Laser micromachining of sputtered DLC films

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Abstract

DLC films with different thicknesses (from 100 nm to 1.9 μm) were deposited using sputtering of graphite target in pure argon atmosphere without substrate heating. Film microstructures (sp²/sp³ ratio) and mechanical properties (modulus, hardness, stress) were characterized as a function of film thickness. A thin layer of aluminum about 60 nm was deposited on the DLC film surface. Laser micromachining of Al/DLC layer was performed to form microcantilever structures, which were released using a reactive ion etching system with SF₆ plasma. Due to the intrinsic stress in DLC films and bimorph Al/DLC structure, the microcantilevers bent up with different curvatures. For DLC film of 100 nm thick, the cantilever even formed microtubes. The relationship between the bimorph beam bending and DLC film properties (such as stress, modulus, etc.) were discussed in details.

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1. Introduction

Due to its unique properties (high hardness and modulus, low coefficient of friction, high wear resistance and chemical inactivity), diamond-like carbon (DLC) has been considered as an excellent candidate material for microelectromechanical application [1–3]. However, large residual stress seems a problem associated with the DLC films as it may influence the adhesion between coating and substrate, the deformation of microstructures, roughness of the coating as well as the tribological, fracture and fatigue properties [4–7]. For thin films or coatings, the residual stress has two sources: thermal and intrinsic [8]. Thermal stress forms during cooling after deposition, and is due to the difference in coefficient of thermal expansion between coating and substrate. Since the sputtered DLC films were normally deposited without external heating, thermal stress is quite low. The intrinsic stress in the DLC films is mainly caused by the existence of non-diamond components, impurities and structural defects (in which sp²/sp³ fraction is one dominant factor) [9]. It is normally quite difficult to reduce the intrinsic stress without sacrificing film properties. There are some attempts to utilize the large stress to fabricate MEMS devices. For example, a microcage based on highly compressively stressed diamond-like carbon and electroplated Ni bimorph structure has been successfully fabricated in our group as shown in Fig. 1 [10]. The deflection angle of the devices can be adjusted by varying the finger length, DLC stress, the total thickness and thickness ratio of the DLC/Ni bilayer [11].

This study aims to utilize the large intrinsic stress of DLC films to fabricate Al/DLC microcantilever structures based on laser micromachining and MEMS techniques. Laser micromachining refers to a material patterning and removal process where a focused laser beam of high energy density and high frequency scans over the material to create a specific microstructures, without causing much material heating. The advantages of using Al/DLC bimorph structure include: (1) large intrinsic compressive stress in the as-deposited DLC films and (2) large differences in coefficient of thermal expansion of DLC (α ~ 1 × 10⁻⁶) and Al (α ~ 2.7 × 10⁻⁶), making this bimorph device very efficient during thermal actuation.

2. Experimental

DLC films of different thicknesses (from 100 nm to 1.9 μm) were deposited using sputtering of graphite target in
pure Ar atmosphere without intentional substrate heating. The base pressure of the chamber was $2 \times 10^{-6}$ Torr. The substrate holder was rotated during the deposition for uniformity. The substrate-to-target distance was 100 mm, and argon gas flow rate was 15 sccm and pressure was 5 mTorr. The RF plasma power was 150 W. The deposition rate was 12.5 nm/min, and the deposition duration was varied to achieve different film thicknesses. Raman scattering was recorded using a Raman spectroscopy (Renishaw System 3000) with He–Ne laser (wavelength of 514.5 nm). The wafer curvature before and after film deposition was measured using a profilometer, and residual stress, $\sigma$, was calculated from the changes of radius of wafer curvature, $R$, of the bi-layer structure using Stoney’s equation:

$$
\sigma = \frac{E_s t_s^2 R}{6(1 - \nu_s^2) t_f}
$$

(1)

where, $E_s$ is the Young’s modulus of the Si-substrate, $\nu_s$ the Poisson’s ratio and $t_s$ and $t_f$ are the thickness of the Si-substrate and the film, respectively.

