

Delamination and fracture of thin films

by Erik Klokholm

The fracture and delamination of thin films is a relatively common occurrence, and prevention of these mechanical failures is essential for the successful manufacture of thin-film devices. Internal elastic stresses are an inherent part of the thin-film deposition process, and are largely responsible for the mechanical failures of thin films. However, it is not the magnitude of the film stress S which governs film fracture or delamination, but the elastic energy U stored in the film. It is the intent of this presentation to show that the mechanical stability of the film and the substrate requires that U be less than a critical value U_c and that U_c is dependent upon the surface energy γ .

Introduction

The elastic stress present in thin films is an inherent part of the deposition process, and can be either tensile or compressive. The sign and the magnitude of the film stress are for the most part determined by the deposition parameters, i.e., substrate temperature, kind of substrate, deposition rate, and method of deposition. Stresses of about 10^9 – 10^{10} dynes/cm² are often observed [1, 2], and it has been commonly found that these stresses cause film fracture, delamination, and occasionally substrate fracture. However, the important criterion for the mechanical stability turns out not to be the magnitude of the stress, as commonly believed, but the elastic energy stored in the film.

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Elastic energy and surface energy

Whatever its origins, the elastic stress in thin films is only sustained by the mechanical constraint of the substrate. Separation of the film from the substrate relieves the film stress entirely; stress relief also occurs with film fracture and occasionally by fracture of the substrate surface due to the film stress. These stress relief modes come about because, as film thickness increases, the elastic energy stored in the film eventually becomes so large that the film catastrophically fails.

(It is also possible in some cases, for instance, in materials that melt at very low temperatures, such as Bi, Cd, Sb, to relieve film stress by plastic deformation; however, in the discussion that follows we will not concern ourselves with this stress-relief mode.)

The elastic energy U stored in a film of unit surface area and of thickness δ is expressed by

$$U = S^2 \frac{\delta(1-\nu)}{E}, \quad (1)$$

where E is Young's modulus and ν is Poisson's ratio [3]. The stress S in Equation (1) is assumed to be biaxial and isotropic in the plane of the film. Often, but not always, S is constant and independent of δ , so that U increases linearly with δ . Figure 1 shows a section of film of thickness δ and of unit surface area, i.e., unit length and width. The energy U is effectively the energy per unit area, as shown by the shaded section at the right of the figure. This presentation will show that the mechanical stability of the film and substrate requires that U be less than a critical value U_c and that U_c is determined by the surface energy γ . The surface energy γ for film fracture is illustrated in Figure 1, where the two new surfaces formed by the fracture contribute an increase of 2γ (ergs/cm²) to the total surface energy.

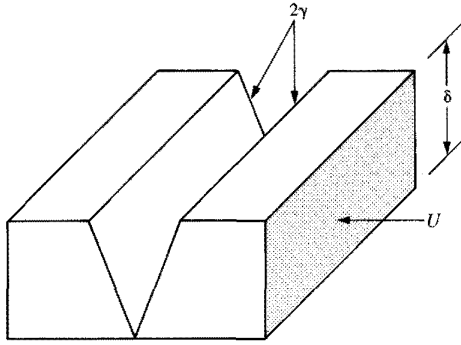


Figure 1

A section of film of thickness δ and of unit surface area, i.e., of unit length and width. The shaded area at the right of the figure indicates the elastic energy per unit area U . A crack is shown which contributes 2γ to the total surface energy via the formation of two new surfaces.

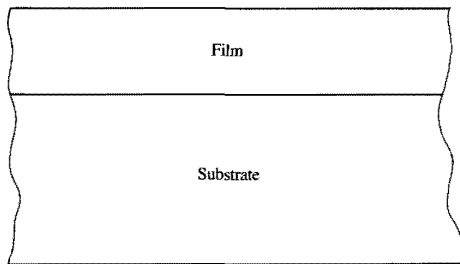


Figure 2

A perfect film-substrate interface having maximum film-substrate adherence.

For film-substrate delamination the definition for the surface energy requires modification. Consider **Figure 2**, where the film and the substrate form a contiguous and perfect joint at the interface; if the film delaminates from the substrate (Figure 6, shown later), then the surface energy gained is

$$\gamma_d = \gamma_s + \gamma_f, \quad (2)$$

where the subscripts refer to delamination, substrate, and film, respectively, and γ_d is the increase in surface energy upon delamination.

