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Periodic cracks and temperature-dependent stress in Mo/Si multilayers on Si substrates

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ABSTRACT

This work examines formation of the peculiar periodic crack patterns observed in the thermally loaded Mo/Si multilayers. Using the substrate curvature measurements, the macroscopic film stress evolution during thermal cycling was investigated. Then high-speed microscopic observation of crack propagation in the annealed Mo/Si multilayers was presented providing experimental evidence of the mechanism underlying formation of the periodic crack patterns. The origin of the peculiar periodic crack patterns was determined. They were observed to form by the slow crack propagation under quasi-static conditions as a result of the interaction between the channelling crack propagation and the advance of the delamination front.

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1. Introduction

Patterns containing seemingly random almost straight cracks are common to coatings and thin films in residual tension. They maybe observed in everyday life on painted surfaces, asphalt pavement and so on. On the contrary, periodic crack patterns with peculiar shapes are observed relatively seldom in nature, as their formation may require a favourable combination of different effects, such as temperature gradients, externally applied stress, influence of boundaries and asymmetry. First notion of helix-like crack patterns in residual tension in Pyrex glass plates was published in [1]. Oscillating cracks along a large temperature gradient in a glass plate rapidly immersed into water were reported in [2]. 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 centre line with the largest tensile stresses. From the experiments conducted with drying precipitates on substrates

[3], 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 [4]. Experimental observations of the simultaneously running outwards spiral, sinusoidal, saw-tooth and crescent-like crack patterns in single-layered drying sol-gel silicate thin films on glass and steel substrates were reported in [5]. Based on the experimental data, it was suggested that the observed curving crack paths were attributable to film delamination. Recently, the research group of [6,7] has been investigating the propagation of two fractures in the single-layered organic silicate sol-gel films (on silicon substrates) connected by a delamination front. Using numerical and experimental approaches, they proposed the conditions for the crack propagation under tensile loading and the selection of the fracture paths with a physical model. Observations of spiral crack paths and other interesting patterns can be found in other papers [8–10].

Sinusoidal and spiral crack patterns were observed in the Mo/Si multilayers deposited on the Si substrates. The samples were subjected to the three-point bending, annealed at high temperature of about 500 °C and slowly cooled in a vacuum chamber, followed by their microscopic observations [11]. Similar to observations of other authors in references [3,5], 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 caused these periodic crack patterns.

This work investigates the formation of the peculiar crack patterns observed in the thermally loaded Mo/Si multilayers on Si substrates. Film stress evolution during thermal cycling is analysed based on the substrate curvature measurements. The crack propagation and crack patterns evolution captured using the high-speed photography are presented and discussed in detail. The observations provide experimental evidence of the mechanism underlying formation of the periodic crack patterns.

2. Experimental details

To investigate the stress evolution in the Mo/Si multilayers on Si substrates due to the thermal cycling, the biaxial residual stress in the film was continuously calculated with the Stoney's equation. The Mo/Si multilayers were deposited on (1 0 0) Si substrates using magnetron direct current sputtering. The sputtered Mo/Si film consisted of 60 alternating layers with the corresponding thicknesses of 2.7 and 4.2 nm each. The Si substrate was 525 µm thick with the diameter of 100 mm. Three heating cycles with the peak temperature of 500 °C were applied to the system. More details of the heating cycles will be discussed in the next section together with Figure 1.

For the microscopy analysis of the crack patterns, high-speed camera was used. The same samples were annealed using a high-temperature oven capable of heating

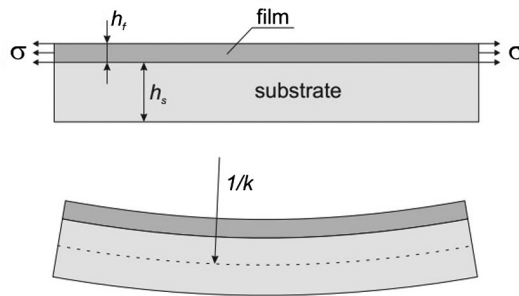


Figure 1. Substrate curvature caused by stress in the film.

up to 1100 °C. Prior to annealing, the investigated samples were cut-out from the wafer into $2 \times 2 \text{ mm}^2$ pieces using a diamond scribe. The cutting procedure induced defects and flaws on the edges of the (1 0 0) Si wafer. The cut-out samples were placed onto a thick ceramic plate and annealed in the oven at 500 °C for 20 min. 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 from one to 5 min. To observe the crack growth, the samples were placed onto a steel stage of the microscope covered with a 1 mm-thick ceramic plate. Ceramic plate was used to thermally insulate the sample from the microscope steel stage and slow down sample cooling during optical observations. To record the crack propagation in time a high-speed digital camera Philips SPC/1300NC, which is the capable of recording 90 frames per second (fps) with the resolution of up to 320×240 pixels, was attached to a long-range optical microscope. 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.

