



Thermal and stress studies of the 30.4 nm Mo/Si multilayer mirror for the moon-based EUV camera



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ARTICLE INFO

Article history:

Received 4 June 2014

Received in revised form 8 August 2014

Accepted 31 August 2014

Available online 6 September 2014

Keywords:

X-rays
Soft X-ray
Extreme ultraviolet (EUV)
Multilayers
Thermal stability
Residual stress

ABSTRACT

To investigate the environmental adaptability of the Mo/Si multilayers on lunar surface, we studied the stability and stress of Mo/Si multilayers under the low and high temperature environment. The *in-situ* X-ray diffraction (XRD) and the *ex-situ* intrinsic stress are measured in the temperature range from -135°C to 600°C and from -190°C to 600°C , respectively. The results demonstrate that the periodic structure of Mo/Si multilayers is stable between -135°C and 300°C . The stress is unaffected under low temperature and it gradually increases from -260 MPa to 1 G MPa when the temperature changes from room temperature to 600°C . Above 600°C , large tensile stress leads to folds and cracks in the film. Thus, the large temperature range on lunar surface has little effect on the structure, performance and stress of the Mo/Si multilayers and the high temperature in lunar day releases the stress of the multilayer mirror.

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1. Introduction

The Earth's plasmasphere, which strongly interacts with the ionosphere, ring current and radiation belt, is the core region for the interaction in the inner magnetosphere [1,2]. The evolution process of the plasmasphere affects the structures of the inner magnetosphere and the near-Earth space environment. Therefore, detection of the plasmasphere is valuable for both scientific researches and applications. On December 14, 2013, the Chinese Chang'E-3 (CE-3) mission has been landed on the lunar surface at Sinus Iridum, afterwards, the moon-based extreme ultraviolet (EUV) camera [3] which was developed by CIOMP (Changchun Institute of Optics, Fine Mechanics and Physics, Chinese Academy of Sciences) as a payload of the CE-3 lunar lander has imaged the Earth's plasmasphere at 30.4 nm. This is the first global observation of the Earth's plasmasphere on the moon. It will provide more information about the impacts of solar activities on the Earth's space environment.

The optical system of EUV camera is mainly composed of a Mo/Si multilayer mirror and a photon counting imaging detector. All the components operate in a harsh environment of lunar

surface. Because of the very rareness atmosphere of lunar surface, the direct solar radiation leads to a very high temperature of about 127°C during the lunar day while temperature will drop to -183°C due to the absence of solar radiation during the lunar night. Mo/Si multilayer mirror is the key optical component of the EUV camera, and the mirror's optical characteristics determine the performance of the EUV camera. So, its adaptability to the harsh temperature environment of lunar surface is critical and must be investigated in detail.

The reflectance of Mo/Si multilayers at 30.4 nm is only around 20%, which is lower than the Mg-based [4] and Al-based [5] multilayers. However, the Mo/Si multilayers have a better stability [6] and have been used in different space instruments [7,8]. The thermal stability of Mo/Si multilayers has been reported in several literatures [9–11], in which the variations of the film thickness and the reflectivity of Mo/Si multilayer mirrors from 25°C to 600°C were investigated. Little work has been devoted to the performance of EUV multilayers at low temperature. Moreover, they only measured the samples' periodic structure in room temperature (RT) after annealing and the structure at different temperatures were not studied. In this paper, *in-situ* and real-time X-ray diffraction (XRD) is used to measure the structure of Mo/Si multilayers at fixed temperature in the range from -135°C to 600°C , which covers the typical operation temperatures of the multilayers on lunar surface.

