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Spatial and kinetic evolutions of telephone cord buckles

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ABSTRACT

We report on the spatial and kinetic evolutions of telephone cord buckles existing in $SiAlN_x$ films sputtered on 6 mm thick glass substrates. The experiment shows that the telephone cord buckles propagate from the film edges to the central regions after annealing. During propagation, the already formed buckle undergoes a reorganization process and it can expand in the longitudinal, transverse and normal directions simultaneously. As a result, various morphological parameters (including wavelength, amplitude, width, maximum deflection etc.) evolve obviously both in spatial scale and time scale, which have been measured and analyzed in detail. The internal stress of the film is also evaluated based on the continuum elastic theory.

1. Introduction

Residual compressive stress is an intrinsic property of thin films deposited on various substrates. To release the residual compressive stress, the films are susceptible to buckling and delaminating from the substrates. The buckling phenomena are found to be strongly dependent on the nature of the substrate. For liquid substrate, the elastic film can move freely on the substrate surface, resulting in the formation of various interesting patterns such as folds [1], smooth cascade of wrinkles [2], rectangular domains [3] and step-like creases [4]. For soft polymer substrate, the film and substrate are conformal during deformation and the film remains well attached to the substrate. The typical wrinkling patterns in this case include period strips, herringbones and labyrinths [5,6]. Because the geometrical structures of these patterns can be precisely controlled and the interfacial adhesion remains good, they have been widely used in the optical and micro-electro-mechanical systems (MEMS), including gratings [7], sensors [8], stretchable electronic components [9], measuring technique [10], masks for lithography [11] and so on. For solid substrate, the elastic film tends to partly or entirely delaminate from the substrate due to the compressive stress. The buckle driven delaminations generally need to be avoided in applications because the structural failure usually leads to the lost of functional properties. However, the delamination phenomena are quite useful for evaluating mechanical properties such as the stress level, adhesion energy and elastic modulus of thin films [12,13]. Therefore, the buckle driven delaminations have been extensively studied in various film/substrate systems.

The typical delaminating modes include straight-sided buckle [14,15], circular blister [16] and telephone cord [14,17]. The mechanism for the straight-sided buckle is now highly understood based on the continuum elastic theory (Föppl–von Karman (FvK) theory) [18,19]. But the mechanism for the telephone cord buckle is still a challenge due to its morphological complexity. The recent works showed that the telephone cord buckles, as well as some circular blisters, can be viewed as equilibrium configurations evolving from the straight-sided buckles, a phenomenon known as "secondary buckling" [20–22]. Faou et al. simulated the process of a propagating telephone cord buckle under an isotropic compressive stress and the results were in good agreement with experiments [23]. However, the detailed morphological evolutions of the telephone cord buckles in a homogeneous film (under the uniform or equi-biaxial stress), which are intrinsic and essential for understanding the mechanics of telephone cord mode, have not been experimentally investigated. In this paper, we report on the spatial and kinetic evolutions of telephone cord buckles in annealed $SiAlN_x$ films on 6 mm thick glass substrates.

2. Experimental details

The $SiAlN_x$ films were prepared at room temperature by an alternating current magnetron sputtering method using the coating production lines (Apollon G 3210/7-H). The rotating target was SiAl alloys (Si 90 at.%, Al 10 at.%). The glass substrate with an area 600×900 mm^2 had a uniform thickness of 6 mm. The deposition chamber was evacuated to a base pressure of 1.2×10^{-4} Pa. Prior to deposition, the substrate was sputter cleaned in argon plasma for 2 h to remove the moisture, oxidation layer and other contaminants completely. Then, a 750 sccm flow rate of Ar gas and a 1000 sccm flow rate of N_2 were injected into the chamber. The working pressure

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was 0.8 Pa, the sputtering power was 70 kW and the moving speed of the sample during deposition was 0.66 m/min. The film thickness, measured by an atomic force microscopy (AFM, XE-100E, PSIA), was about 380 nm. During deposition, the temperature of the substrate would increase obviously owing to the heat radiation from the target and the bombardment of the atoms. The stable temperature of the substrate during deposition, measured by a thermocouple, was about 105 °C.

After deposition, the initial samples were cut into small pieces with about $10 \times 10\ mm^2$ in size. The small samples were put into a 700 °C muffle furnace for 4.5 min. Then the samples were removed from the furnace and naturally cooled in the atmosphere condition. Our experiment shows that the surface of as-prepared $SiAlN_x$ films is flat and free of buckles. After annealing, various buckling patterns start to nucleate at the film edges and then propagate into the central regions of the sample gradually. We suggest that during annealing, the temperature of the whole sample reaches about 700 °C. During cooling, the cooling rate of $SiAlN_x$ film was much higher than that of the substrate for the film thickness is much thinner. The mismatch of the thermal contracts places the $SiAlN_x$ film under a high compressive stress, which is relieved by buckling and delaminating of the film from the substrate.

The buckle morphologies were studied with an optical microscope (Leica DMLM) equipped with a charge coupled device (CCD) camera (Leica DC 300) interfaced to a computer for data processing. To investigate the kinetic evolution of the telephone cord buckles, consecutive micrographs were automatically taken by the optical microscope at regular intervals. The cross sections of the buckles were determined by the atomic force microscopy (XE-100E, PSIA) operated in non-contact mode using an etched single-crystal Si tip with a radius of 10 nm. Collected data consisted of height information on square 256×256 arrays of pixels from area scans with lengths from 10 to 45 μm.