For film-substrate combinations where the adhesion is not perfect, as shown schematically in **Figure 3**, γ_d requires further definition. In this instance there is assumed to be a surface energy γ_i already expended, which is a measure of the degree of adhesion [4]. For instance, if $\gamma_i = 0$, then the adhesion is for the film-substrate of **Figure 1**; on the other hand, if $\gamma_i = \gamma_s + \gamma_f$, then $\gamma_d = 0$, since in this case no "new" surface energy is formed because the film and substrate were never physically joined. So, for imperfect adhesion,

$$\gamma_d = \gamma_s + \gamma_f - \gamma_i. \quad (3)$$

Values of γ (in ergs/cm²) range from ≈ 5000 for diamond and ≈ 1000 – 3000 for metals to ≈ 500 for glasses (or glass-like materials) [5].

For the tensile fracture of thin films, the well-known Griffith fracture theory will be used [5]. A less well-known theory, that of Barenblatt, will be applied to the conditions for delamination [6]. In the derivations, ν is neglected since its omission will not significantly affect the results. Although the derivations are phenomenological, and imperfect in detail, they have the advantage of being simple in concept and application.

Film fracture

Figure 4 shows schematically a film fracture for a film firmly bonded to the substrate; the crack is essentially perpendicular to the film plane and does not penetrate, or cause film separation from, the substrate at the intersection of the crack and the substrate. Cracks of this kind are caused by tensile stress. The relation between the critical film fracture stress S_c normal to the crack plane and the Griffith crack length h [5] for the geometry of **Figure 1** is

$$S_c \approx \sqrt{\frac{2E\gamma}{h}}. \quad (4)$$

The probability of film fracture increases as S_c approaches and exceeds the value defined by the parameters on the right side. By squaring both sides and rearranging terms, Equation (4) becomes

$$\frac{S_c^2 h}{E} \approx 2\gamma. \quad (5)$$

The critical energy U_c for fracture is therefore

$$U_c = \frac{S_c^2 h}{E} \approx 2\gamma. \quad (5a)$$

For the crack shown in **Figure 1**, it is intuitively plausible that $\delta = h$, and substituting δ for h in Equation (5a) yields the following relation:

Table 1 δ_c for two values of γ and S .

γ	δ_c at 10^9 (μm)	δ_c at 10^{10} (μm)
500	10	0.1
2000	40	0.4

$$U = \frac{S^2 \delta}{E} \geq U_c. \quad (6)$$

Equation (6) demonstrates the supposition that the criterion for film fracture is $U > U_c$, and U_c is governed by the magnitude of the surface energy γ while U is dependent upon the product δS^2 . There is, then, for U , when S is constant and independent of δ , a critical film thickness δ_c at which U exceeds U_c . We therefore define δ_c by the condition $U \approx U_c$, and δ_c is then given by the following equation:

$$\delta_c \approx \frac{2E\gamma}{S^2}. \quad (6a)$$

Film fracture will occur when $\delta \geq \delta_c$.

On the other hand for constant δ there is a critical value of film stress at which film fracture will take place as S approaches and exceeds S_c ; namely, Equation (4). In practice S tends to be independent of δ [2], and therefore δ is generally the critical variable for film fracture. We can calculate the relative magnitudes of δ_c by using Equation (6a) with two typical values of S — 10^9 and 10^{10} dynes/cm² (which cover the range of observed film stresses) and for $\gamma = 500$ and 2000 ergs/cm². We further assume that $E \approx 10^{12}$ dynes/cm². The results of the calculations are shown in Table 1. These results illustrate that, at constant S , δ_c is directly proportional to γ . For the typical values of γ used, δ_c varies from 10 to 40 μm for 10^9 d/cm², but for 10^{10} d/cm² the δ_c are 1/100 of the values for the smaller stress. This is due to the dependence of δ_c upon S^2 . If we assume that $\delta_c \approx 1 \mu\text{m}$, then S_c is $\approx 3 \cdot 10^9$ and $6 \cdot 10^9$ for $\gamma = 500$ and 2000 ergs/cm², respectively.

The values assumed for E , γ , and S are typical of many thin-film materials, so the data of Table 1 are fair approximations for the limits of mechanical stability of various films and substrates [7].

The photographs of Figure 5 illustrate the critical dependence of the film fracture criterion upon δ [8]. The upper photograph is a top view and the lower an oblique view. These permalloy films were deposited in the same pump-down by an electron gun evaporation source through a shuttered mask. Note the severe film fractures from column a to column d, and that even in column e there are indications of film cracking. The adhesion of these films to the substrate is very strong and, after film fracture, substrate fracture also occurred underneath the films in columns a, b,

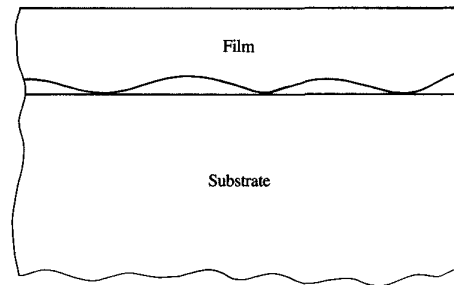


Figure 3

Imperfect film-substrate interface with imperfect film-substrate adhesion; adhesion exists only in contact areas.