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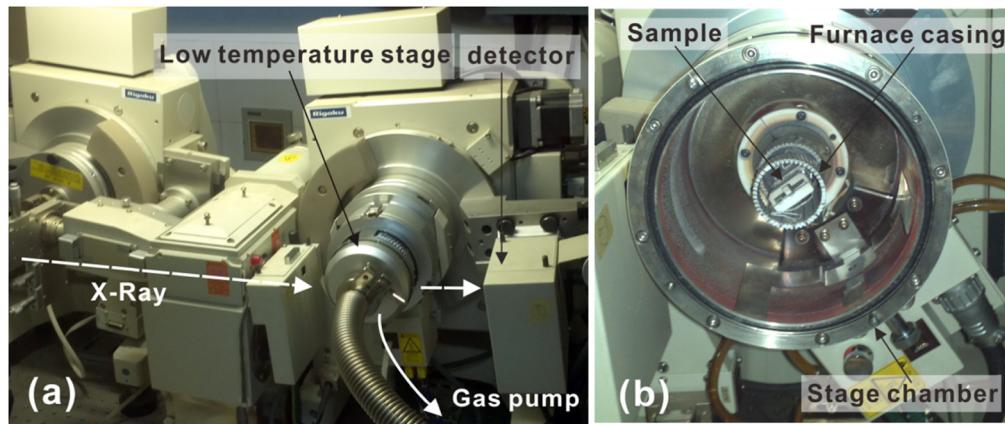


Fig. 1. (a) Rigaku DMAX2000 X-ray diffractometer (equipped with low temperature stage), (b) high temperature stage.

The residual stress changes with temperature and the film may be disfigured or cracked when the stress is large enough. Therefore, it's very necessary to investigate the relationship between stress and temperature of Mo/Si multilayers. Several authors have reported such works [5,12,13], but all of them focused on the research of the multilayers at wavelength of 6~14 nm for EUV lithography at high temperatures, few studies have been done at wavelength of 30.4 nm, and the stress of EUV multilayers at low temperature also have been neglected. The intrinsic stress is strongly affected by the periodic thickness, Gamma value ($\text{Gamma} = d_{\text{Si}}/(d_{\text{Si}} + d_{\text{Mo}})$) and materials. All these parameters at 30.4 nm are different from those at 6~14 nm, so the work for 30.4 nm is very necessary. This paper studies the variations of the stress at temperatures between -190°C and 600°C for 30.4 nm Mo/Si multilayers. In addition, the relationship between intrinsic stress and periodic structure of Mo/Si multilayers as a function of the temperature is also investigated.

2. Experimental details

2.1. Mo/Si multilayers synthesis and characterization

The experiments were composed of the *in-situ* XRD measurements and the *ex-situ* intrinsic tests in low and high temperature. The substrates used in the *in-situ* XRD measurements were (100) silicon wafers with a thickness of 1.5 mm, and a surface roughness (RMS) of 1.2 nm. The substrates used in the *ex-situ* intrinsic tests were silica substrates with a diameter of 33 mm, a thickness of 1.5 mm, and a surface roughness (RMS) of 0.6 nm. All the Mo/Si multilayers with a Gamma value of 0.7 were deposited by direct current (DC) magnetron sputtering coating machine that has been described previously [14]. The number of period is 30, and the periodic thickness is 16.5 nm.

X-ray diffraction (XRD) was performed by the Rigaku DMAX2000 X-ray diffractometer which uses the Cu K α line ($\lambda = 0.154 \text{ nm}$) with an angle resolution of 0.005° as shown in Fig. 1.

The surface shape of each substrate was measured before and after coating using zygo GPI XP/D interferometer to calculating the sample's curvature. Then, the film stress can be calculated by Stoney equation [15]:

$$\delta = \frac{E_s}{6(1-\nu_s)} \frac{t_s^2}{t_f} \left(\frac{1}{R} - \frac{1}{R_0} \right) \quad (1)$$

where δ is the stress in the multilayers, $E_s = 71.7 \text{ GPa}$ is the Young's modulus, $\nu_s = 0.17$ is the Poisson's ratio. t_s is the thickness of the substrate, t_f is the thickness of the multilayers, and R_0 and R are

the curvatures of the substrate before and after film deposition respectively.

EUV reflectances of the multilayers were measured by EUV/soft X-ray reflectometer (EXRR) near normal incidence angle of 7° . The measured reflectance repeatability and the wavelength repeatability of EXRR were $\pm 1\%$ and $\pm 0.04 \text{ nm}$, respectively. Details of EUV/soft X-ray reflectometer were described in ref [16]. The surface profilometer (New View 6000STP, zygo) was used to measure the surface roughness, and the atomic force microscopy (AFM) was used to measure the surface morphology.