3. Results and discussion

3.1. Spatial evolution of telephone cord buckles

The typical buckling patterns in the $SiAlN_x$ films are shown in Fig. 1. We find that they generally undergo a regular sinusoidal motion in the plane of the film and possess a well-defined period (or wavelength) and amplitude, which are defined as λ and A, respectively (see Fig. 1). The telephone cord mode is very common for it has been proven to permit an optimal equi-biaxial compressive stress release [18,19]. Because the local internal stress near the imperfection of thin film is quite large and the interfacial adhesion energy in the imperfection region is comparatively small [24,25], the buckling patterns always start from the film edges [14,26], substrate dislocations [27] or impurities [15,25,26].

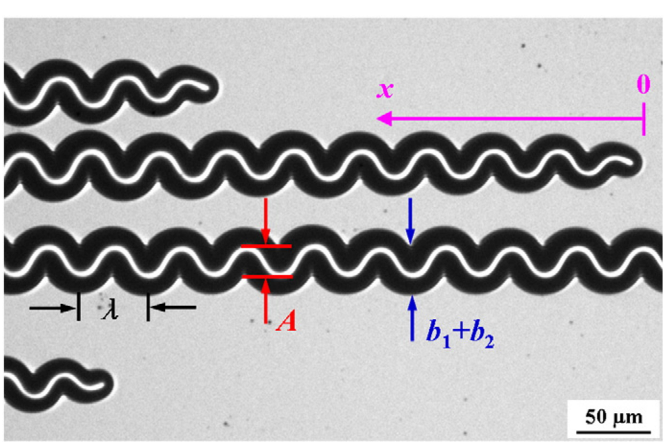

Fig. 1. Typical telephone cord buckles in $SiAlN_x$ films deposited on 6 mm thick glass substrates. The wavelength and amplitude of the telephone cord buckle are denoted as λ and A, respectively. The distance from the propagating tip is denoted as x. The width of the buckle is denoted as $b_1 + b_2$ (details see Fig. 2).

Fig. 2 shows the AFM images and the corresponding profiles at the propagating tip (a) and middle position (b) of the telephone cord buckle. We find that the buckle resembles a straight-sided shape at the propagating tip. When the distance from the propagating tip (namely x, see Fig. 1) increases, the displacement w increases quickly and then it approaches a stable value (see line 1 in Fig. 2(a)). Because the telephone cord buckle is elongated tunnel with anti-symmetric undulation at the edges, the profiles along different trajectories are not identical [17,28]. The profile across the most rounded point of the buckle (i.e., the wave crest or wave trough) is asymmetric. The half-separation towards the center of the curved buckle is always larger than that towards the opposite direction. The smaller and larger half-separations of the buckle are defined as b_1 and b_2, respectively, as shown in Fig. 2(a). Accordingly, the total buckle width is also defined as $B = b_1 + b_2$ (see Fig. 1). The ratio b_1/b_2 mirrors the symmetry degree of the buckle profile. If b_1/b_2 equals one, the buckle profile is completely symmetric. The asymmetry of the buckle morphology means that the left and right slopes of the profile line are not equal, and therefore the energy release rate and interfacial toughness are different at the two sides [29].

When the profile deviates from the most rounded point gradually, the symmetry degree b_1/b_2 increases gradually (see lines 1–5 in Figs. 2(b) and 3(a)). When the profile across the middle point between the two neighboring rounded parts, b_1/b_2 increases to about 1 and the profile becomes symmetric (see line 5 in Fig. 2(b)). In fact, the buckle part between the two neighboring rounded points resembles a straight-sided shape. When the profile deviates from the middle point and moves towards the neighboring most rounded point gradually, b_1/b_2 decreases gradually (see lines 5–9 in Figs. 2(b) and 3(a)). The final profile (line 9 in Fig. 2(b)) is quite similar to the initial shape (line 1 in Fig. 2(b)) except the anti-symmetric characteristic of the buckle. Fig. 3(a) shows that the symmetry degree of the buckle changes slowly near the most rounded point, but it evolves quickly when the profile approaches the middle point. Fig. 3(b) shows the evolution of the smallest symmetry degree at the most rounded point with the distance x. We find that when the distance increases, the smallest symmetry degree decreases drastically first, and then it approaches a saturation value of about 0.65. Furthermore, Fig. 2(b) shows that the maximum deflection at the middle point is somewhat smaller than that at the most rounded point, in agreement with the previous studies [29]. Our experiment also shows that if the profile line is perpendicular to the straight-sided buckle part at the middle point, the buckle width is slightly smaller than that at the most rounded point. It is reasonable that the width and maximum deflection of the straight-sided buckle are somewhat smaller than those of the telephone cord buckle in the same sample [21,30].

It can be seen in Fig. 1 that the dimensions of the telephone cord buckles decay obviously when propagating. In order to further understand the spatial evolution of the telephone cord buckles, we measured the dependences of the wavelength λ, amplitude A, width $B = b_1 + b_2$ and maximum deflection δ on the distance x, and the results are shown in Fig. 4. We find that λ and δ both increase drastically with x at the first several periods, and then they reach the saturation values of about 40 and 1.5 μm, respectively. The evolution behaviors of the amplitude and width are similar: they both increase quickly with x first, then slow down and finally approach the saturation values of about 20 and 32 μm, respectively. The experimental data can be fitted by the same exponential growth equation (see Fig. 4). For example, the buckle width can be fitted by

$$B(x) = B(\infty) - B_{add} e^{-x/x_B} \quad (1)$$