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Nanomechanics of Hard Films on Compliant Substrates

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1. Introduction

Development of flexible thin film systems for biomedical, homeland security and environmental sensing applications has increased dramatically in recent years [1,2,3,4]. These systems typically combine traditional semiconductor technology with new flexible substrates, allowing for both the high electron mobility of semiconductors and the flexibility of polymers. The devices have the ability to be easily integrated into components and show promise for advanced design concepts, ranging from innovative microelectronics to MEMS and NEMS devices. These devices often contain layers of thin polymer, ceramic and metallic films where differing properties can lead to large residual stresses [5]. As long as the films remain substrate-bonded, they may deform far beyond their freestanding counterpart. Once debonded, substrate constraint disappears leading to film failure where compressive stresses can lead to wrinkling, delamination, and buckling [6,7,8] while tensile stresses can lead to film fracture and decohesion [9,10,11]. In all cases, performance depends on film adhesion. Experimentally it is difficult to measure adhesion. It is often studied using tape [12], pull off [13,14,15], and peel tests [16,17]. More recent techniques for measuring adhesion include scratch testing [18,19,20,21], four point bending [22,23,24], indentation [25,26,27], spontaneous blisters [28,29] and stressed overlayers [7,26,30,31,32,33]. Nevertheless, sample design and test techniques must be tailored for each system.

There is a large body of elastic thin film fracture and elastic contact mechanics solutions for elastic films on rigid substrates in the published literature [5,7,34,35,36]. More recent work has extended these solutions to films on compliant substrates and show that increasing compliance markedly changes fracture energies compared with rigid elastic solution results [37,38]. However, the introduction of inelastic substrate response significantly complicates the problem [10,39,40]. As a result, our understanding of the the critical relationship between adhesion, properties, and fracture for hard films on compliant substrates is limited.

To address this issue, we integrated nanomechanical testing and mechanics-based modeling in a program to define the critical relationship between deformation and fracture of nanoscale films on compliant substrates. The approach involved designing model film systems and employing nano-scale experimental characterization techniques to isolate effects of compliance, viscoelasticity, and plasticity on deformation and fracture of thin hard films on substrates that spanned more than two orders of compliance magnitude exhibit different interface structures, have different adhesion strengths, and function differently under stress.

The results of this work are described in six chapters. Chapter 1 provides the motivation for this work. Chapter 2 presents experimental results covering film system design, sample preparation, indentation response, and fracture including discussion on the effects of substrate compliance on fracture energies and buckle formation from existing models. Chapter 3 describes the use of analytical and finite element simulations to define the role of substrate compliance and film geometry on the indentation response of thin hard films

on compliant substrates. Chapter 4 describes the development and application of cohesive zone model based finite element simulations to determine how substrate compliance affects debond growth. Chapter 5 describes the use of molecular dynamics simulations to define the effects of substrate compliance on interfacial fracture of thin hard tungsten films on silicon substrates. Chapter 6 describes the Workshops sponsored through this program to advance understanding of material and system behavior.

2. Deformation and Fracture in Thin Hard Films on Compliant Substrates

N. R. Moody, D. P. Adams, M. S. Kennedy, M. J. Cordill, J. Yeager, D. F. Bahr

2.1 Introduction

The experimental approach involved designing model film systems and employing nano-scale experimental characterization techniques to isolate the effects of compliance, viscoelasticity, and plasticity on deformation and fracture of thin hard films on compliant substrates. The systems chosen included hard tungsten films deposited onto substrates spanning more than two orders of magnitude in compliance. These systems were shown to exhibit different interface structures, adhesion strengths, and residual stress states. This section first describes the film system design, sample preparation, and test techniques. It then presents results covering the effects of substrate compliance on mechanical properties, indentation response, and fracture. This includes discussion on the effects of substrate compliance on fracture energies and buckle formation from existing models.

2.2 Materials

The samples used in this study were fabricated by sputter depositing tungsten (W) onto 1cm x 1cm x 1mm thick polystyrene (PS), polymethylmethacrylate (PMMA), fused silica (FS), silicon (Si), and basal oriented sapphire (Al_2O_3) substrates with and without an aluminum oxide layer creating film systems spanning more than two orders of magnitude in substrate compliance. The aluminum oxide interlayer was added to alter interface composition and adhesion. Tungsten deposition was conducted with low and high background pressures to create films with high compressive and high tensile stress respectively. The samples were fabricated in two series. One series used commercial PS and PMMA substrates with UV absorbers. A second series used pure PS and PMMA substrates from [®]Goodfellow Corporation. Sample configurations are shown schematically in Figure 2.1. The matrix of test samples is given in Table 2.1. Properties of the films and substrates are given in Table 2.3.

In the first series of tests, substrates were cut from commercial polystyrene (PS), commercial polymethylmethacrylate (PMMA), fused silica (FS), silicon wafers with a 3nm thick native oxide (Si), and basal oriented sapphire (Al_2O_3). The FS, Si, and substrates were cleaned with an RF argon (Ar) plasma etch for 20 minutes to remove contaminants. The PS and PMMA substrates were prepared with an additional cleaning step by first removing residue with an IPA rinse. A 10 nm thick layer of aluminum oxide was sputter deposited onto the substrates of each substrate type using a D.C. magnetron sputtering unit with a base pressure of 8×10^{-8} Torr (10^{-5} Pa). The substrates were then divided into two groups with each group comprised of substrates with and without an

Figure 2.1. Schematic showing tungsten films on polystyrene (PS), polymehtylmethacrylate (PMMA), fused silica, silicon, and sapphire substrates (a) with and (b) without a 10nm thick aluminum oxide interlayer.

Table 2.1. Matrix of test samples

Series 1 Commercial PS and PMMA Substrates		Series 2 Pure PS and PMMA Substrates	
100 nmW 200 nm W		100 nmW 200 nm W 400 nm W 600 nm W	
Compression Tension		Compression Tension	
Al ₂ O ₃	10nmAl ₂ O ₃ /Al ₂ O ₃	Al ₂ O ₃	10nmAl ₂ O ₃ /Al ₂ O ₃
Si	10nmAl ₂ O ₃ /Si	Si	10nmAl ₂ O ₃ /Si
FS	10nmAl ₂ O ₃ /FS	FS	10nmAl ₂ O ₃ /FS
PS com	10nmAl ₂ O ₃ /PS	PS com	10nmAl ₂ O ₃ /PS
PMMA com	10nmAl ₂ O ₃ /PMMA	PMMA com	10nmAl ₂ O ₃ /PMMA

