Sub-critical telephone cord delamination propagation and adhesion measurements

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ABSTRACT

Thin film delamination can occur when the stored elastic energy per unit area in the film due to the residual stress exceeds the interfacial toughness. Telephone cord morphology is commonly observed in delaminating thin films under compressive stresses. Here, the biaxial film stress is partially relieved by film buckling in the direction perpendicular to the telephone cord propagation, and by "secondary" blister buckling in the direction of telephone cord propagation, which results in the sinusoidal fracture patterns.

A superlayer indentation test, in which additional stress is supplied to the crack tip using a nanoindenter, can be used to measure the interfacial toughness. Estimates of the energy release rate for diamond-like carbon (DLC) films on magnetic media were obtained using the superlayer indentation test, as well as the delaminated buckling profiles. The results obtained by these two independent methods are in good agreement with each other. We find the average adhesion energy to be 6 J/m^2 for DLC films on magnetic media.

Normally telephone cord blisters "run out of steam" and stop once the interfacial toughness exceeds the strain energy release rate. It is possible to make blisters propagate further by either putting mechanical energy into the system, or by introducing liquids at the crack tip, thus reducing the film interfacial toughness. Environmental species can assist cracking and contribute to thin film delamination, which is readily observed in vintage mirrors. Crack propagation rates on the order of microns per minute were measured for DLC films in different fluid environments. We identify how telephone cord buckling delamination can be used as a test vehicle for studying crack propagation rates and environmentally assisted cracking in thin films.

INTRODUCTION

Thin film adhesion is a very important property, specifically for the microelectronics and the hard drive industry, as well as for the growing field of microelectromechanical systems (MEMS). With the growing number of applications, there is a wide variety of environments that these devices are being exposed to. Designers are not only concerned with the electrical and mechanical properties of the materials in these devices, but also how they will hold up in potentially corrosive environments. In certain applications thin films are used as protective barriers against damage caused by friction due to moving parts or by corrosion due to the environment [1]. One example would be diamond-like carbon (DLC) films used as a protective and low-friction barrier layer between the writing head and the magnetic media in hard drives. Regardless of the application though, it is important to have strongly adhered thin films.

There are several methods currently being used to measure adhesion of thin films, including indentation, scratch and pull off tests [2]. Buckling profile analysis and the superlayer indentation test were used to measure adhesion of a 20 nm DLC film. The superlayer indentation test is based on indentation-induced delamination [3], which was extended to ductile and better adhered films with the use of a stressed superlayer to provide additional driving force for delamination [4]. A 20 nm thick DLC film on top of 20 nm CoCrPtTi magnetic layer was coated with the sputter-deposited tungsten superlayer, 900 nm thick, with 1.9 GPa compressive residual stress. Adhesion values in a dry lab environment were measured by means of the superlayer indentation technique.

In previous experiments adhesion values were measured in a dry lab environment [5, 6], while this paper reports on the effects of fluids on interfacial adhesion. When performing indentation tests for adhesion measurements of DLC films it was discovered that the introduction of fluid causes delamination blisters to propagate further. This effect is similar to the mechanically-induced propagation of telephone cords caused by the microprobe manipulation [7, 8]. Different crack propagation rates of telephone cord blisters were observed when different fluids were introduced to the crack tip. This is known as environmentally-assisted fracture, with a ubiquitous example of vintage mirrors exhibiting faded and delamination spots. Other examples are delaminations and fading of telescope mirrors losing reflectivity [9], as well as environmentally assisted fracture in recently developed silica-based low-k dielectric thin films [10].

ADHESION ESTIMATES USING BUCKLING PROFILE ANALYSIS

Residual stress values were measured by the wafer curvature technique, applying Stoney's equation and also verified through calculations based on the buckling profiles. The following analysis is based on the Hutchinson's and Suo's developments for buckling-driven delamination of thin films [11].