3. Results and discussion

3.1. Stress evolution in the Mo/Si multilayers stack as a function of temperature

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 [12] that stress in the film strains the substrate, and so it bends (Figure 1).

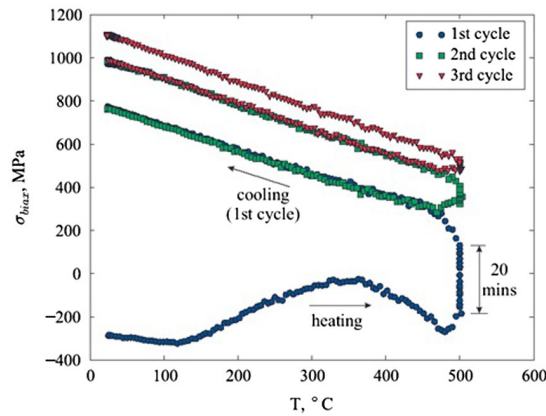


Figure 2. (colour online) Stress evolutions in the Mo/Si multilayers on Si substrate due to the thermal cycling.

The biaxial stress in the film can be calculated from the substrate curvature using the Stoney's equation:

$$\sigma = \frac{kM_s h_s^2}{6h_f} \quad (1)$$

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:

$$M_s = \frac{E_s}{1 - \nu_s} \quad (2)$$

where E_s and ν_s are the elastic modulus and the Poisson's ratio of the substrate material, respectively. Note, that the Stoney formula in Equation (1) does not contain material properties of the film, only those of the substrate. Effects of the film thickness on substrate curvature in bimetals were first analysed in detail in reference [13].

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 [14]. 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 (discussed in Section 3.2).

Stress evolution in the Mo/Si multilayers stack as a function of temperature is presented in Figure 2. Three heating cycles with the peak temperature of 500 °C were applied to the system. In the initial state at room temperature, the total

stress in the Mo/Si multilayers (further referred as the film stress) is compressive, about -360 MPa.

The first heating cycle contains different stages of the film stress evolution. One can see that there are four following stages:

- (1) Stage one ($23 < T < 110$ °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 Equations (3) and (4), which can be found in the latter section. Since the coefficient of thermal expansion of the Mo/Si multilayers stack is higher than Si (due to the Mo layers), increase in temperature results in the proportional increase of compressive stress in the Mo/Si multilayers.
- 2) Stage two ($110 < T < 375$ °C): relaxation of the compressive film stress due to inter-diffusion between the Mo and Si layers. Results of the X-ray diffractometry investigations carried out by [15] show that the Mo/Si multilayers are thermally stable for temperatures below 110 °C. Heating above this temperature stimulates inter-diffusion between the Mo and Si layers. According to [16], the hexagonal MoSi_2 -phase (h- MoSi_2) nucleates at the temperature as low as 275 °C. However, the crystallisation does not take place until the temperature is raised to 375 °C.
- (3) Stage three ($375 < T < 475$ °C): rapid increase of compressive stress in the Mo/Si multilayers during formation of the h- MoSi_2 phase.
- (4) Stage four ($475 < T$ and $T = 500$ °C for 20 min): stress increase in the Mo/Si multilayers changing from compressive to tensile after 10 min. In the crystallisation process, the denser h- MoSi_2 phase consumes Mo and Si materials 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 crystallisation process continues until the temperature falls below 475 °C. This short portion can also be characterised by a rapid increase of tensile stress in forming the 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 and 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 475 °C, where the stress starts to increase rapidly, which is 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. At 475 °C, the two competing mechanisms equilibrate each other. The rate of the crystallisation 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 crystallisation process occurred in the first cycle, the increase of the film stress is now much smaller, although the annealing times were equal. The cooling portion of the curve is linear apart from the temperatures close to 500 °C where the crystallisation 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 in tension.

