on geometry distortions is negligibly small. Therefore, the thermal expansion mismatch between the Si wafer and the thick substrate is the only relevant source of the optical element distortions for the obtained temperature fields. The deformed shape results agree very well with this assumption. Since Si/glass combination has a higher thermal expansion mismatch than Si/invar, the former would have higher geometry distortions given the equal temperature difference. In the essentially symmetric case of the [X-ray mirror/glass/Si wafer] structure (Model 4) the thermal expansion mismatch between Si
and glass is compensated, which leads to comparatively small geometry distortions of the optical element.

In the above text the X-ray mirror geometry distortions were described and mutually compared based on the qualitative visual observations using the simulations plots. Now, let us turn our attention to the quantitative description of the mirror deformation behaviour.

Geometrical distortions of the structure result in curvature of the reflective mirror surface. In order to compute surface curvatures, the corresponding simulation results were imported from the ANSYS model database followed by the computation steps described in Appendix A.

The contour plots in Figures 40(a)–40(d) present Gaussian curvature of the X-ray reflective surface for Models 1–4, respectively. Since the model is quarter-symmetric, only one-fourth of the surface is shown. It can be observed, that the central area of the mirror has a uniform curvature for all simulated models. In the vicinity of the outer edges the curvature of the surface is nonuniform changing its sign and value. This behaviour is attributed to the well known edge singularity effect [Bog71], which is connected to the elastic mismatch between the bonded materials. It could be mentioned, that the edge singularity could have been better captured by the finite element model.
either by decreasing element size near the material corners or by utilization of different element
types, however it was not intended in the present work.

Comparing the uniformly curved parts of the surface, the conclusions drawn from the deformed
shape plots can be confirmed. Particularly, the values for Model 2 (X-ray mirror on Si substrate) are
close to zero indicating that the surface remains flat. Model 1 (X-ray mirror on the glass substrate)
has the highest curvature. The compensated X-ray mirror on glass (Model 4) performs better than
X-ray mirror on the invar substrate, although the latter combination of materials has a lower thermal
expansion mismatch.

The actual area of interest of the mirror which reflects the incoming X-rays is only 1 mm wide
(see Figure 32). Curvature of this area is therefore critical for the beam conditioning. To illustrate
curvature of the beam exposed area, the curves shown in Figure 41 were extracted along the sym-
metry plane (xz). Considering the uniform portion of the curves away from free edges, the Gaussian
curvature of Model 1 is about 8 times higher than for Model 3. Model 4, in turn, has the curvature
of more than order of magnitude smaller, than Model 3. The curvature of Model 2 cannot be distin-
guished from zero on the plot. It is necessary to note that the right portion of the curves close to the
edge depicts the deformation singularity at the corners of dissimilar bonded materials, as mentioned
earlier.

![Figure 41. Comparison of Gaussian curvature along the X-ray beam line for different model config-
urations](image)

The surface curvatures in longitudinal and transverse directions to the beam application line are equal because of negligible effect of temperature gradients in the structure and the assumed materials isotropy.

It is useful to compare surface curvature of the X-ray mirror models using the surface unflatness parameter (see equation (27) in section A). Results of the surface unflatness are illustrated in Figures 42(a)–42(d). Comparison shows their full correspondence to the Gaussian curvature behaviour discussed above. The figures also show the integral value of the surface unflatness

$$U_{surf} = \int_S udS,$$  \hspace{1cm} (14)

in case one needs to compare the overall deviation of surface from flatness.

Figure 42. Unflatness of surface for different model configurations

Figure 43 illustrates the surface unflatness curves extracted in the longitudinal direction of the mirror along the beam application line. Considering only the uniform parts of the curves away from the surface edges, the unflatness parameter $u_{line}$ is about 3 and 10 times higher for reflective surfaces of Model 1 than for Models 3 and 4, respectively. Unflatness of Model 2 is zero. It is important to
note that the right portion of the plot starting approximately from 0.025 m demonstrates nonuniform surface curvature due to the mentioned above edge singularity and should be disregarded.

Figure 43. Comparison of surface unflatness along the X-ray beam line for different model configurations

Table 6 summarizes results illustrated in Figures 42 and 43. The values of unflatness along the beam line $u_{\text{line}}$ correspond to the uniform part of the curves. In cases when the whole surface area is utilized to reflect the X-ray beam, it may be necessary to compare the corresponding integral parameter. The values of unflatness over the surface are also listed in the table. Note, that due to the free edge singularity, caution must be taken when interpreting these values (especially for Model 4).