Upon buckling, the stress in the film, σ_B, is estimated as:

$$\sigma_B = \frac{\pi^2}{12}\frac{E}{(1-\nu^2)}\left(\frac{h}{b}\right)^2 = 275\,MPa \qquad (1),$$

where h is the film thickness, b is the blister half-width, E and ν are Young's modulus and Poison's ratio, respectively. The buckling stress is acting in the vertical direction, perpendicular to the straight blisters shown in Figure 1 a.

Residual stress, σ_r, must be compressive in order to produce buckling delamination:

$$\sigma_r = \frac{3}{4}\sigma_B\left(\frac{\delta^2}{h^2}+1\right) = 1.9\,GPa \qquad (2),$$

where δ is the blister height (Figure 1 b). This estimate of 1.9 GPa for the residual compressive stress is in good agreement with the wafer curvature stress measurements performed on the non-delaminated samples.

Now we can estimate the film steady state interfacial toughness in the direction of blister propagation (Figure 1 b):

$$\Gamma_{SS} = \frac{(1-\nu^2)h\sigma_r^2}{2E}\left(1-\frac{\sigma_B}{\sigma_r}\right)^2 = 3.6\ J/m^2 \qquad (3).$$

Mode-dependent interfacial toughness in the buckling direction, perpendicular to blister propagation is:

$$\Gamma(\Psi) = \frac{(1-\nu^2)h}{2E}(\sigma_r-\sigma_B)(\sigma_r+3\sigma_B) = 6\ J/m^2 \qquad (4),$$

Figure 1 is a good example of both straight cord and telephone cord delaminations existing in one sample. The type of delamination a sample displays depends on the magnitude of the residual stress in the film and on the crack propagation rate [8]. The residual stress component acting in the direction of blister propagation drives the delamination. If crack propagation is fast enough, straight blisters are observed, although a conversion to telephone cord shape is possible after crack arrest. Profilometer measurements displayed in Figure 1 b give the height and width dimensions of the straight cord delaminations.

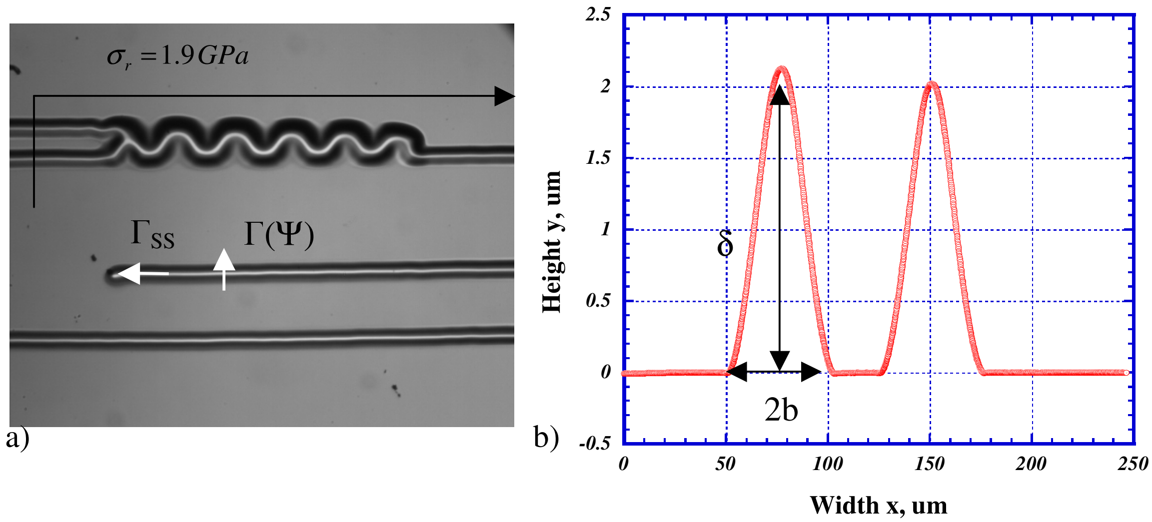

Figure 1. (a) Telephone cord delamination. (b) Blister heights profiles.