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 the formation of the h-MoSi₂ phase indicates that almost all Mo and Si elements were consumed in the crystallisation process. The film stress reaches 1100 MPa when the structure is cooled to room temperature.

3.2. The thermal expansion coefficient and elastic mismatch strain of the film

The multilayered Mo/Si film and the substrate have different coefficients of thermal expansion. Therefore, the thermal mismatch strain of the film with respect to the substrate is determined as:

$$\varepsilon_{th} = (\alpha_s - \alpha_f)\Delta T \quad (3)$$

where α_s and α_f are the coefficients of linear thermal expansion of the substrate and the film, respectively; ΔT is the change of temperature. Then the corresponding mismatch stress is:

$$\sigma_{th} = \varepsilon_{th}M_f \quad (4)$$

where M_f is the biaxial modulus of the film. From Equations (1) and (2), the coefficient of thermal expansion of the film will be determined as:

$$\alpha_f = \alpha_s - \frac{\sigma(T)}{M_f(T - T_{ref})} \quad (5)$$

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

Similar to the Stoney's stress formula [12], the elastic mismatch strain of the film with respect to the substrate will also be calculated from the curvature measurements:

$$\varepsilon_m = \frac{kM_s h_s^2}{6M_f h_f} \quad (6)$$

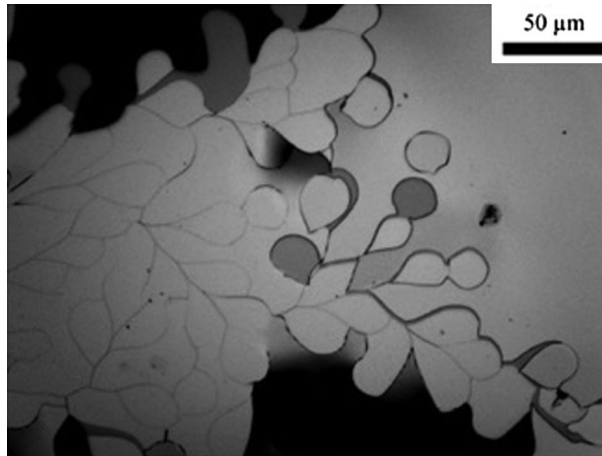


Figure 3. Tree leaf-like branching channelling cracks.

where h_s and h_f are the thicknesses of the substrate and the film, respectively; M_s is the biaxial modulus of the substrate and k is the curvature of the substrate resulting from the stress of the film.

3.3. Optical observations

The optical observations of the crack propagation in the annealed Mo/Si multilayers using the high-speed photography showed that in most cases, no cracks were usually observed in the Mo/Si films on Si wafers annealed at 500 °C, but the cooling of the tested samples led to the formation and growth of cracks in the Mo/Si films. It also can be seen that the cooling rate of the samples correlated with the speed of cracks propagation in the annealed Mo/Si films and only the slow propagating cracks revealed the 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 the sample was brought in contact with the microscope stage and as the sample cooled down. 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 simultaneously observing the whole surface of the sample.

During the cooling process, there were some periodic crack patterns observed, such as tree leaf-like, grass-like, square-sinusoidal-like, sinusoidal-like, saw tooth-like and spiral cracks. Figure 3 shows the tree leaf-like cracks, which were formed by the crack 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 black area at the upper left corner of this image represents a piece of a broken delaminated film, which was displaced by a

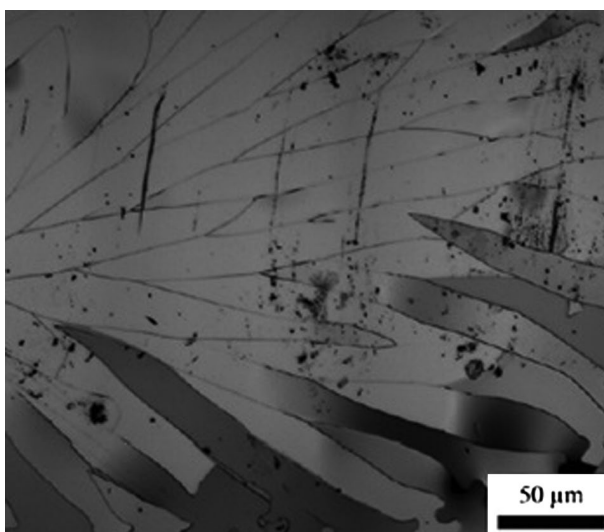


Figure 4. Grass-like pattern cracks.