2.2. *In-situ* XRD measurements

The *in-situ* XRD measurements were performed at the Rigaku DMAX2000 X-ray diffractometer. As seen from Fig. 1(a), the diffractometer is equipped with a low temperature stage, which is replaced by the high temperature stage in Fig. 1(b). The low and high temperature stage in combination with the programmable temperature controller maintain samples at different temperatures and executes the X-ray diffraction while the temperature is increasing or remains constant temperature. The samples can be measured in vacuum, air, or an inert gas. During the low temperature experiments, the temperature cooled down by the liquid nitrogen with a cooling rate of $7^{\circ}\text{C}/\text{min}$, and the sample was kept at RT (22°C), -50°C , -100°C , -135°C for an hour before each measurement. During the high temperature experiments, the sample was placed in the furnace casing of high temperature stage to be heated with a heating rate of $5^{\circ}\text{C}/\text{min}$, and it was kept for an hour from 100 to 600°C with the step of 100°C before each measurement. The stage chamber was vacuumed to 0.1 Pa by mechanical pump during the high temperature measurements.

2.3. *Ex-situ* intrinsic stress tests

The sample was treated in the vacuum chamber at different temperature from -190°C to 600°C , respectively. The thermal annealing was performed in a molybdenum heating furnace with a base pressure of $2 \times 10^{-5} \text{ Pa}$ from RT to 600°C with a step of 100°C and each step was held for 1 h. The low temperature treatment was performed in another vacuum chamber whose temperature can decrease to -190°C and can be maintained for 1 h. After the treatment at each temperature, the *ex-situ* intrinsic stress tests were performed at zygo GPI XP/D interferometer at RT.

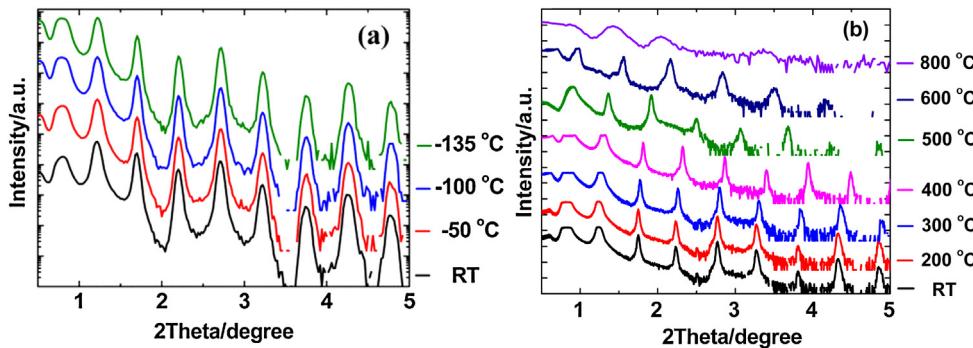


Fig. 2. SAXRD curves of Mo/Si multilayers as a function of temperature. (a) Low temperature, (b) high temperature.

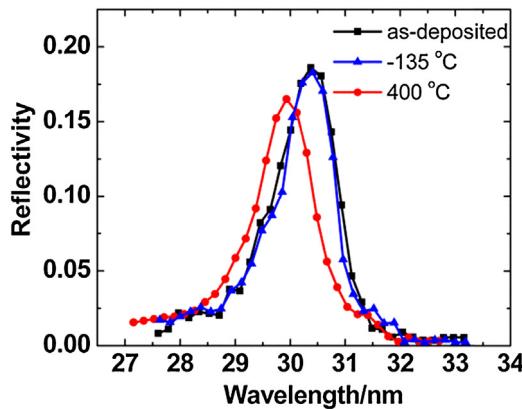


Fig. 3. Reflectance at 7° incidence of as-deposited sample and the sample at -135°C , 400°C .