<table>
<thead>
<tr>
<th>Model Variation</th>
<th>$u_{\text{line}}, \text{1/m}^2$</th>
<th>$U_{\text{surf}}, \text{m}^2/\text{m}^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Model 1</td>
<td>$5.88 \times 10^{-3}$</td>
<td>$1.55 \times 10^{-6}$</td>
</tr>
<tr>
<td>Model 2</td>
<td>$5.95 \times 10^{-6}$</td>
<td>$1.77 \times 10^{-9}$</td>
</tr>
<tr>
<td>Model 3</td>
<td>$2.05 \times 10^{-3}$</td>
<td>$5.51 \times 10^{-7}$</td>
</tr>
<tr>
<td>Model 4</td>
<td>$5.78 \times 10^{-4}$</td>
<td>$2.10 \times 10^{-6}$</td>
</tr>
</tbody>
</table>
3.3 Conclusions for Chapter Three

Analysis of thermally-induced Si wafer deformations showed that a spherical curvature with the target radius of 10 m can be achieved by application of a constant through-thickness temperature gradient of about 20 °C to a 525 μm thick wafer. Taking the room temperature as the lowest temperature of operation, the found temperature gradient lies within the prescribed temperature range with the upper limit, which is 100 °C. This indicates good potential for achieving the necessary curvature in X-ray optics applications using the thermal loading. However, to achieve the required parabolic surface curvature, further finite element studies involving optimization procedures need to be conducted.

Deformations of X-ray optics bonded to the thick substrates, due to the X-ray beam irradiation, were analyzed with the help of sequential thermal and structural finite element simulations. Results of thermal simulations showed that a low power X-ray beam with intensity of 1 W/cm² heats up the optical element under the natural surface convection to air conditions up to about 15 °C with the resulting almost uniform temperature distribution in the structure. Consequently, it was shown that the thermal expansion mismatch between the Si wafer and the thick substrate is the major influencing factor causing thermal deformations of the optical element. Results of the finite element simulations and the surface curvature analysis revealed that, the best performance in terms of the lowest geometry distortions of the X-ray mirror were achieved when the thermal mismatch is compensated. Thermal analysis of a Si monochromator exposed to a high energy X-ray beam showed that a more efficient heat removal than the natural convection to air is necessary to avoid its overheating.
This work was organized in two parts. The first part (Chapter Two) discussed experimental observations of cracks propagation and formation of periodic crack patterns in the annealed Mo/Si multilayer film deposited on the Si substrate. In the second part (Chapter Three), analysis of thermal deformations in the X-ray optics elements was carried out using the finite element modeling.

In Chapter Two, results of stress measurements during thermal cycling have shown that high tensile stresses arise as a result of h-MoSi$_2$-phase formation when the Mo/Si multilayer is annealed and cooled down to the room temperature. Microscopic observations have shown that these tensile stresses lead to cracking and delamination of the film from the Si substrate forming the periodic crack patterns. The cracks propagation has been recorded using a high-speed camera and analyzed. These observations revealed that interaction of a propagating crack and the film delamination causes such peculiar patterns. Based on these observations, a qualitative description has been given that explains the conditions, under which the phenomenon occurs.

The conducted experimental observations on cracking in the annealed Mo/Si multilayers has demonstrated that the crack patterns can easily be reproduced in a simple experiment. However, recording of the cracking event is a challenging task. An important open question still to be definitively answered is whether the channeling cracks propagate behind or before the delamination front. This result could be used for further interpretation and building of a numerical model to accurately capture the cracking behaviour. A higher image resolution of the microscope and the camera could provide the necessary experimental evidence. The analysis of the results can also be improved by conducting the temperature and film stress measurements during the microscopy observations.

In Chapter Three, the finite element modeling was used to analyze deformation behaviour of the X-ray optics elements due to the thermal loading caused by the X-ray irradiation. Here, it has been shown that a through-thickness temperature gradient could be an effective way of controlling
curvature of a Si wafer coated with the Mo/Si reflective multilayers. This initial assessment needs to be further elaborated to include the optimization procedure for finding the optimum thermal loading conditions and the structural composition in order to produce the desired wafer surface curvatures. In addition, it has to be verified if the found optimum thermal loading is realistic to be achieved by the readily available means supported by the experimental evidence.