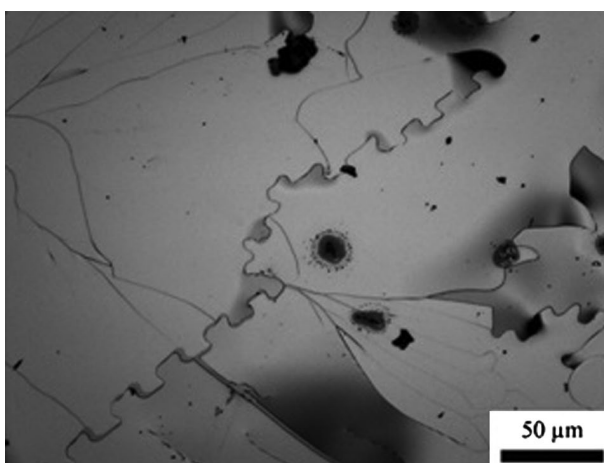


Figure 5. Square periodic channelling cracks accompanied by delamination.

large distance as a result of its sudden delamination and cracking. Figure 4 shows a grass-like pattern, which was formed as a result of growth and branching of the channelling cracks, accompanied by their intersection and termination. The square periodic crack (Figure 5) discovered in the annealed Mo/Si films is believed to be a new type of a crack pattern that was not previously reported in the literature, to the best knowledge of the authors. It can be observed that the period and the amplitude of the pattern remained constant as the crack propagated. Figure 6 presents the sinusoidal-like film cracks accompanied by delamination. Regarding the relation between the delamination and the crack patterns, the authors in reference [17] suggested that local delamination would reduce the effective toughness

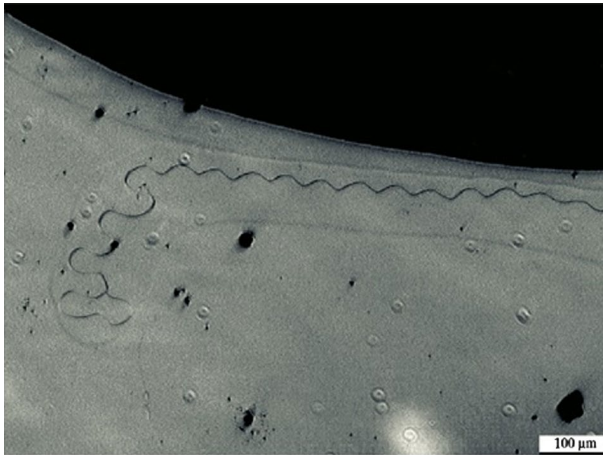


Figure 6. Sinusoidal channelling cracks accompanied by delamination.

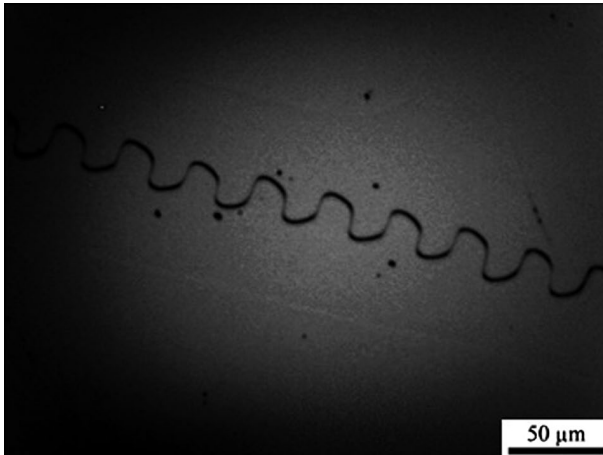


Figure 7. Saw tooth-type channelling crack accompanied by delamination.

of the film, which is equivalent to the role of the interlayer in their study. In the image, the delamination can be recognised as a slightly raised area around the crack relative to the rest of the sample surface. The delamination follows and completely encloses the channelling crack, which terminated by looping on itself. Since stress in the film is tensile, such closed delamination cannot take place alone in the film, as opposed to the case of buckling delamination under compressive film stresses [18,19]. In the present case, the channelling crack releases the film edges necessary for the delamination to take place. During crack propagation the direction, amplitude and period of the pattern may suddenly change, as also seen from the microscope images (Figure 6). However, the undisturbed crack propagation under the uniform conditions results in a constant geometry of the crack pattern, as presented in Figure 7 showing a saw tooth-like crack pattern.