3. Results and discussion

3.1. SAXRD

Fig. 2(a) shows a series of SAXRD curves of the Mo/Si multilayers at different temperatures below RT. For clarity, the reflectivity curves of the annealed samples are shifted upward successively. It is revealed that there is no significant change in the intensity of the Bragg peaks under different temperature, but the angles of the Bragg peaks increase slightly, indicating that the periodic thickness of the multilayers decreases with temperature. The thickness is decreased by $\sim 0.9 \text{ \AA}$ at -135°C . The decreasing of the periodic thickness at low temperature is due to thermal constriction of the solids. Nevertheless, such little change has no effect on the performance of multilayers. Fig. 2(b) shows a series of SAXRD curves above RT. It is shown that there is no obvious change below 300°C while dramatic changes appear above 400°C . The minor changes in the angles of the Bragg peaks below 300°C reveal that there are little inter-diffusion between layers. At and above 400°C , both the intensities and angles of the Bragg peaks change dramatically. The peaks are still clear at 400°C although there are clear increases in the angles of peaks. Above 500°C , however, the intensities of the Bragg peaks strongly decrease with the angles of the peaks increasing significantly and high order peaks disappear, indicating an increasing inter-diffusion and a formation of Mo silicide alloy. At 800°C , the absence of sharp Bragg peaks and the low intensities of the XRD curve show that the periodic structure of multilayers has been completely destroyed. Changes in the multilayers are also reflected in the reflectance as shown in Fig. 3. The reflectance wasn't affected by the low temperature treatment at -135°C at all. However, after the high temperature of 400°C annealing, the peak reflectance decreased

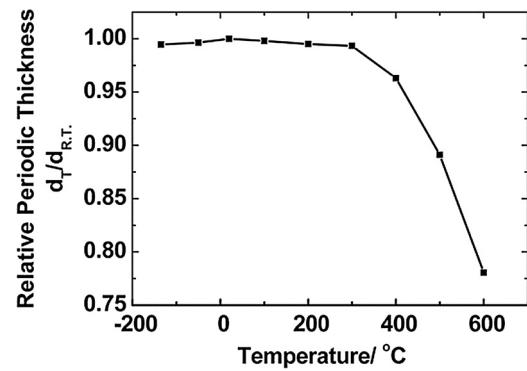


Fig. 4. Relative periodic thickness of Mo/Si multilayers as a function of temperature.

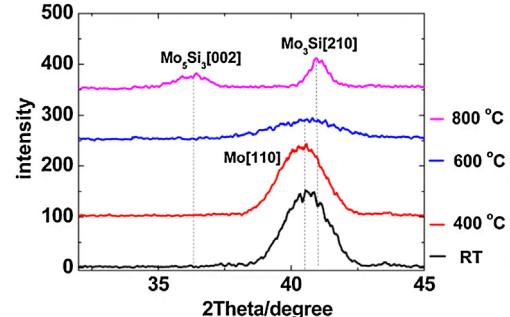


Fig. 5. HAXRD curves of Mo/Si multilayers as a function of temperature.

by 2.1% and the center wavelength shifted by 0.5 nm towards short wavelength.

Fig. 4 shows the relative periodic thickness of the multilayers as a function of temperature. It demonstrates that the periodic structure changes little at temperatures between -135°C and 300°C . Although the highest temperature during lunar day is $\sim 130^{\circ}\text{C}$ and the lowest temperature during lunar night is $\sim -180^{\circ}\text{C}$, the thermal control system in EUV camera can ensure that the temperature of the multilayer mirror is between -20°C and 90°C , and the Mo/Si multilayer mirror can operate normally on lunar surface.

3.2. HAXRD

Because the EUV camera mainly work during lunar day with high temperature, we have performed a series of HAXRD measurements to assess the effect of high temperature on the structure of the multilayers. The HAXRD curves at temperatures from RT to 800°C are shown in Fig. 5. As shown in Fig. 5, there is only a Mo (1 1 0) diffraction peak at RT, and it remains stable at 400°C . As the