Table 2.2. Film and substrate properties

Film Substrate	Elastic Modulus (GPa)	Poissons Ratio
Sputtered Tungsten (W)	410	0.28
Sapphire (single crystal Al ₂ O ₃) c-axis	499	0.23
Silicon (single crystal Si) (110)	169	0.23
Fused Silica	72	0.17
Polymethylmethacrylate (PMMA)	3	0.35
Polystyrene (PS)	3	0.35

aluminum oxide coating. For the first group of samples, the chamber was back-filled with 0.4 Pa argon. A thin hard film of tungsten was then deposited at a rate of 0.3 nm/s at 1 kW using a 99.95% pure tungsten target to thicknesses of 100 nm and 200 nm. For a second group of samples, the chamber was back-filled with 2.0 Pa argon and tungsten deposited at a rate of 0.25 nm/s also to thicknesses of 100 nm and 200 nm. Silicon wafer monitors showed that deposition with a background Ar pressure of 0.4 Pa produced a residual compressive stress in the tungsten films of -1.7 GPa. Deposition with a background Ar pressure of 2.0 Pa produced a residual tensile stress in the tungsten films of 1.0 GPa. The film thickness was measured in separate controlled experiments immediately prior to deposition using a VLSI-calibrated DEKTAK profilometer. Film thicknesses on the polystyrene and polymethylmethacrylate substrates were confirmed using AFM and Scanning Auger Microscopy composition profiling. Scanning Auger Microscopy also confirmed the presence of the aluminum oxide interlayer (Figure 2.2). In a second series of tests, samples were fabricated by sputter depositing tungsten onto pure polystyrene, pure polymethylmethacrylate, fused silica (FS), silicon (Si), and basal oriented sapphire (Al_2O_3) substrates to thicknesses of 100 nm, 200 nm, 400 nm, and 600 nm following the same procedures. X-ray diffraction XRD showed that all films in tension had a highly oriented (200) structure. Films in compression had a two phase structure composed of simple cubic and body centered cubic phases (Figure 2.3). There was no difference in structure between films on commercial and pure PS and PMMA substrates.

Figure 2.2. Scanning Auger composition profiles for 100 nm thick W/PMMA film systems (a) with and (b) without an aluminum oxide interlayer.

2.3 Mechanical Property Measurements

Mechanical properties were measured using the continuous stiffness measurement option on a MTS Nano Instruments Nano Indenter[®] XP and a Nano Indenter[®] DCM with Berkovich diamond indenters tips. For tests on the XP, a frequency of 45 Hz, amplitude of 2 nm, nominal strain rate of 0.05, and maximum test depth of 1000 nm were used.

With DCM testing, a frequency of 75 Hz, an amplitude of 1 nm, a nominal strain rate of 0.05, and a maximum test depth of 500 nm were used.

Nanoindentation was also used to measure the load versus displacement response of the film systems using a conical diamond tip with a 2.0 μm tip radius on the Nano Indenter XP™ test system to facilitate finite element analysis of the film system response. For this study, a series of ten indentations were run at strain rates of 0.01, 0.05, 0.1, and 0.5 to a depth of 250 nm for the 100 nm thick films and 500 nm for the thicker films, held for 10 seconds, and then unloaded. Drift rates were measured on the film systems and on a fused silica sample before and after tests on the polystyrene and polymethylmethacrylate samples were on the order of 0.05 nm/s.

In films that buckled, a Digital Instruments multimode Atomic Force Microscope (AFM) was used to characterize blister heights and widths using both tapping and contact modes. The films were then removed and the substrates were imaged with tapping mode to characterize deformation on the substrate surfaces. A Veeco optical profilometer was used to measure the heights and widths of buckles on films where size exceeded AFM measurement limits.

Figure 2.3. X-ray diffraction XRD showed that (a) films in tension had a highly oriented (200) structure and (b) films in compression had a two phase structure composed of simple cubic and body centered cubic phases.

2.4 Results

2.4.1 Mechanical Properties

Results from the nanoindentation tests showed that the measured mechanical properties of the films (modulus and hardness) were strongly influenced by the substrate compliance. Near surface modulus and hardness (Figure 2.4) values for hard substrate systems (sapphire and silicon) matched bulk properties for tungsten ($E=410$ GPa, $H=12-15$ GPa). However, near surface properties measured in more compliant substrate systems (FS, PS, and PMMA) were markedly lower than those measured in rigid substrate systems (silicon and sapphire) due to substrate deformation. In addition, measured

Figure 2.4. (a) Modulus and (b) hardness for 100nm thick tungsten films on sapphire, silicon, fused silica, and commercial PS substrates.

Figure 2.5. (a) Modulus and (b) hardness for 100nm thick tungsten films on sapphire, silicon, fused silica, and commercial PS substrates with a 10nm thick aluminum oxide interlayer.

hardness exhibited a reversal in behavior at shallow contact depths. Although measured properties varied between substrates, true properties of the W films are the same for all these systems. The low values measured in the compliant substrate systems were caused in part by determining the contact area from indenter displacement rather than true contact area from direct observation. Modulus and hardness values for all substrates with an Al_2O_3 interlayer exhibited the same behavior as systems without an interlayer, but measured values were slightly lower (Figure 2.5). Work by Pharr and co-workers [41] showed that sputter deposited aluminum oxide formed an amorphous film with a modulus near 150 GPa and hardness between 5-6 GPa. These values are lower than W and significantly lower than sapphire or alumina.

2.4.2 Indentation Response

The load-displacement response superimposed for all rates for films on sapphire, silicon, and fused silica substrates. For films on commercial PS and PMMA substrates, the load-displacement responses superimposed for all rates tested to a depth of almost 50 nm for the 100 nm thick films and 100 nm for the 200 nm thick films. At greater indentation depths, the load-displacement responses increased with increasing strain rate. This is shown for the films on PS substrates in Figure 2.6. Indentation to greater depths in each film system led to a rate dependence in the response, with higher loads for higher strain rates, becoming essentially equal to the rate dependence of tests on polystyrene substrates at three times the film thickness. The rate dependence results from substrate yielding and the onset of film fracture under the hard films beginning at displacement depths near one-half the film thickness and full film fracture at displacement depths of three times the film thickness. There was no significant effect of stress state or an aluminum oxide interlayer on response for both commercial substrate systems and shown in Figure 2.7 for films on PS substrates.

Figure 2.6. The load-displacement response superimposed for all rates tested to a depth of almost (a) 50 nm for the 100 nm thick films and (b) 100 nm for the 200 nm thick films shown for the films on commercial PS substrates. At greater indentation depths, the load-displacement responses increased with increasing strain rate.