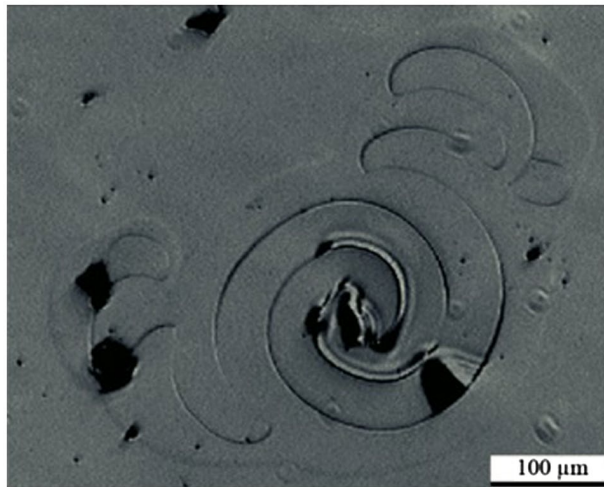


Figure 8. Spiral channel cracking accompanied by delamination.

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 centre line. Spiral crack patterns were also found in the annealed Mo/Si film. The example representing such cracks is shown in Figure 8. Close to the imaginary centre, the channel cracks show some similarity with an Archimedes spiral. After propagating approximately three spiral cycles, the crack formed a crescent pattern followed by termination and looping on it. 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 films. One may suggest, that these patterns form under much slower cooling rates, which provide uniform thermal loading conditions [11].

To shed light on the process of the crack patterns formation results of high-speed photography are presented. Figure 9 shows selected frames captured during propagation of a straight channelling crack in the annealed Mo/Si film. 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 orthogonal to the crack direction continues to advance in time and is much slower than the film crack. The average speed of the channelling crack is approximately 0.4 mm/s, which is relatively slow compared to the cracks forming the leaf or grass-like patterns. Unfortunately, magnification and resolution of the optical equipment does not allow to unambiguously determine whether the delamination runs ahead of the channelling crack or vice versa. However, it should be noted that the frames f-40 and f-55 may indicate that the channelling crack runs behind the advancing delamination.

Formation of the periodical crack patterns from the bursting grass-like pattern cracks, discussed above, was captured using the fast-motion Photron camera. Figure 10 presents different stages of the crack pattern formation recorded at a