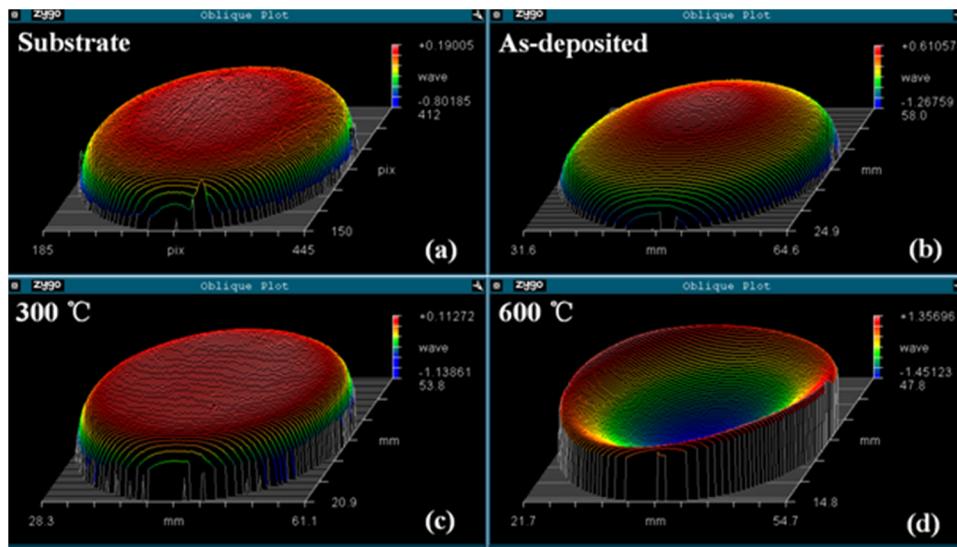


Fig. 6. Surface shape of Mo/Si multilayer mirror measured by interferometer as a function of temperature (a) substrate, (b) as-deposited, (c) 300 °C, (d) 600 °C.

temperature increases to 600 °C, the full-width at half maximum (FWHM) increases with a significant decrease of the peak intensity of Mo (110). This is due to the inter-diffusion of Si into Mo, resulting in destruction of the crystalline structure of the Mo layer [11]. The results are the decrease of periodic thickness as shown in Fig. 4. At 800 °C, the peak of Mo (110) disappears and two Mo silicide diffraction peaks marked as Mo₅Si₃ (002) and Mo₃Si (210) appear. This indicates that Mo silicide alloy have been crystallized [17]. Simultaneously, the periodic structure has been completely destroyed which is demonstrated in Section 3.1.

3.3. Residual stress

The residual stress is strongly affected by the temperature [12]. Therefore, it's very necessary to study the relationship between the stress and the temperature of Mo/Si multilayers. The film stresses can be roughly classified into three types [9]: thermal stress, coherence stress and intrinsic stress. The first two types are generated by mismatch of the thermal expansion coefficient and the crystalline lattice parameter respectively between the substrate and the first layer during the deposition. The intrinsic stress is resulted from the structure evolution inside the film during the film growth. The effect of the coherence stress is small because the first Si layer has an amorphous structure. The thermal stress of the multilayers can be calculated by equation (2) [18]:

$$\delta_{th} = \frac{E_f}{1 - \nu_f} (\alpha_s - \alpha_f)(T - T_0) \quad (2)$$

where δ_{th} is the thermal stress of the multilayers, $E_f/(1 - \nu_f) = 2.36 \times 10^5$ MPa is the biaxial modulus of the film. α_s and α_f , the thermal expansion coefficient of the substrate and multilayers, is 55 ppm/°C and 4.8 ppm/°C [19], respectively. T is the deposition temperature of 38 °C, and T_0 is the measuring temperature of 22 °C (RT). From equation (2), δ_{th} is determined to be -16 MPa (compressive), which is much lower than the as-deposited stress of -260 MPa. Therefore, the contribution of intrinsic stress dominates in our experiment.