THE SUPERLAYER INDENTATION ADHESION MEASUREMENTS

In order to verify our estimates presented in the previous section, we conducted the superlayer indentation test to measure DLC film adhesion. As the indenter tip is pressed against the superlayer film stack, it supplies additional energy necessary for crack initiation and propagation. The blister radius is measured optically (Figure 2 a). The indentation volume is obtained from the plastic depth of the load-displacement curve

(Figure 2 b) and the tip geometry. Both the blister radius and the indentation volume are then used to calculate the strain energy release rate (measure of the practical work of adhesion) [4]. Calculations for adhesion measurements were made by following the solution developed by Marshall and Evans [3] that was further expanded by Kriese and Gerberich for multilayer films [4]. Here, both Berkovich and conical tip (1 µm tip radius) geometries were used and an indentation depth of at least 1.3 times the thickness of the tungsten superlayer film was reached. Figure 2 shows a typical delamination blister seen from making indents with a conical tip at 300 mN maximum load and a corresponding load-displacement curve. From Figure 2 b, the plastic indentation depth is obtained by using the power law fit of the top 65% of the unloading curve [12], and used to calculate the indentation volume, based on the tip geometry.

Several indents were performed on a sample, yielding 6.4±0.4 J/m^2 average adhesion values. These are in very good agreement with the buckling profiles mode mix adhesion estimates of 6 J/m^2. Based on the adhesion results obtained from the DLC films with and without magnetic layer underneath, we concluded that delamination occurred along the DLC/magnetic layer interface.

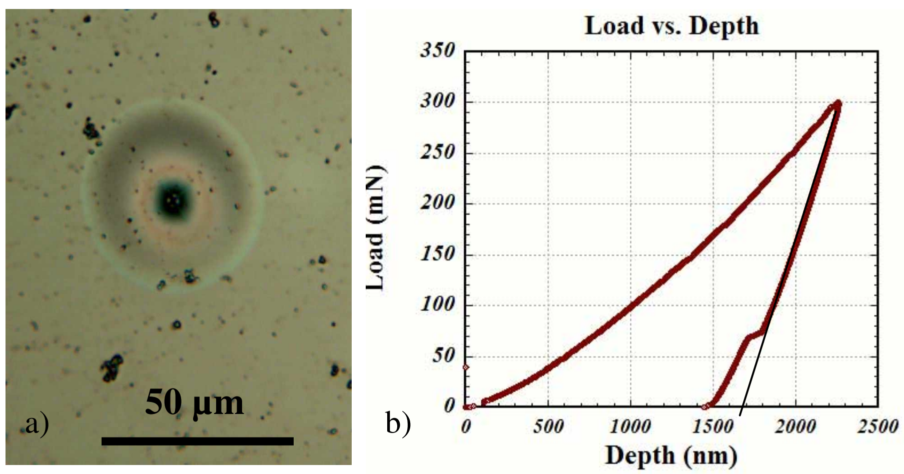

Figure 2. a) Delamination blister and b) corresponding load-displacement curve.

In a recent paper from Nix's group, which compared adhesion calculations for indentation tests and telephone cord delaminations, it was reported that the indentation results significantly overestimated "true" adhesion values [13]. We found that if indents were placed deep enough, realistic values of adhesion were calculated. Successful results were obtained when indentation depths versus film thickness ratios were greater then 1.3, which also relates to the delamination radius being at least 5 times the indentation radius. More indentation tests were performed with a 1 µm radius conical tip for confirmation of values measured using the Berkovich tip. Adhesion values obtained with the conical tip were somewhat lower. This small deviation happened because the dimensions used for calculating the Berkovich indentation volume did not factor in the radius of the tip.