Figure 2.7. There was no significant effect of stress state or presence of an interlayer on indentation response of films on commercial polystyrene and polymethymethacrylate substrates as shown for films on commercial polystyrene substrates in (a) compression and (b) tension and for films (c) with and (d) without an aluminum oxide interlayer.

Figure 2.8. There was no significant effect of stress state or presence of an interlayer on indentation response of films on pure polystyrene and polymethymethacrylate substrates as shown for films in (a) compression and (b) tension and for films (c) with and (d) without an aluminum oxide interlayer on pure polystyrene substrates.

There was no significant effect of stress state or an aluminum oxide interlayer on response for both pure substrate systems and shown in Figure 2.8 for films on PS substrates. However, films on pure PS and PMMA substrates exhibited a rate effect not observed in films on commercial PS and PMMA substrates as shown for films on pure PS substrates in Figure 2.9.

Figure 2.9. Films on pure PS and PMMA substrates exhibited a rate effect not observed in films on commercial substrates as shown here for (a) 100 nm thick, (b) 200 nm thick, (c) 400 nm thick, and (d) 600 nm thick tungsten films on pure PS substrates

2.4.3 Film Failure

Deposition of films on commercial and pure PS and PMMA substrates with high tensile stresses led to channel cracking which increased in occurrence with increasing film thickness as shown in Figure 2.10 for commercial PS and PMMA substrates. Debonding was observed along channel cracks in the 400 nm and 600 nm thick films on PMMA. No debonding was observed in films on PS. Addition of an Al_2O_3 interlayer promoted cracking and changed crack morphology in W films on PS substrates (Figure 2.11). The same cracking patterns were observed on commercial PS and PMMA substrates. No cracking was observed in highly tensile stressed films on fused silica, silicon, or sapphire substrates.

Figure 2.10. All tensile stressed tungsten films deposited onto PS and PMMA substrates exhibited cracking that increased in density with increasing film thickness. (a) 100nmW/PS (b) 200nmW/PS (c) 400nmW/PS (d) 600nmW/PS (e) 100nmW/PMMA (f) 200nmW/PMMA (g) 400nmW/PMMA and (h) 600nmW/PMMA.

Figure 2.11. Addition of an Al_2O_3 interlayer promoted cracking and changed crack morphology in W films on PS substrates. It promoted adhesion on PMMA substrates. (a) 400nmW/PS (b) 400nmW/ Al_2O_3 /PS (c) 400nmW/ PMMA (d) 400nmW/ Al_2O_3 /PMMA.

Of most interest in this study, buckles formed spontaneously on commercial and pure PMMA substrates following film deposition. For 100 nm thick films on commercial PMMA substrates without the aluminum oxide interlayer, an extensive network of small telephone cord buckles formed following deposition, interspersed with regions of large telephone cord buckles (Figure 2.12a). For 100 nm thick films on substrates with an aluminum oxide interlayer, telephone cord buckles, similar in size to large buckles on the substrate with an interlayer, formed a uniform widely spaced pattern (Figure 2.12b). The buckles in 200 nm thick films on commercial PMMA substrates were larger than in 100 nm thick films. They also differed in morphology. On substrates without an interlayer the buckles formed an extensive ring-like network of uniform width buckles (Figure 2.12c). On substrates with an interlayer, they formed a widely spaced lattice-like pattern of small and large buckles (Figure 2.12d). A few large buckled regions continued to grow over several months until they spalled from the substrates. Buckles on films with an interlayer remained stable. Through-substrate optical observations revealed matching buckle patterns along the film substrate interface suggesting delamination occurred for large and small buckles with and without an interlayer.

Figure 2.12. An extensive network of small telephone cord blisters formed ring-like patterns in films deposited on commercial PMMA without aluminum oxide interlayers and more widely spaced lattice-like patterns when deposited on substrates with interlayers as shown for (a) 100nmW/PMMA, (b) 100nmW/Al₂O₃/PMMA, (c) 200nmW/PMMA, (d) 200nmW/Al₂O₃/PMMA.

The same behavior was observed for buckles in 100 nm and 200 nm thick films on pure PMMA substrates (Figures 2.13a-d). Differences in buckle morphology did appear in 400 nm and 600 nm thick films (Figures 2.13e-f). An extensive network of small telephone cord blisters forming a ring-like structure formed spontaneously following deposition in 400 nm and 600 nm thick films on pure PMMA substrates without an interlayer. Small isolated regions of larger buckles also formed in the 600 nm thick films. An extensive network of large blisters formed under the ring like networks suggesting that additional delamination occurred in the substrate parallel to the film substrate interface. Buckles in 400 nm and 600 nm thick films on pure PMMA substrate with an aluminum oxide interlayer formed more widely spaced lattice-like patterns interspersed with isolated larger telephone cord blisters similar in appearance to those that formed in the 100 nm and 200 nm thick film systems. However, there were numerous observations of centerline buckle cracking in the 400 nm and 600 nm thick films. In addition, there were many observations of delamination spreading from the small buckles and parallel buckle formation. Buckles in films on all pure substrates remained stable over time. As with films on commercial substrates, through-substrate optical observations revealed matching buckle patterns along the film substrate interface suggesting delamination occurred for large and small buckles with and without an interlayer.

Figure 2.13. An extensive network of small telephone cord blisters formed ring-like patterns in films on pure PMMA without interlayers and more widely spaced lattice-like patterns with interlayers as shown for (a) 100nmW/PMMA, (b) 100 nmW/ Al_2O_3 /PMMA, (c) 200nmW/PMMA, (d) 200nmW/ Al_2O_3 /PMMA, (e) 400nmW/PMMA, (f) 400nmW/ Al_2O_3 /PMMA, (g) 600nmW/PMMA, and (h) 600nmW/ Al_2O_3 /PMMA.

Figure 2.14. (a) Optical imaging of substrate surfaces under spalled regions in the 100 nm thick tungsten on commercial PMMA film systems revealed extensive deformation occurred along buckle edges, creating ‘river patterns’ on the substrate surface. These patterns reflect the extent and severity of deformation created by the crack tip plastic zones along (b) small and (c) large buckle edges.

Optical imaging of substrate surfaces under spalled regions in the 100 nm thick tungsten on commercial PMMA film systems revealed extensive deformation occurred along buckle edges, creating ‘river patterns’ on the substrate surface (Figure 2.14). These patterns result from an increase in interfacial toughness between W and PMMA where crack tip plastic zones formed in the substrate such that when the W film spalled, these regions deformed more extensively than surrounding regions. These regions reflect the extent and severity of deformation created by the crack tip plastic zones along buckle edges. Optical imaging and optical profilometry showed the same behavior for films on pure PMMA substrates. The differences in buckle morphology appear to result from the effects of local substrate response rather than effects of interface composition or film thickness. These patterns are not observed in rigid substrate systems and strongly suggest that localized substrate yielding and localized plasticity influences behavior in these systems.