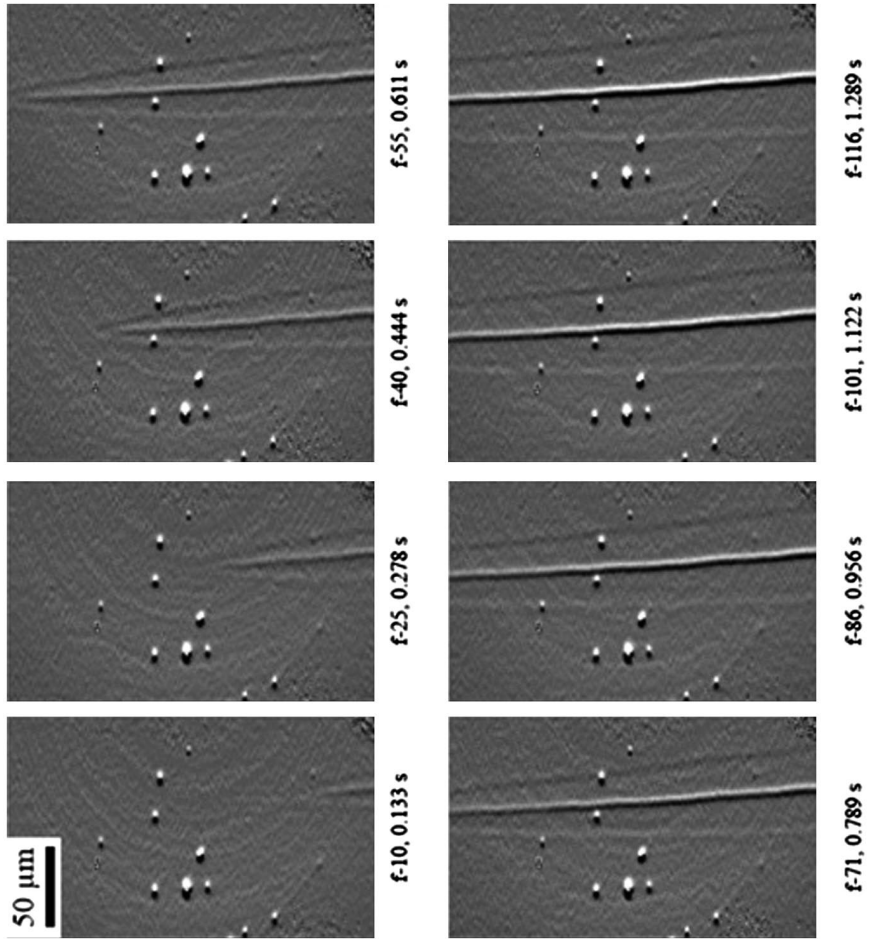


Figure 9. Propagation of a straight channelling crack accompanied by delamination at selected instants of time.

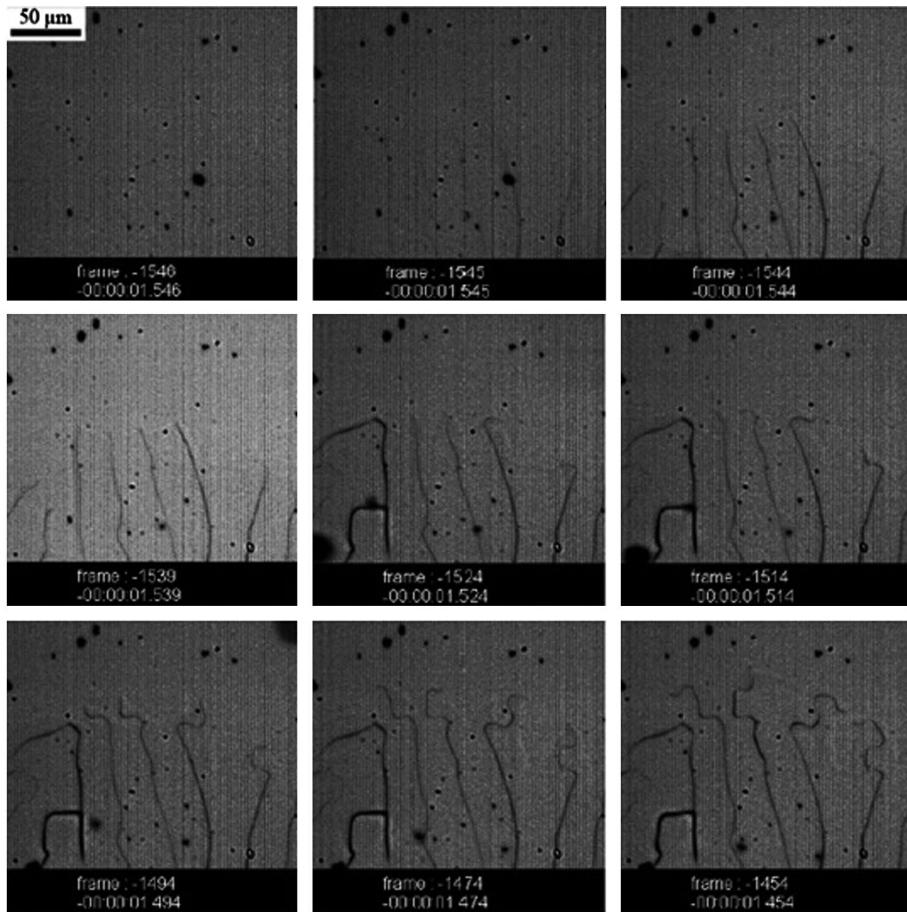


Figure 10. Selected frames showing growth of the bursting grass-like channelling cracks followed by the formation of the periodic crack patterns.