Aluminum film of 60 nm thick was sputter-deposited on the DLC films (RF power of 150 W for 5 min). A laser micromachining system (New-Wave QuikLaze, USA) was used in this study with solid-state YAG ultraviolet (UV) laser (frequency of 355 nm). The laser power was 50 W, and the focused laser spot had a dimension of 5 $\mu$m $\times$ 5 $\mu$m. The frequency was 50 Hz, and the scan rate 20 $\mu$m/s. After laser micromachining, etching of Si substrate beneath the Al/DLC films was performed using a reactive ion etching (RIE) system with SF$_6$ plasma. The gas pressure during etching was 100 mTorr, gas flow rate was 70 sccm, and the plasma power was 100 W at a frequency of 13.56 MHz. The etching rate is about 3 $\mu$m/min.

3. Film characterization

Fig. 2 shows Raman spectra of the DLC films with different thicknesses. With the increase of film thickness, there is a gradual shift of Raman peak to lower wave numbers. The Raman curves can be fitted with G-band and D-band components, and the intensity ratios of $I_D/I_G$ of different films have been calculated with the results shown in Fig. 3 [12,13]. With the increase of film thickness, the $I_D/I_G$ ratio decreases initially, but increases with film thickness larger than 300 nm. The results indicate that sp$^2$ component decreases with film thickness increased from 100 to 300 nm. With further increase of film thickness, sp$^2$ content increases with film thickness, i.e., a higher content of non-diamond component for a thick film. Fig. 4 shows the peak positions of G-band and D-band for all the films. G-peak position decreases initially but gradually shifts to high wave number due to the increase in sp$^2$ component. While D-peak position decreases initially until a film thickness of 500 nm, above which the D-peak position increases with film thickness. Because the film stress does not change significantly in this range, the position change in G-peak and D-peak actually comes from the structural change, i.e., the increase in sp$^2$/sp$^3$ ratio (or increase of non-diamond component). Results clearly indicate that there is transition in sp$^2$/sp$^3$ structure with the growth of DLC films [14].
Fig. 5 shows the hardness and modulus results from the nano-indentation tests. Both the hardness and modulus increase with the film thickness from 100 to 300 nm. However, above 300 nm, both the values decrease slightly with film thickness. From the Raman results shown in Fig. 3, it is clear that the change in mechanical properties (such as hardness and modulus) with thickness is due to the change in film structure, i.e., there is a minimum ratio of sp²/sp³ (or \( \frac{I_D}{I_G} \)) for the 300 nm film. The increase of sp³ components (diamond structure) results in the enhanced mechanical properties. Fig. 6 shows the change of the film stress as a function of film thickness. There is an increase in film stress with film thickness increased from 100 to 300 nm, but with further increase in film thickness, the stress gradually decreases, and then varies slightly with film thickness above 1000 nm. The well-accepted reason for this trend is the change in sp²/sp³ ratio in the films.

4. The bending of DLC/Al bimorph structures

Because the DLC film has large compressive stress, while the aluminum film has tensile stress (the measured stress value in Al films with a thickness 60 nm is about 262 MPa, tensile). Once the bimorph structure is released from Si-substrate, the overall residual stress causes the bending of the bi-layer structure. The radius, \( R \), of curvature of such a bimorph structure can be simply expressed by Eq. (2) [15]:

\[
\frac{1}{R} = \frac{6\varepsilon(1 + m)^2}{d[3(1 + m)^2 + (1 + mn)(m^2 + (mn)^{-1})]}
\]