Telephone cord buckles formed spontaneously on silicon wafers with a native oxide surface layer following deposition of 200 nm thick highly compression stressed films. The buckles exhibit a classic telephone cord buckle morphology as shown in Figure 2.15. No buckles formed on the silicon wafers with the aluminum oxide interlayer showing the effectiveness of using aluminum oxide in strengthening the interface. The buckle morphology and conditions for buckle formation match those observed in earlier studies and form a baseline for comparison.

Figure 2.15. Well-defined uniform width telephone cord buckles formed in the (a) 200nm thick tungsten films on silicon wafers with a native oxide. (b) No buckles formed on silicon wafers with a 10 nm thick aluminum oxide interlayer.

2.5 Fracture Energy

There is a large body of elastic film solutions describing delamination and buckling of films on rigid substrates, linking buckle morphology to interfacial fracture energy. [5,7,34,35,36]. The most widely used are the analyses of Hutchinson and Suo [5] for a one dimensional or straight-walled buckle and Moon and Hutchinson [42] for a pinned circular buckle. Cordill [33] and Kennedy [43,44] have shown that the differences in values from these methods are well within typical standard deviations.

The analysis of Hutchinson and Suo [5] for a straight-walled buckle lends itself to the measurements of buckle height and widths taken in this study. The analysis is based on the assumption that the film and substrate are elastic isotropic solids, the film is subject to a uniform, equi-biaxial compressive in-plane stress, and the film thickness is much less than the buckle width. The buckle is then modeled as a wide, clamped Euler column of length $2b$ as shown in Figure 2.16.

Figure 2.16. A cross-section schematic showing buckle height, δ , buckle width, $2b$, film thickness, h , and film stress, σ .

For a blister to form in the as-deposited film under these conditions, the compressive residual stress, σ_r , must exceed the stress for delamination, σ_b , as follows [5],

$$\sigma_b = \frac{\pi^2 E}{12(1-\nu^2)} \left(\frac{h}{b}\right)^2 \quad (2.1)$$

The conditions for buckling can also be expressed in terms of the critical half-width at which the film first deflects from the substrate [38,45],

$$b_* = \frac{\pi h}{2\sqrt{3}} \sqrt{\frac{E_f}{\sigma(1-\nu^2)}} \quad (2.2)$$

In these expressions, E is the elastic modulus of the film, ν is Poisson's ratio, and h is the film thickness.

The strain energy per unit area, G_o , stored in the film and available for fracture is a function of the residual stress as follows [5],

$$G_o = \frac{(1-\nu^2)h\sigma_r^2}{2E} \quad (2.3)$$

When the film buckles, the separation between the film and substrate loads the edge of the interfacial crack in tension. The tensile stresses drive interfacial crack advance with a release of strain energy given by [5],

$$G = \left[\frac{(1-\nu^2)h}{2E} \right] (\sigma_r - \sigma_b)(\sigma_r + 3\sigma_b) \quad (2.4)$$

This value is often designated $\Gamma(\psi)$ at fracture due to its mixed mode character.

The stresses at the crack tip of a buckled film are comprised of normal mode I and shear mode II components. For one-dimensional blisters, the relative contributions of mode I and mode II loads are given by the phase angle of loading [5],

$$\tan \psi = \frac{k_{II}}{k_I} = \frac{4\cos\omega + \sqrt{3}\xi\sin\omega}{(-4\sin\omega + \sqrt{3}\xi\cos\omega)} \quad (2.5)$$

In this equation, k_I and k_{II} are the mode I and mode II crack tip stress intensities, ξ equals δ/h , and ω is a dimensionless function of the Dundurs's parameters, α and β . These parameters describe the elastic mismatch between the film and the substrate as follow,

$$\alpha = \frac{(\bar{E}_f - \bar{E}_s)}{(\bar{E}_f + \bar{E}_s)} \quad (2.6a)$$

$$\beta = \frac{1}{2} \frac{\mu_f(1-2\nu_s) - \mu_s(1-2\nu_f)}{\mu_f(1-\nu_s) + \mu_s(1-\nu_f)} \quad (2.6b)$$

with $E_i = E_i / (1 - \nu_i^2)$ (2.6c)

and $\mu = E / 2(1 + \nu)$ (2.6d)

With no elastic mismatch between the films and substrates, ω reduces to 52.1° [5]. With significant differences in elastic mismatch between film and substrates, as in this study, ω can vary greatly.

Treating the fractures as inherently mode I failures [46], the mode I contributions to fracture can be estimated using the following empirical relationship between mixed mode and mode I fracture energies [5,35],

$$G_I = \frac{G(\psi)}{\{1 + \tan^2[(1-\lambda)\psi]\}} \quad (2.7)$$

where λ is an empirical material constant that adjusts the influence of the mode II contribution [5]. The values of λ range from 0 to 1 with 0 depending on only mode I component and 1 being ideally brittle. For most systems including the film systems in this study in this study, λ is approximately 0.3 [48].

Work by Cotterell and Chen [37] and Yu and Hutchinson [38] show that substrate compliance has a marked effect on fracture energies and phase angles of loading due to the release of elastic energy in regions of the film still attached to the substrate. The effect increases with increasing elastic mismatch, becoming significant with high elastic mismatch ($\alpha > 0.5$) and small buckle widths. Increasing substrate compliance also increases the mode I contribution thereby promoting failure. Yu and Hutchinson examined the effect of substrate compliance on strain energy release rate using two approaches: one based on stress intensity and the other on the total energy of the system as a function of the film stress, σ_f , modulus, E_f , and Poisson's ratio, ν_f . In theory the results should superimpose but differences occur due most likely to solution accuracy when the substrate is very compliant, the buckles are small, and the films stresses very high [38]. In this study, the results of Yu and Hutchinson [38] from both approaches are

used to analyze the role of substrate compliance on interfacial fracture of all films on commercial and pure PMMA substrates.