ADDITIONAL OBSRVATIONS OF WATER-INDUCED SUB-CRITICAL BLISTER DEBONDING

After performing adhesion measurements in dry lab environment, we simulated environmentally assisted cracking. In order to observe telephone cord delamination propagation caused by the introduction of water, the samples were first mounted on a stage of an optical microscope by a small amount of adhesive. Since the samples were scribed from a 4-inch <100> Si wafer, small delamination blisters were randomly present at the edges of the samples. After an edge of the sample containing blisters was located under the microscope, distilled water was placed on the stage adjacent to the sample. Delamination was immediately seen when the water came into contact with the edge of the sample. In Figure 3, delamination propagation is seen for the blister coming from the top where distilled water was introduced, while no growth in the bottom "dry" blister is observed. Propagation of the telephone cord delaminations continued until either the water was taken away from the sample, or the blister reached the edge on the opposite side of the wafer.

This effect has been demonstrated with other fluids, including various oils. The propagation rates with the oils were much lower than that with distilled water. Isopropyl alcohol was also tried, but no additional delamination was observed. In addition, the fluid temperature was increased, which resulted in slower crack propagation rates. Contributions to accelerated crack growth include crack tip reactions, and a decrease in the newly formed fracture surface energies.

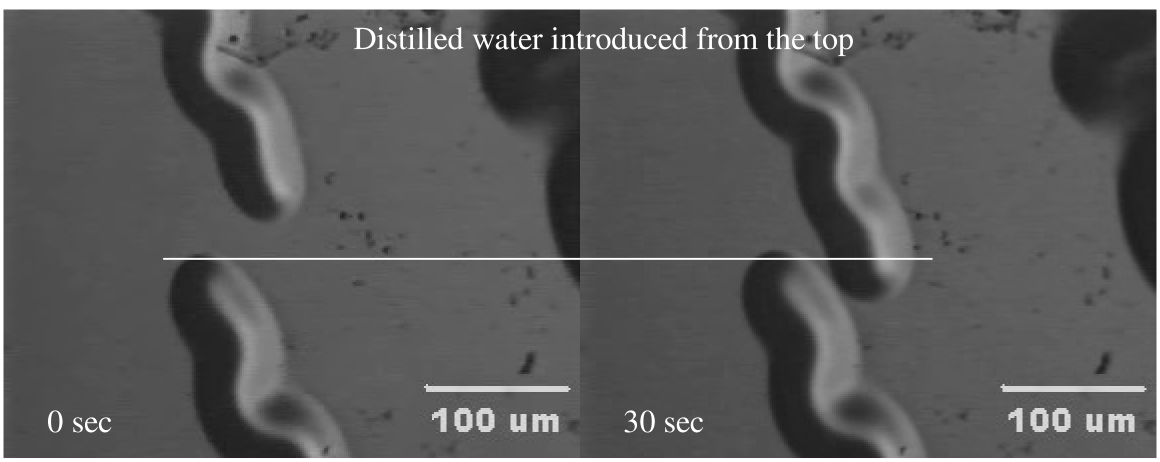

Figure 3. Water-induced blister growth on the top vs. no growth on the dry bottom.

CONCLUSIONS

Buckling delamination blisters are useful in studying environmental degradation and stress corrosion cracking of thin films. It has been shown that fluids can induce crack propagation at the thin films interfaces. The propagation can be attributed to the residual stress present in the thin films along with a combination of lower surface energies at the interface and added mechanical force to the delamination blisters due to the fluid pressure. It was shown that similar results for adhesion could be obtained by nanoindentation and by using telephone cord delamination profiles. Currently indentation tests are being performed to improve a technique for measuring adhesion values of thin films in fluid environments.

Additional possible applications may be in the microfluids field using these delaminations as microchannels for fluid transport [14]. Progress will need to be made on controlling the delamination propagation rate and size if the delamination channels are to be used in microfluids. It has been already shown through the use of lithographic technique that delamination can be confined to predetermined areas and sizes [15]. Detailed dynamics of real time telephone cord blister propagation can be observed online [16].

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