The multilayers deposited by the magnetron sputtering system with low pressure exhibit properties of high density and compactness due to the strong collisions of energetic Ar ions with the material. The peening-effect [20] during the deposition process results in high compressive stress of the films. Fig. 6 presents the surface shape of the substrate and the Mo/Si multilayers at different

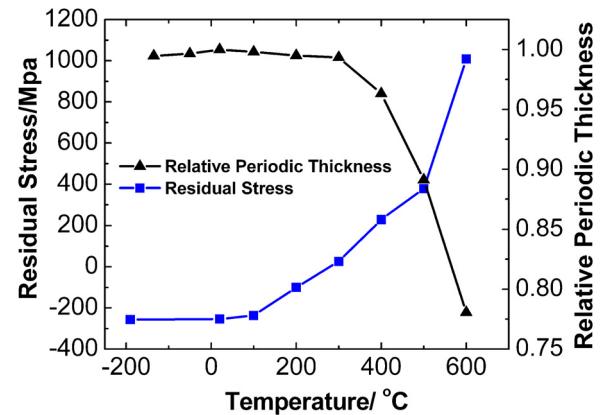


Fig. 7. Stress and relative periodic thickness of Mo/Si multilayers as a function of temperature.

annealing temperature, the corresponding stress is given in Fig. 7 and Table 1. The uniform stress of -260 MPa at RT as shown in Fig. 7 makes the surface shape approximately like a sphere in Fig. 6(b). When the annealing temperature increases from 100 °C to 500 °C, the stress decreases almost linearly. In this temperature range, the stress transforms from compressive to tensile and achieves zero at ~300 °C when the surface shape returns to the state of the uncoated substrate as is shown in Fig. 6(a) and (c). As the temperature further increases from 300 °C on and the inter-diffusion between Mo and Si becomes significant as shown by HAXRD in Fig. 5, the stress increases rapidly to about 1 GPa at 600 °C in Fig. 7 and the surface shape concaves downward in Fig. 6(d). However, after -190 °C

Table 1

Stress and relative periodic thickness of Mo/Si multilayers as a function of temperatures between -190 °C and 600 °C.

T (°C)	σ (MPa)	Relative periodic thickness
As-deposited	-260	1
-190 °C	-263	1
100 °C	-237	0.998
200 °C	-100	0.995
300 °C	26	0.993
400 °C	229	0.963
500 °C	378	0.891
600 °C	1008	0.78

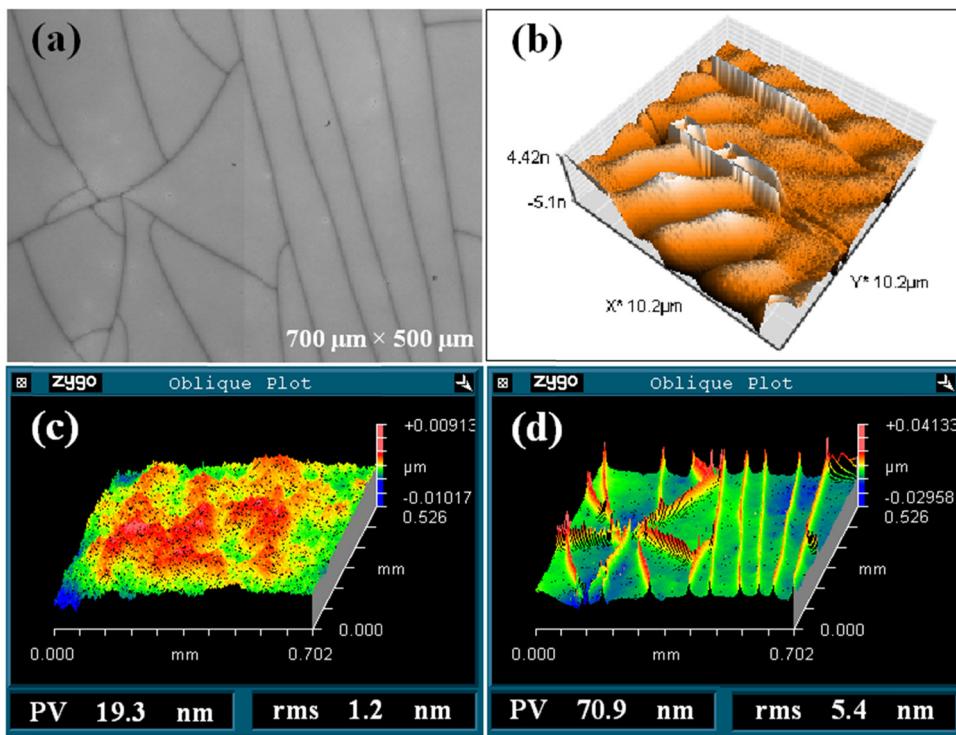


Fig. 8. (a) Surface morphology image of Mo/Si multilayers after 600 °C annealing measured by optical microscope (b) AFM image ($10 \mu\text{m} \times 10 \mu\text{m}$) of Mo/Si multilayers after 600 °C annealing (c) surface roughness measuring pattern of as-deposited Mo/Si multilayers (d) surface roughness measuring pattern of Mo/Si multilayers after 600 °C annealing.