The critical buckling stresses for the W/PMMA and W/Al₂O₃/PMMA systems were found using the rigid substrate-elastic film solutions of Hutchinson and Suo [5] and the compliant substrate solutions of Yu and Hutchinson [37]. The residual stress, σ_r , of the W films, and the blister heights and widths measured with AFM, were used to calculate the energy release rate for plane strain ($\epsilon_{zz}=0$) buckles, assuming a rigid elastic case. (Table 2.3 and Table 2.4) Wafer curvature showed the residual stress in these films was -1.7 GPa. The energy release rate, G , and phase angle of loading, ψ , were then determined from Yu and Hutchinson's solutions for normalized strain energy release using stress intensity and total energy approaches for $\alpha=0.99$ and $\sigma_r/\bar{E}_f$ equal to 0.01%, 0.1%, and 1%., where α is the elastic mismatch coefficient and $\bar{E}_f = E_f/(1-\nu^2)$ [37]. For the W on PMMA systems, $\sigma_r/\bar{E}_f$ is near 0.4%. The corresponding energy release rates for blisters in our systems were found by interpolating the Yu and Hutchinson $\sigma_r/\bar{E}_f$ solutions between 0.1% and 1% to obtain values for 0.4%. The mode I release rates, Γ_I , were determined using the empirical relationships between mixed mode and mode I energies given by Hutchinson and Suo [5]. They were first determined assuming no elastic mismatch. They were then determined from the Yu and Hutchinson [37] analysis for the effects of substrate compliance on the phase angle of loading. The results are given in Table 2.3 for films on commercial PMMA substrates and in Table 2.4 for films on pure PMMA substrates. The fracture energies determined using these solutions more than doubled from rigid elastic values for large buckles in the W/PMMA and W/Al₂O₃/PMMA systems and increased almost 8-fold for small buckles in the W/PMMA system. There was also a dramatic change in the phase angle of loading from pure shear in the W/Si system to predominantly mode I loading in the W/PMMA system with small buckles.

2.6 Buckle morphology

The relationship between buckle deflection and film stress for the straight-sided buckling of a thin film on a rigid substrate is given explicitly by [5,47],

$$\frac{\delta}{h} = \sqrt{\frac{4}{3} \left(\frac{\sigma_r}{\sigma_c} - 1 \right)} \quad (2.8)$$

and provides an accurate description of the buckling of a thin film on a rigid substrate including telephone cord buckles [48,49,50,51].

Parry et al. [47] showed that substrate compliance markedly alters this relationship. Using non-linear finite element simulations, they found that compliance could reduce the effective critical stress by up to forty percent and increase deflections up to two-times

that for a rigid substrate, depending on elastic mismatch. They also found that the normalized deflection δ/h depends on the normalized stress σ_r/σ_c and that the fundamental relationship between the two parameters is preserved as follows

$$\frac{\delta}{h} = \gamma_\delta \sqrt{\frac{4}{3} \left(\frac{\sigma_r}{\gamma_\sigma \sigma_c} - 1 \right)} \quad (2.9)$$

The correction factor for the critical stress, γ_σ , and the correction factor for the deflection, γ_δ , vary with α and are given by Parry et al. [47] and Coupeau [52].

Although substrate compliance has little effect on the fracture energies for buckles formed on silicon substrates, it has a strong effect on buckle heights. This is shown in Figure 2.17 where normalized buckle heights are much greater than predicted from the rigid elastic plate solution but in good agreement with normalized heights predicted from eq. (2.9) for the W/SiO₂/Si system ($\alpha=0.7$). Using measured buckle heights, the rigid elastic solution for the blisters observed in the W/SiO₂/Si film system predicts a residual stress near -3.0 GPa. Correcting for substrate compliance in eq. (2.9) gives a value of -2.0 GPa in good agreement with the wafer curvature stress of -1.7 GPa.

Figure 2.17. Normalized buckle heights are greater than predicted from the rigid elastic plate solution ($\alpha=-1$) but in good agreement with normalized heights predicted from eq. (2.9) for the W/SiO₂/Si system ($\alpha=0.7$).

Substrate compliance dramatically alters the relationship between buckle heights and stress for W/PMMA and W/Al₂O₃/PMMA systems. Figure 2.18 shows it varies with film thickness, the presence of an Al₂O₃ interlayer, and with substrate composition. It also varies within each sample where large and small networks of buckles formed in the same

Figure 2.18. Substrate compliance markedly alters the relationship between buckle heights and stress for films on (a) commercial and (b) pure PMMA substrates.

Figure 2.19. Growth of small buckles is deformation limited while growth of large buckles is delamination driven. The two behaviors are shown in (a) commercial and (b) pure PMMA substrates.

sample. The small buckles are associated with relatively large plastic zones and intense local plasticity and the large buckles are associated with relatively small plastic zones. This results in one population of buckles where growth is deformation limited and in another is where growth is delamination dominated (Figure 2.19).

Superimposing Parry et al. [47] buckle morphology relationships show they roughly correspond to trends in observed data where effects are significantly more pronounced than predicted by rigid elastic systems (Figure 2.20). However, they do not account for the large variance in behavior even when considering the sensitivity of predictions as α approaches 1. Moreover, the small blisters fall completely outside predicted behaviors.

Figure 2.20. Superimposing buckle height and stress relationships from Parry et al. show they roughly correspond to trends in observed data where effects are significantly more pronounced than predicted by rigid elastic systems in (a) commercial and (b) pure PMMA substrate systems.

Height and width measurements rely on the assumption that AFM and optical profilometry measurements accurately reflect full buckle formation. Through-substrate observations indicate that fully defined buckles formed along the film-substrate interface in patterns matching large and small buckle networks. Indentation of the buckles in 100 nm thick films on commercial PMMA with and without an aluminum oxide interlayer further support these observations. The tests were conducted by Megan Cordill at the University of Minnesota on a Hysitron TriboIndenter fitted with a sharp conical tip ($R = 150$ nm) on small and large buckles with similar heights but markedly different widths. Fracture occurred during indentation of both buckles (Figure 2.21) with displacement excursions corresponding to buckle heights and the presence of large cavities of similar size under the buckles (Figure 2.22). These results provide evidence that buckles in the thin film systems formed by delamination and not wrinkling of the film and substrate.

AFM imaging clearly shows that regions of intense local plasticity occur along buckle walls. The deformation along small buckle walls is much larger and more intense than that along large blister walls. This deformation alters buckle morphology beyond the effects indicated by compliant elastic substrate models. As a consequence, interfacial fracture energies calculated using elastic buckle theory even when the effect of compliance is included may not accurately describe the energy required for telephone cord blister formation.

Figure 2.21. Fracture occurred during indentation of both (a) large and (b) small buckles with similar heights but markedly different widths in 100nm thick films on commercial PMMA

Figure 2.22. Displacement excursions during indentation of large and small buckles corresponds to respective buckle heights indicating the presence of large cavities under the large and small buckles.