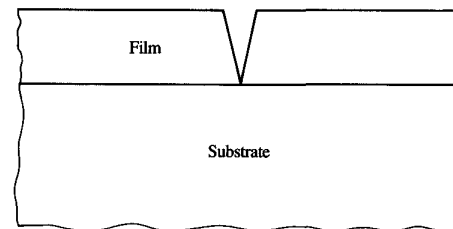


Figure 4

A "Griffith" film crack. The crack does not penetrate the substrate and is essentially normal to the film-substrate interface.

c, and d. The film thicknesses are as follows: column a, $\delta = 1.2 \mu\text{m}$; column b, $\delta = 1 \mu\text{m}$; column c, $\delta = 0.8 \mu\text{m}$; column d, $\delta = 0.6 \mu\text{m}$; column e, $\delta = 0.4 \mu\text{m}$; and column f, $\delta = 0.2 \mu\text{m}$. The tensile stress in all of these films is $\approx 5 \cdot 10^9$ d/cm², and from Figure 5 the critical δ appears to be at column e, where $\delta = 0.4 \mu\text{m}$. Substituting these in Equation (6a) indicates that $\gamma \approx 1000$ ergs/cm², which is in fair agreement with the preceding discussion.

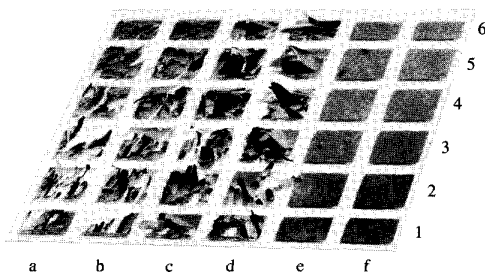
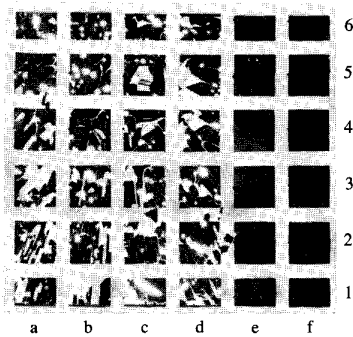


Figure 5

Fracture and delamination of permalloy films: the upper photograph is a top view; the lower, an oblique view. The film thicknesses (in μm) are 1.2 in column a, 1 in column b, 0.8 in column c, 0.6 in column d, 0.4 in column e, and 0.2 in column f. (Photographs courtesy of K. Y. Ahn, IBM Thomas J. Watson Research Center.)

Film delamination

The separation of the film from the substrate, as illustrated in **Figure 6**, is also a fracture phenomenon; however, the Barenblatt [6] rather than the Griffith fracture model is more appropriate. In the Griffith model the crack tip has a small but finite radius of curvature, while in the Barenblatt picture the crack has the shape of a cusp, and is very much like that of the delamination “crack” in **Figure 6**. The basic parameter in the Barenblatt model is a modulus of cohesion K and is defined by the energy

$$U_K = \frac{K^2(1 - \nu)}{E\pi} \quad (7)$$

required to initiate separation of the two surfaces by

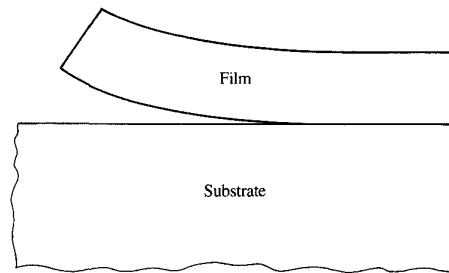


Figure 6

Delamination of film and substrate; a “Barenblatt” crack.

delamination crack formation. The constant K [6] is approximately defined by

$$K^2 \approx \frac{\pi E \gamma}{(1 - \nu^2)} \quad (8)$$

(In the following ν will again be neglected.)