treatment, the stress of multilayers only increases 3 MPa compared with the as-deposited stress of -260 MPa , indicating that low temperature has no effect on the stress of the multilayers. In addition, compared with the similar studies by Montcalm [21], the initial stress of our films is -260 MPa , which is lower than their studies of -410 MPa due to the different technological parameters, but the linearly relationship between the stress and temperature is similar, and both of the stress reduce to 0 MPa at $\sim 300^\circ\text{C}$.

The correlation between the residual stress and the relative periodic thickness of Mo/Si multilayers as a function of the temperature in Fig. 7 and Table 1 demonstrates that the compaction of the Mo/Si multilayered structure results in the change of stress. It is known that the compaction of Mo/Si multilayered structure is the combined results of free volume annihilation (porosity defects reduction) and interlayer formation [22]. Moreover, de Rooij-Lohmann et al. [13] found that all free volume was annihilated after annealing at 325°C . According to previous studies and the results in Section 3.1 of this paper that the inter-diffusion of Mo/Si multilayers starts after 300°C , we could divide the process of stress changes into two phases. In the first phase, from RT to 300°C , the free volume annihilation is dominant without interdiffusion, and the stress decreases almost linearly. In the second phase, from 300°C to 600°C , the free volume are all disappeared and the increasing inter-diffusion effect results in the formation of interlayer which dominates the changes of stress. From 500°C to 600°C , the relative periodic thickness contracts rapidly from 0.89 to 0.78 and large amount of Mo silicide alloy forms as demonstrated by the HAXRD results in Fig. 5. Simultaneously, the stress decreases abruptly from 378 MPa to 1008 MPa as shown in Table 1.

Consequently, the temperature from -190°C to 150°C on lunar surface has little effect on the stress of Mo/Si multilayer mirror and the high temperature in lunar day releases the stress of the multilayer mirror.

3.4. Surface morphology

To further investigate the changes of the films at high temperature, the samples are measured using optical microscope, AFM, and surface profilometer, as shown in Fig. 8. At 600°C , the stress increases to 1 GPa , and such high stress results in significant folding and cracking of the multilayers as shown in Fig. 8(a) and (b). Moreover, the surface roughness (RMS) increases from 1.2 nm to 5.4 nm after 600°C annealing as shown in Fig. 8(c) and (d). Thus, the multilayers is completely damaged because of the so large stress after 600°C annealing.

4. Summary

We have studied the performance, structure, stability and stress of Mo/Si multilayers at low and high temperature environments. The *in-situ* XRD measurements demonstrate that the structure of Mo/Si multilayers is stable from -135°C to 300°C . Above 300°C , interlayer and Mo silicide alloy appear because of inter-diffusion. The measured peak reflectance declines by 2.1%, and the center wavelength shifts by 0.5 nm towards short wavelength at 400°C . At 800°C , the Mo silicide compounds have been crystallized to Mo_5Si_3 (002) and Mo_3Si (2 1 0). In the stress measurements, it is found that the stress of the as-deposited Mo/Si multilayers is -260 MPa , which increases by only 3 MPa even at the low temperature of -190°C but changes almost linearly as the annealing temperature increases from 100°C to 500°C during when the stress transforms from compressive to tensile at 300°C . The extremely large tensile stress at 600°C results in folding and cracking of the multilayers. According to these results, it is concluded that the large temperature range on lunar surface has little effect on the structure and performance of the Mo/Si multilayers and the high temperature in lunar day can help release the stress of the multilayer mirror. Furthermore, because the EUV camera will experience several thermal cycles

during its mission on the lunar surface, the evolution of periodic structure and stress with numerous thermal cycles require further investigation.

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