2.7 Summary

In the experimental portion of this program we have studied how substrate compliance affects deformation and fracture, employing sputter deposited tungsten films in both tension and compression onto sapphire, silicon, fused quartz, polystyrene (PS) and polymethylmethacrylate (PMMA) substrates with and without 10 nm thick Al_2O_3 interlayers. These interlayers were used to alter composition and tailor adhesion between film and substrate. Buckles formed spontaneously in compressively stressed W/PMMA systems with and without interlayers and on compressively stressed W/Si with a native oxide. Buckles did not form on the W/Si system with an Al_2O_3 interlayer. In contrast, channel cracking occurred in tensile stressed W on PS with and without an interlayer but no buckle formation. Together, the results show a complex interaction between adhesion, fracture, and substrate compliance not fully described by current material models. While classic rigid elastic analysis ($\alpha = -1$) gave fracture energies near 0.1 J/m^2 for all blisters on PMMA, accounting for compliance increased fracture energies from 0.3 J/m^2 for large blisters to 0.8 J/m^2 for small blisters on PMMA ($\alpha = 0.98$). An even greater effect of substrate compliance was observed on buckle morphology. The surprising observation was the co-existence of large and small buckles on the same sample. Large blisters followed predicted trends in behavior. Small blisters fell well outside predicted behavior, indicating a fundamental difference in response to stress. Indentation of the buckles indicated that the same mechanism of delamination and growth governed buckle formation. However, optical imaging of PMMA substrate surfaces revealed extensive deformation occurred along buckle edges not observed in rigid substrate systems. AFM imaging clearly showed that these regions of deformation along small buckle walls were much larger and more intense than those along large blister walls. These regions reflect the extent and severity of deformation created by the crack tip plastic zones along buckle edges. This deformation alters buckle morphology and fracture energies beyond the effects suggested by compliant substrate models. As a result, the building of models that accurately describe and predict localized plasticity and substrate compliance effects became a primary focus of this study and a major contribution to the study of flexible substrate systems.

Table 2.3. Fracture energies for films on commercial PMMA substrates

Film System	Elastic			Elastic			Yu and Hutchinson ^a			Yu and Hutchinson ^b		
	G(ψ) (J/m ²)	$\psi=f(\omega=52.1^\circ)$	GI (J/m ²)	G(ψ) (J/m ²)	$\psi=f(\omega(\alpha,\beta))$	GI (J/m ²)	G(ψ) (J/m ²)	ψ	GI (J/m ²)	G(ψ)	ψ	GI
200W/SiO ₂ /Si	0.71 ± 0.006	-90	0.15 ± 0.001	0.71 ± 0.006	-90	0.15 ± 0.001	0.68 ± 0.007	-90	0.14 ± 0.001			
100W/Al ₂ O ₃ /PMMA	0.37 ± 0.006	-90	0.08 ± 0.001	0.37 ± 0.006	-62.5 ± 2.5	0.19 ± 0.014	0.87 ± 0.07	-62.5 ± 2.5	0.46 ± 0.07	0.842 ± 0.06	-62.5 ± 2.5	0.44 ± 0.057
100W/PMMA Large	0.36 ± 0.02	-90	0.075 ± 0.004	0.36 ± 0.02	-64 ± 8.3	0.19 ± 0.05	0.87 ± 0.24	-64 ± 8.3	0.46 ± 0.22	0.844 ± 0.21	-64 ± 8.3	0.45 ± 0.20
Small	0.4 ± 0.037	-90	0.082 ± 0.008	0.4 ± 0.037	-33.2 ± 3.5	0.33 ± 0.022	2.69 ± 0.27	-33.2 ± 3.5	2.28 ± 0.31	2.04 ± 0.126	-33.2 ± 3.5	1.72 ± 0.166
200W/Al ₂ O ₃ /PMMA	0.719 ± 0.016	-90	0.148 ± 0.003	0.719 ± 0.016	-64.8 ± 3.7	0.356 ± 0.040	1.62 ± 0.18	-64.8 ± 3.7	0.81 ± 0.16	1.582 ± 0.153	-64.8 ± 3.7	0.789 ± 0.143
200W/PMMA	0.801 ± 0.039	-85.1 ± 5.57	0.21 ± 0.060	0.801 ± 0.039	-49.8 ± 6.5	0.54 ± 0.085	2.87 ± 0.76	-49.8 ± 6.5	1.98 ± 0.74	2.6 ± 0.557	-49.8 ± 6.5	1.782 ± 0.571

^a stress intensity approach

^b energy approach

Table 2.4. Fracture energies for films on pure PMMA substrates

Film System	Elastic			Elastic			Yu and Hutchinson ^a			Yu and Hutchinson ^b		
	G(ψ) (J/m ²)	$\psi=f(\omega=52.1^\circ)$	GI (J/m ²)	G(ψ) (J/m ²)	$\psi=f(\omega(\alpha,\beta))$	GI (J/m ²)	G(ψ) (J/m ²)	ψ	GI (J/m ²)	G(ψ)	ψ	GI
100W/Al ₂ O ₃ /PMMA large	0.359	-90	0.074	0.359	-65	0.173	0.806	-65	0.398	0.788	-65	0.389
	±0.006		±0.001	±0.006	±2.5	±0.014	±0.065	±2.5	±0.058	±0.054	±2.5	±0.053
100W/PMMA small large	0.406	-90	0.098	0.406	-49.2	0.269	1.417	-49.2	0.966	1.357	-49.2	0.925
	±0.009		±0.002	±0.009	±2.6	±0.026	±0.151	±2.6	±0.147	±0.128	±02.6	±0.128
	0.342	-90	0.071	0.342	-74.6	0.12	0.615	-74.6	0.233	0.74	-74.6	0.279
	±0.004		±0.001	±0.004	±2.8	±0.007	±0.042	±2.8	±0.036	±0.032	±2.8	±0.035
200W/Al ₂ O ₃ /PMMA small large	0.83	-90	0.171	0.83	-46.4	0.591	3.174	-46.4	2.266	2.869	-46.4	2.047
	±0.019		±0.004	±0.019	±2.79	±0.039	±0.334	±2.79	±0.326	±0.256	±2.79	±0.263
	0.708	-90	0.146	0.708	-68	0.323	1.483	-68	0.685	1.464	-68	0.675
	±0.018		±0.004	±0.018	±4.37	±0.046	±0.200	±4.37	±0.173	±0.171	±4.37	±0.160
200W/PMMA small large	0.814	-90	0.323	0.814	-39	0.638	4.435	-39	3.569	3.565	-39	2.848
	±0.045		±0.086	±0.045	±7.44	±0.060	±1.199	±7.44	±1.242	±0.677	±7.44	±0.774
	0.728	-90	0.15	0.728	-64	0.369	1.722	-64	0.9	1.66	-64	0.864
	±0.033		±0.007	±0.033	±7.54	±0.083	±0.391	±7.54	±0.348	±0.335	±0.335	±0.315
400W/PMMA small	1.545	-90	0.319	1.545	-34.269	1.277	10.572	-30.019	9.223	8.287	-34.269	7.219
	±0.266		±0.055	±0.266	±6.028	±0.168	±1.873	±8.612	±2.191	±0.964	±6.028	±1.385
400W/Al ₂ O ₃ /PMMA small large	1.648	-90	1.224	1.648	-41.61	1.252	8.138	-41.6	6.377	6.703	-41.6	5.215
	0.115		±0.238	±1.115	±8.756	±0.191	±2.378	±8.756	±2.41	±1.502	±8.756	±1.636
	1.372	-90	0.283	1.372	-74.1	0.511	2.483	-74.1	0.949	2.47	-74.1	0.945
	±0.011		±0.002	±0.011	±1.979	±0.037	±0.126	±1.979	±0.109	±0.141	±1.979	±0.115