Thermomechanical analysis of deformation behaviour in the X-ray mirrors bonded to the thick substrates can be used to minimize geometry distortions of the optics reflective surface. Different models representing various structural compositions and used materials have been analyzed. In case of the low power X-ray beam irradiation, the study has shown that the optics deformations are primarily caused by the thermal expansion mismatch between the thick substrate and the Si wafer. The thermal simulation results have indicated that a high power density X-ray synchrotron source would result in excessive overheating of the Si monochromator unless it is effectively cooled.

Refinement of these analyzes could be achieved by comparing the simulation results with experimental measurements of the X-ray mirror temperature and deformations. Knowledge of the heat flow coming into the system due to the X-ray beam exposure could also contribute to the accuracy of the model.
References


<table>
<thead>
<tr>
<th>Reference</th>
<th>Author(s)</th>
<th>Title</th>
<th>Source</th>
</tr>
</thead>
</table>


[Sta] DESY Staff. From personal communication.


Appendices
Appendix A: Surface curvature

Deformation of X-ray optics causes geometry changes of the reflective surface. Depending on geometry, material properties, structural composition, boundary conditions etc., the resulting surface distortions may exceed the prescribed tolerance values. In addition, the final surface geometry may take complicated shapes prohibiting simple analysis of the distorted state on the reflective performance.

Differential geometry is a powerful tool for the analysis of surface geometry properties. However, as of time of writing, the current version of the finite element program ANSYS, utilized during this work, does not contain postprocessing procedures for computation of surface properties, such as curvatures. In the course of this work the author has implemented the necessary numerical procedures in a separate Python-based [VR08] program code for postprocessing of the output simulation results.

This section briefly reviews material of differential geometry applied to analysis of the X-ray mirror deformations, discussed in section 3.2. The review is followed by outlining some aspects of numerical implementation. Thereafter, the program code verification is carried out by comparing numerically computed surface curvatures with the analytical results.

A surface can be defined in parametric form when the surface coordinates are expressed as functions of two independent variables \( u \) and \( v \) [I.74]:

\[
\begin{align*}
x &= \phi(u, v), \\
y &= \psi(u, v), \\
z &= \omega(u, v).
\end{align*}
\] (15)

It is assumed that the above functions are single-valued, continuous and have continuous derivatives up to the second order at some domain of change of \((u, v)\).

Besides the coordinate description in terms of \((x, y, z)\) values, one can describe a surface by defining a variable radius-vector \( \mathbf{r}(u, v) \) going from some fixed point \( O \) to a point \( M \) at the surface. Partial derivatives of this radius-vector with respect to the parameters \((u, v)\) give tangent vectors to the coordinate lines \( \mathbf{r}'_u, \mathbf{r}'_v \).
Appendix A (Continued)

A unit normal vector to the surface can then be written in terms of the partial derivatives of the radius-vector, as follows:

\[ \mathbf{m} = \frac{\mathbf{r}_u' \times \mathbf{r}_v'}{|\mathbf{r}_u' \times \mathbf{r}_v'|}. \quad (16) \]

In expanded form the vector cross product is

\[ \mathbf{r}_u' \times \mathbf{r}_v' = \left( \frac{\partial y}{\partial u} \frac{\partial z}{\partial v} - \frac{\partial z}{\partial u} \frac{\partial y}{\partial v} \right) + \left( \frac{\partial z}{\partial u} \frac{\partial x}{\partial v} - \frac{\partial x}{\partial u} \frac{\partial z}{\partial v} \right) + \left( \frac{\partial x}{\partial u} \frac{\partial y}{\partial v} - \frac{\partial y}{\partial u} \frac{\partial x}{\partial v} \right). \quad (17) \]

Coefficients of the first fundamental form are given by

\[ E(u, v) = r_{uu}'' = \left( \frac{\partial x}{\partial u} \right)^2 + \left( \frac{\partial y}{\partial u} \right)^2 + \left( \frac{\partial z}{\partial u} \right)^2, \]
\[ F(u, v) = r_{uv}'' = \frac{\partial x}{\partial u} \frac{\partial x}{\partial v} + \frac{\partial y}{\partial u} \frac{\partial y}{\partial v} + \frac{\partial z}{\partial u} \frac{\partial z}{\partial v}, \quad (18) \]
\[ G(u, v) = r_{vv}'' = \left( \frac{\partial x}{\partial v} \right)^2 + \left( \frac{\partial y}{\partial v} \right)^2 + \left( \frac{\partial z}{\partial v} \right)^2. \]

For cases when \( F = 0 \), the coordinate lines \( u = C_1 \) and \( v = C_2 \) are orthogonal.