where \( \varepsilon \) is the strain, \( d = d_1 + d_2 \), with \( d_1 \) and \( d_2 \) the thicknesses of the DLC and the Al layer, respectively, \( n = \frac{E_1}{E_2} \) and \( m = \frac{d_1}{d_2} \) the ratios of the Young’s modulus and the layer thickness. \( 1/R \) is a smooth function of \( n \) (or \( m \)) at a fixed \( m \) (or \( n \)), and has a maximum value at \( n = 1 \) (or \( m = 1 \)). A small radius of curvature can be obtained if two materials with similar Young’s moduli are used. Eq. (2) also implies that once the materials and the Al layer thickness are fixed, the radius of curvature can be adjusted by varying the stress and thickness of the DLC layer. Following assumptions are made in calculation of radius of curvature of the DLC/Al bimorph structure: (1) thermal stress in the film is quite low; (2) the stress is uniaxial mechanical loading; (3) the stress of the DLC layer is fully converted into the strain of the DLC layer. Therefore, the strain...
\( \varepsilon \) is related to the stress \( \sigma \) and the Young’s modulus \( E_{\text{DLC}} \) of the DLC layer, by \( \varepsilon = \sigma / E_{\text{DLC}} \). Based on the Eq. (2) and the related measured parameters in Figs. 5 and 6, the radius of curvature can be calculated, and the results are shown in Fig. 7. For a fixed Al thickness, increasing the DLC thickness increases the radius of curvature significantly.

Fig. 8 shows the typical morphologies of the Al/DLC cantilevers fabricated by laser micromachining and RIE etching process. The length of the microcantilevers was varied from 80 to 160 \( \mu \text{m} \). Decreasing the thickness ratios of the two layers makes the cantilever curling dramatically. For the DLC film of 100 nm thick, the Al/DLC cantilever even forms microtubes. Variation in beam length of microcantilever does not show significant change in the bending curvature. The tip deflection of each cantilever was measured and thus the radius of curvature can be estimated with results shown in Fig. 7. The experimental results are quite agreeable with those from the calculated ones.

Once the structure and the dimensions are fixed, the subtended angle, \( \theta \), of the cantilever (or the tip inclination in beam theory) is related to the finger length \( L \) by Eq. (3):

\[
\theta = \frac{180L}{\pi R}
\]

For a fixed material combination and stress state (thus, radius of curvature \( R \) is fixed), increase of beam length can increase the subtended angle, which are the cases for those cantilevers shown in Fig. 8. If the radius of curvature reaches to \( L/2\pi \), a
closed microcantilever could form (i.e., cantilever is curled by 360°). The subtended angles \( \theta \) were calculated according to DLC film thicknesses and beam lengths, and the results are shown in Fig. 9. Take the DLC film of 100 nm as an example, the calculated results confirmed that the DLC/Al bimorph structure can form a microtube (i.e., the subtended angle is larger than 360° as shown in Fig. 8(a and b)). For the cantilevers with a DLC thickness in the range of 300 and 1900 nm, the beam length is too short to form closed microtubes.

The disparities of the experimental and calculated results in Fig. 7 can be explained using the following reasons. Firstly, the residual stress consists of a mean stress and a gradient stress, the latter has not been considered in calculation. DLC film has a large through-thickness stress gradient, which also makes the released layers curl upwards. The stress gradient is normally larger for a thinner film, thus this effect becomes significant when the DLC film is thin. The bending of the bimorph structure can be also due to the thermal stress developed during RIE process. However, this effect is relatively unimportant since the temperature rise is only about tens of degrees.

5. Conclusions

As DLC film thickness increases from 100 to 1900 nm, there are maximum values in the film hardness, modulus and stress. The reasons are well explained through the change in sp²/sp³ ratio as a function of film thickness. Laser micromachining of Al/DLC layer was performed to form the microcantilever structures, which were released using a reactive ion etching system with SF₆ plasma. Due to the intrinsic stress in DLC films and bimorph Al/DLC structure, the microcantilevers bend up to different curvatures. For DLC film of 100 nm thick, the cantilever even forms microtubes. For a fixed Al thickness, increasing the DLC thickness significantly increases the radius of curvature, as is verified by the calculated results based on the cantilever dimensions and film properties.

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