^a stress intensity approach^b energy approach

3. Modeling the Indentation Response of Stiff Films on Compliant Substrates

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3.1 Introduction

The modeling study presented here aims to quantify the effects of compliance, film thickness, and displacement on the elastic indentation response of model compliant substrate systems using a computational and theoretical approach. Several analytical models have been developed for the indentation response of compliant substrate systems. Most are based on classical plate bending solutions that treat the stiff film as a plate bending on an elastic foundation [53] or plastic foundation [54-56]. In addition, a number of finite element studies have been applied to examine the effects of substrate plasticity on the indentation force-displacement response [57-59]. Most relevant to this study is the elastic plate bending model of Ramsey et. al. [53]. The model treats the system as an infinite circular plate perfectly bonded to an elastic half space. The indenter is modeled as a concentrated force acting at the center of the plate. Assuming small displacements, the model predicts a linear indentation force-displacement relationship, $P = K\delta$. The effective stiffness K scales linearly with the film thickness and depends on the film and substrate moduli. For larger displacements, Ramsey et. al. [53] extended the plate bending model to incorporate nonlinear effects from large membrane stresses. Interestingly, Vanimisetti and Narasimhan [60] also obtained similar results in their finite element study of hard films on soft substrate. Though the substrate exhibited significant plasticity, they obtained a near linear force-displacements relationship for displacements in the range of 0.1-0.5 the film thickness. Moreover, the indentation force scaled almost linearly with the film thickness in this bending dominated region.

The finite element modeling study examined the ability of the elastic plate bending models of Ramsey et. al. [53] to reproduce accurately the results of nanoindentation experiments of the tungsten film systems, described in Sec. 2.2.4, and finite element simulations of model elastic compliant substrate systems. The model systems were chosen to explore a wide range of film thicknesses and ratios of film and substrate moduli. The finite element simulations also investigated the influence of the indenter radius. The results of the study showed that the linear plate bending model provided accurate predictions of the indentation force-displacement relationships for displacements smaller than the film thickness and ratios of the film to substrate moduli greater than 100. The indenter radius had a small but noticeable effect on the indentation force-displacement response. A smaller indenter radius produced better agreement between the finite element and plate bending models for displacements less than the film thickness. For larger displacements, the indentation response exhibited a scaling relationship that reminiscent of the Hertzian contact problem.

3.2 Methods

3.2.1 Analytical Plate Bending Model

Figure 3.1: Schematic of the plate bending model. The film and substrate are modeled as an infinite plate on an elastic half space

An analytical model has been developed for the indentation response of compliant substrate systems by Ramsey et al. [53] using classical plate theory. The compliant substrate system is modeled as an infinite axisymmetric plate perfectly bonded to an elastic half space. The indenter is modeled as a concentrated force applied at the center of the plate as illustrated in Fig. 3.1. These idealizations reduce the complex indentation problem to a classical problem of a plate bending on an elastic foundation. The analytical solution for the force-displacement relationship is developed in many textbooks on plate and shell theory, and the reader is directed to the classic work of Timoshenko and Woinowsky-Krieger [61] for a detailed treatment. The solution for the maximum displacement of the plate can be written as [61 Ch.8],

$$P = K\delta, \quad K = \frac{27^{1/2} E_f^{1/3} E_s^{2/3} h_f}{48^{1/3} (1 - \nu_f^2)^{1/3} (1 - \nu_s^2)^{2/3}} \quad (3.1)$$

where K is the effective stiffness of the system. When applied to the indentation problem, P and δ are interpreted as the indentation force and displacement. The plate bending model predicts that the indentation force is related linearly to the displacement by an effective stiffness that scales linearly with the film thickness and depends more so on the substrate than the film moduli.

Ramsey et al. [53] developed a nonlinear extension of the plate bending model that incorporated the effects of large in-plane deformations for large indentation displacement. Though they did not derive an analytical solution for the nonlinear model, they showed that the nonlinear force-displacement relationship submits to the same dimensionless analysis as the linear model and can be written as,

$$\frac{P}{Kh_f} = g\left(\frac{\delta}{h_f}\right) \quad (3.2)$$

where $g(\delta/h_f)$ is a nonlinear function that must be determined numerically. The accuracy of the linear relationship in eq. (3.1) and validity of the non-dimensional relationship in eq. (3.2) are examined in the following sections using finite element analysis.

3.2.2 Finite Element Models

Figure 3.2: Finite element geometry of indentation of a thin stiff film on a compliant substrate.

Figure 3.2 shows a schematic of the axisymmetric finite element (FE) model for the spherical indentation of a compliant substrate system. The indenter was modeled as a hemisphere with radii ranging from $R_i = 0.25\text{-}2.0\text{mm}$. The interface between the indenter and film was modeled using frictionless contact elements. The vertical displacement u_z of the bottom surface of the substrate and the radial displacement u_r of the central axis of the entire system were fixed to eliminate rigid body motions. Otherwise, the film and substrate were free to deform, such that the remaining surfaces remained traction free. Loading was provided by uniformly displacing the top surface of the indenter into the film. The finite element study considered film thicknesses ranging from $h_f = 100\text{-}800\text{ nm}$ and substrate dimensions ranging from $50\text{-}400\mu\text{m}$. The dimensions of the substrate corresponded to $500\text{-}1000h_f$ in terms of film thickness and $25\text{-}400R_i$ in terms of indenter radii. Mesarovic and Fleck [62] found that the displacements on the outer boundaries of the substrate became negligibly small for substrate dimension of $10R_i$ for indentation depths up to $0.2R_i$. The maximum applied displacement in this study was $10h_f$, and corresponded to substantially larger displacements. However, the in-plane displacement u_r at the outer radius of the film substrate for all the cases examined here were at most 1.5% percent of the applied displacement, and thus did not significantly influence the indentation response. The element size was chosen such that at least ten elements spanned the film thickness. The interface between the film and substrate was assumed to be perfectly bonded.