In addition, it can be shown that

\[ |\mathbf{r}_u' \times \mathbf{r}_v'| = EG - F^2. \quad (19) \]

Coefficients of the second fundamental form are given by expressions

\[ L = r_{uu}'' \cdot \mathbf{m}, \quad N = r_{vv}'' \cdot \mathbf{m}, \quad M = r_{uv}'' \cdot \mathbf{m}. \quad (20) \]
Or, by recalling the expressions (16) and (19) one can rewrite the equations (20) as

\[
L = \frac{\mathbf{r}_{uu}'' \cdot (\mathbf{r}_u' \times \mathbf{r}_v')}{\sqrt{EG - F^2}}, \quad M = \frac{\mathbf{r}_{uv}'' \cdot (\mathbf{r}_u' \times \mathbf{r}_v')}{\sqrt{EG - F^2}},
\]

\[
N = \frac{\mathbf{r}_{vv}'' \cdot (\mathbf{r}_u' \times \mathbf{r}_v')}{\sqrt{EG - F^2}}.
\]

(21)

In the case of explicitly given surface \( z = f(x, y) \), \( x \) and \( y \) play a role of parameters that results in the following expressions for the radius-vector constituents and its derivatives:

\[
\mathbf{r}(x, y, z), \quad \mathbf{r}_x'(1, 0, p), \quad \mathbf{r}_y'(0, 1, q),
\]

\[
\mathbf{r}_{xx}''(0, 0, r), \quad \mathbf{r}_{xy}''(0, 0, s), \quad \mathbf{r}_{yy}''(0, 0, t),
\]

(22)

where

\[
p = \frac{\partial f}{\partial x}, \quad q = \frac{\partial f}{\partial y}, \quad r = \frac{\partial^2 f}{\partial x^2}, \quad t = \frac{\partial^2 f}{\partial y^2}, \quad s = \frac{\partial^2 f}{\partial x \partial y}.
\]

(23)

Applying formulas (18) and (21) one arrives at the following expressions for coefficients of the two fundamental forms:

\[
E = 1 + p^2, \quad F = pq, \quad G = 1 + q^2,
\]

\[
L = \frac{r}{\sqrt{1 + p^2 + q^2}}, \quad M = \frac{s}{\sqrt{1 + p^2 + q^2}}, \quad N = \frac{t}{\sqrt{1 + p^2 + q^2}}.
\]

(24)

Mean and Gaussian curvatures of a surface can be obtained from the following expressions:

\[
H = \frac{EN - 2FM + GL}{2(EG - F^2)}, \quad K = \frac{LN - M^2}{EG - F^2}.
\]

(25)
Appendix A (Continued)

Principal curvatures \( P_{\min} \) and \( P_{\max} \) are then found from

\[
P_{\min} = H + \sqrt{H^2 - K}, \quad P_{\max} = H - \sqrt{H^2 - K}.
\] (26)

Deviation from flatness is a useful way of measuring the local unflatness, which can be presented in the following form:

\[
u = P_{\min}^2 + P_{\max}^2.
\] (27)

Consider a 2-dimensional array of function values \( z \) defined on a mesh grid \((x, y)\). The base parameters \( p, q, r, t, s \) (see equations (23)) are obtained by applying numerical gradient [Oli06] to the function \( z \). For example, the first derivatives with respect to \( x \) and \( y \) directions are

\[
\begin{pmatrix}
p \\
q
\end{pmatrix}
= \nabla z = \begin{pmatrix}
z_x \\
z_y
\end{pmatrix}.
\] (28)

Analogously, the second derivatives are calculated by applying the numerical gradient to the previously found values \( p \) and \( q \). After all the base parameters \( p, q, r, t, s \) are found, computation of the fundamental forms (equation (A)) and the surface properties is straightforward.