Substituting for K in Equation (7), then,

$$U_K = \gamma \quad (9)$$

For the delamination model, substitute γ_d for γ in Equation (9), and then the critical energy for delamination is defined by

$$U_{cd} = \gamma_d \quad (10)$$

Equation 10 is the delamination analog of Equation (5a) for fracture. As the film elastic energy U approaches and exceeds U_{cd} , the probability of film delamination becomes increasingly greater; hence, the criterion for delamination is

$$U = \frac{S^2 \delta}{E} \geq U_{cd} \quad (11)$$

The critical thickness for delamination is then

$$\delta_{cd} \approx \frac{\gamma_d E}{S^2} \quad (11a)$$

and similarly the critical delamination stress is

$$S_{cd} \approx \sqrt{\frac{E \gamma_d}{\delta}} \quad (11b)$$

Equations (11), (11a), and (11b) are of the same form as Equations (6), (6a), and (6b) for fracture; however,

Equations (11), (11a), and (11b) contain the important variable γ_i . As mentioned previously, γ_i depends upon the degree of adhesion of the film to the substrate, and is 0 for perfect adhesion (cf. Figure 2), but is a maximum $\approx \gamma_f + \gamma_s$ when adhesion of the film to the substrate is very poor. Moreover, γ_i is not necessarily a material constant, but is largely dependent upon the physical and chemical nature of the substrate, namely, substrate surface cleanliness, smoothness, ambient deposition conditions, etc.

Discussion

From Equation (6) and (11) some general inferences with regard to film fracture and delamination can be drawn. If $U \gtrsim (U_c = U_{cd})$, then film fracture and delamination could occur simultaneously, as in Figure 7. For $U_c > U_{cd}$, delamination will probably occur before fracture, and if $U_c < U_{cd}$, the films will fracture before delamination. These considerations depend upon the relative values of γ and γ_d . For a given film material γ is essentially a known constant, but γ_d depends upon the degree of adhesion as determined by γ_i in Equation (3). For films that adhere strongly, $\gamma_d \approx 2\gamma$ probably satisfies the conditions for simultaneous—or nearly simultaneous—fracture and delamination. There is some evidence for this in Figure 5. Films that adhere weakly (for instance, Al, Cu, or Au on glass or silicon substrates) delaminate long before film fracture occurs. Metallic films for the most part delaminate before fracture, while dielectric films, glass, quartz, etc., tend to fracture before delamination.

For films in compression, a common mechanical instability is “blistering,” as illustrated in Figure 8 [5]. The blisters are often circular and of uniform size and distribution. Occasionally, film fracture occurs at the periphery of the blister. The criterion for blistering is described by Equation (11). For distributed blistering, γ_d must be smaller inside the blister areas than outside, therefore $U > U_{cd}$, but outside of the blisters, $U < U_{cd}$. The lack of adhesion in the interior of the blister can be caused by the presence of a foreign substance, substrate imperfections, etc.

If γ_d values are equal to 2γ of Table 1, then similar δ_c would be obtained for the same range of S . Hence, the same conclusions as drawn for fracture can be applied to delamination.

The application of the simpler aspects of fracture theory shows that the criterion for film-substrate mechanical stability is governed by γ , γ_d , S , and δ , and that the equations which describe the criteria for fracture and delamination have the same form. The surface energy γ of film and substrate materials is approximately known, but γ_d may not be known *a priori* since it is dependent upon γ_i —an unknown quantity. There is also a dependence of U_c upon E , but the variation of E among common materials is only from about 0.5 to $2 \cdot 10^{12}$ d/cm², and a value of

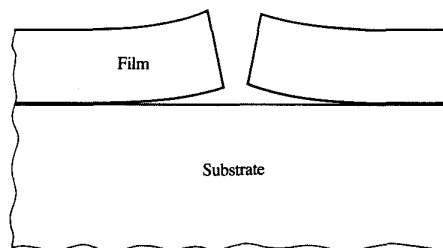


Figure 7

Simultaneous fracture and delamination.

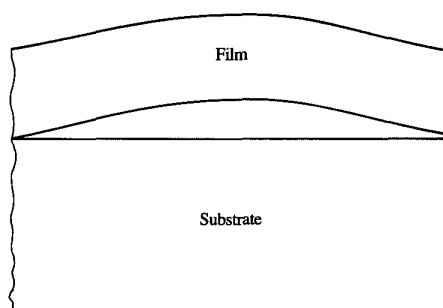


Figure 8

Blister formation by compressive film stress.

$E \approx 10^{12}$ is a reasonable approximation for a wide range of materials. The equations which describe the criteria for fracture and delamination can be combined into a single equation as follows:

$$\frac{\delta_c}{2\gamma} \approx \frac{1}{S^2} \cdot 10^{-6}, \quad (12)$$

where S is in units of 10^9 dynes/cm². By substituting for S values of $1/3$, $1/2$, 1, 2, 4, 7, 10, Equation (12) can be plotted on a log-log scale, as shown in Figure 9. The result is a straight line which covers the common range of S in thin films as well for γ , and is a universal curve describing the

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