rate of 1000 fps. Comparing the two first frames (frame-1546 and frame-1545) the average crack propagation speed of at least 200 mm/s can be estimated. The third frame (frame-1544) reveals that after 0.002 s the cracks slow down substantially. The fast crack growth was always observed to be accompanied by large displacement of the dust particles initially resting on the film surface. These flying particles suggest that the fast growth of the channelling 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 11 presents frames showing the formation of the periodic crack patterns selected every 0.005 s. At all stages the channelling cracks interact with the

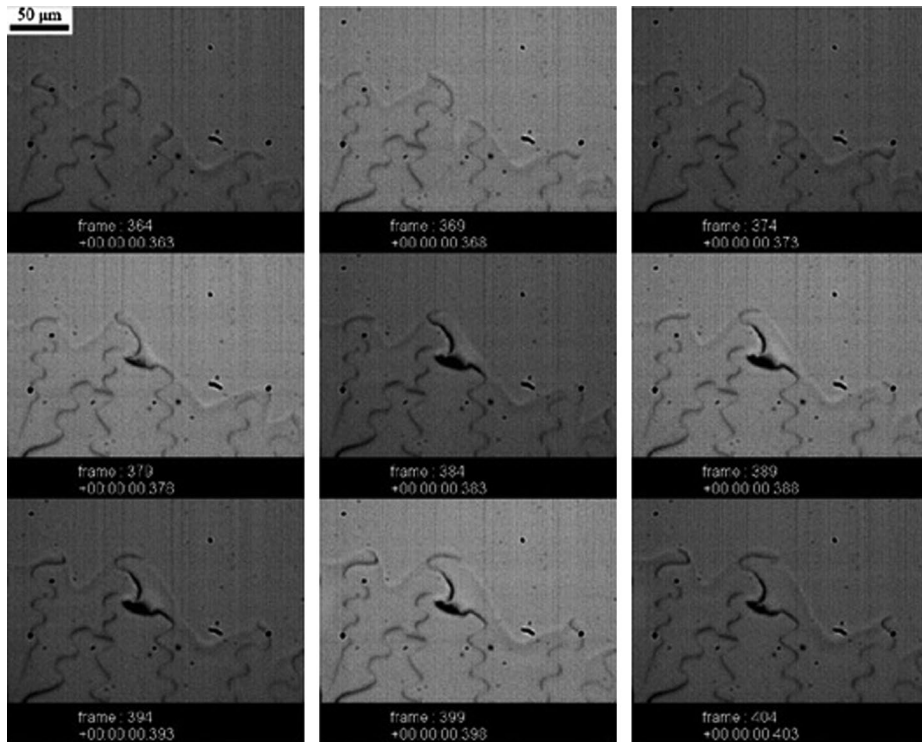


Figure 11. Selected frames showing formation of the sinusoidal crack patterns.

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 channelling cracks as the latter provide the necessary free film edges. However, it is seen that after delamination attributed to a channelling crack the former also continues to grow, although at a slower rate.

The interaction between the channelling crack and the advancing delamination front is the key point in the formation of the observed periodic crack patterns, which is confirmed by the references [6,7]. 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 maybe suggested, that during the slow crack growth phase, the strain energy stored in the film is lower than that necessary for a channelling 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 channelling crack always propagates in close vicinity of the delamination front.

4. Conclusions

Periodic crack patterns were observed in h-MoSi₂ thin films on Si substrates. The h-MoSi₂ phase was formed by thermally annealing Mo/Si thin film multilayers at 500 °C. As a result of this phase transformation and the thermal mismatch between the film and the Si substrate, high tensile film stress arose when the structure was cooled. At a certain temperature, the strain energy stored in the system was 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 slow crack propagation under quasi-static conditions. The thermal expansion coefficient and elastic mismatch strain of the film were also determined based on the substrate curvature measurements.

Microscopic observations using the high-speed camera revealed that the peculiar crack patterns form as a result of the interaction between the channelling crack propagation and the advance of the delamination front. Periodicity of the patterns can be explained by a higher speed of channelling cracks relative to the delamination and by the periodical process when a channelling crack turns into the direction of the self-emitted delamination occurring at certain favourable conditions. These successive crack turns produce the observed periodic crack patterns.

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Disclosure statement

No potential conflict of interest was reported by the authors.

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