As an example for verification of the implemented numerical procedure, consider a parabola of revolution \( z = x^2 + y^2 \). The function was discretized on a grid of 100 \( \times \) 100 points in the following interval

\((x, y) = (-2, 2; -2, 2)\).

Figure 44 shows contour plot of the discretized function on the \( xy \)-plane. Gradations of gray color represent the corresponding values of \( z \).
Appendix A (Continued)

Applying analysis procedure described above one can find analytical expressions for the mean and Gaussian curvatures, as follows

\[
H = \frac{2 + 4x^2 + 4y^2}{(1 + 4x^2)(1 + 4y^2)\sqrt{1 + 4x^2 + 4y^2}},
\]

(29)

\[
K = \frac{4}{(1 + 4x^2)(1 + 4y^2)(1 + 4x^2 + 4y^2)}.
\]

(30)

Numerically calculated values of the surface curvatures are presented in Figures 45(a) and 45(b). As expected, the maximum values are reached at the axes origin with values of 2 and 4 for the mean and the Gaussian curvatures, respectively. Note that the curvature functions retain the axisymmetry of the examined function \(z(x, y)\).

Side-by-side comparison of the results calculated analytically using equations (29) and (30) with the numerical data extracted along the \(y = 0\) axis is shown in Figure 46. Excellent agreement can be observed.

It can be concluded that the implemented numerical code is capable of accurately calculating the geometrical surface properties provided the input data has adequate discretization.
Appendix A (Continued)

(a) Mean curvature

(b) Gaussian curvature

Figure 45. Curvature of function $z = x^2 + y^2$ on $100 \times 100$ grid points

Figure 46. Accuracy of numerically computed curvatures on $100 \times 100$ grid points for $y = 0$
Appendix B: Finite element models convergence study

Convergence study of the finite element model representing the X-ray mirror subjected to the through-thickness temperature gradient was performed using a finer finite element mesh, presented in Figure 47. Size of the elements in the radial and the through-thickness directions (refer to Figure 28) was taken 2.0 and 1.5 times smaller than for the coarse model. For the mesh representing the Mo/Si and tungsten layers, the elements size in thickness direction was not varied.

Figure 47. Finite element model used for mesh convergence study of the X-ray mirror subjected to the through-thickness temperature gradient

Figures 48 and 49 show distribution of the total radial strains and the out-of-plane displacements in the X-ray mirror subjected to the through-thickness temperature gradient calculated using the finer finite element model.

Figure 48. Total radial strains in the X-ray mirror due to the through-thickness temperature gradient calculated using the fine mesh

Convergence study was also performed for the model representing the X-ray mirror exposed to an X-ray beam, described in section 3.2.5. Comparison of the coarse and fine finite element
Appendix B (Continued)

Figure 49. Out-of-plane displacements of the X-ray mirror due to the through-thickness temperature gradient calculated using the fine mesh

Meshes used for the convergence study is shown in Figure 50. For the in-plane direction, size of the elements of the fine mesh (Figure 50(b)) is 4 times smaller compared to the coarse mesh (Figure 50(a)), which was used throughout the work (see section 3.2). The number of elements in the thickness direction in the glass substrate was also increased by a factor of 1.25.

Figure 50. Finite element model meshes used for convergence study (Model 4)

Comparison of the calculated average heat transfer coefficient (Figures 51(a) and 51(b)) shows that the coarse and the fine models give essentially the same result.
Appendix B (Continued)

Figure 51. Calculated average heat transfer coefficient (Model 4)

Results of the temperature and the through-thickness gradient distributions calculated using the coarse and the fine models are presented in Figures 52 and 53. Comparison of the figures shows that the coarse and the fine models produce almost identical results. Slight differences, however, can be observed for the temperature gradient results at the intersections of the free edges and materials interfaces, as seen from the Figures 53(a) and 53(b).

Simulation plots of the out-of-plane displacements $u_z$ calculated using the coarse (Figure 54(a)) and the fine (Figure 54(b)) models show, that there are only minor differences between them. Finally, it can also be concluded, that the finite element mesh of the coarse model is fully sufficient to reproduce the thermal deformations of the X-ray mirror.
Appendix B (Continued)

Figure 53. Calculated through-thickness temperature gradient (Model 4)

Figure 54. Calculated out-of-plane displacements $u_z$ (Model 4)