To accommodate for potentially large deformation from large indentation displacements, an updated Lagrangian formulation was used for the finite elements method, and a compressible hyperelastic Neo-Hookean model was used for the constitutive behavior of the film and substrate materials. The Young's modulus and Poisson's ratio for the

tungsten film and various substrates used in the experiments are listed in Table 3.1. The finite element models employed a wider range of substrate moduli $E_s = 0.31\text{-}101$ GPa and film moduli $E_f = 40\text{-}4000$ GPa. To approximate a rigid indenter, the Young's modulus and Poisson's ratio of the indenter was set to, $E_i = 1000E_f$ and $\nu_i = \nu_f$.

Table 3.1: Elastic Properties

Material	E (GPa)	ν
tungsten (W)	400	0.28
fused silica (FS)	72	0.18
polystyrene (PS)	3.1	0.35

3.3 Results and Discussions

Figure 3.3: The force-displacement curves comparing the results of nanoindentation experiments (Exp) and FE simulations (Sim) for 100 nm and 200 nm W/PS systems.

The indentation force-displacement curves calculated from finite element simulations of the 100 nm and 200 nm W/PS systems are plotted in Figure 3.3, along with the nanoindentation data for comparison. Unless otherwise stated, an $R_i = 1.0\mu\text{m}$ indenter radius was used in the FE simulations. A linear fit of the region $\delta < 0.5h_f$ was used to calculate the effective stiffness of the FE indentation force-displacement curves. The effective stiffness of the 100 nm and 200 nm W/PS systems were also calculated using eq. (3.1) of the linear plate bending model. The results for the FE model, linear plate bending model, and nanoindentation experiments are presented in Table 3.2. All three showed good quantitative agreement. The largest difference was 6.0% between the plate bending and finite element models for the 100 nm W/PS system.

Table 3.2: Effective Indentation Stiffness (kN/m) of the W/PS systems.

	$h_f = 100 \text{ nm}$	$h_f = 200 \text{ nm}$
Experiment	2.52	5.09
Finite Element Model	2.66	5.12
Linear Plate Bending Model	2.51	5.02

Figure 3.4: (a) The force-displacement curve from finite element simulation of W/PS system with 100 nm film. (b) The effective stiffness K for W/PS systems with different film thicknesses comparing the results from nanoindentation experiments, FE simulations, and plate bending model.

To investigate further the effects of film thickness on the indentation response, FE simulations were performed for displacements up to ten times the film thickness for W/PS systems with film thicknesses $h_f = 100, 200, 400$ and 800 nm . The force-displacement curve for the 100 nm system is plotted on a log-log scale in Figure 3.4(a) to demonstrate the typical response of the different cases. The indentation force-displacement curves for a small displacement region, $0.1h_f < \delta < 0.5h_f$, and a large displacement region, $6.0h_f < \delta < 10.0h_f$, were fit to the power law relationship $P = A\delta^n$ to determine the scaling exponent n . The results in Table 3.3 showed that the indentation response exhibited two distinct regions. The force-displacement curve was nearly linear in the small displacement region as predicted by the plate bending model. Moreover, the scaling exponent did not change significantly for larger displacements up the film thickness in all cases. Increasing the fitting region to $\delta = h_f$ caused the scaling exponent to increase by less than 0.8% for all cases. In contrast, the scaling exponent increased rapidly to $n = 1.5$ for displacements $\delta > 4.0h_f$ and remained relatively unchanged for the remainder of the simulations as shown in Fig. 3.4(a). Plots of the radial stress at $r = 0$ (not shown) showed that the tensile radial stress developed at the film/substrate interface surpassed the compressive radial stress at the film surface soon after the displacement exceeded the film thickness for all cases. The former increased linearly with displacements, while the later decreased linearly, becoming tensile. These observations

indicated the development of significant membrane stresses under the indenter for $\delta > h_f$, and this most likely caused the force-displacement response to stiffen.

Table 3.3: Scaling exponent of force-displacement curves for small and large displacements.

Film thickness (nm)	100	200	400	800
$0.1h_f < \delta < 0.5h_f$	1.06	1.05	1.06	1.12
$6.0h_f < \delta < 10.0h_f$	1.52	1.52	1.53	1.48

To determine the region of applicability of the linear bending model, the effective stiffness were calculated for the FE model systems by fitting the linear region of the force-displacement curves. The results are plotted as a function of film thickness in Fig. 3.4(b) along with eq. (3.1) of the linear plate bending model and the results of the nanoindentation experiments for the W/PS systems. The analytical prediction of the plate bending model agreed well with the experimental data and finite element results for all cases.

Figure 3.5: The effective modulus K/h_f from finite element simulations of (a) tungsten film systems with different substrate moduli, and (b) polystyrene substrate systems with different film moduli. The effective stiffness measured for the W/PS, W/SU8, and W/FS systems and calculated from the linear plate bending model are plotted also for comparison.

The FE simulations were applied next to examine the effects of substrate and film compliance on the small displacement response. The effective modulus, defined as K/h_f , was calculated from the FE simulation results and from eq. (3.1) of the linear plate bending model for 100 nm tungsten film systems with different substrate modulus, $E_s^* = E_s / (1 - \nu_s^2)$. The results are plotted in Fig. 3.5(a) along with experimental data from 100 nm W/PS and 100 nm W/FS systems. The figure also includes data from a previous set of nanoindentation experiments for a tungsten-epoxy system with a 500nm thick film (W/SU8 500nm). Figure 3.5(b) plots the effective modulus for 100 nm thick film/polystyrene substrate systems with different film modulus, $E_f^* = E_f / (1 - \nu_f^2)$. The

results in both figures showed good quantitative agreement between the plate-bending model, FE models, and nanoindentation experiment for large moduli ratios, $E_f^*/E_s^* > 100$. The moduli ratios of the W/PS and W/FS systems were 129 and 5.6. Consequently, the plate bending model accurately reproduced the effective stiffness of the W/PS system, but not of the W/FS system. The moduli ratio of the W/SU8 system was 67, and good agreement was also observed for the W/SU8 data, which were measured in a similar but separate set of experiments.

Figure 3.6: The force-displacement curves from finite element simulations of: (a) W/PS systems with different film thicknesses, (b) 100 nm tungsten film systems with different substrate moduli, (c) polystyrene substrate systems with a 100 nm film and different film moduli, (d) The W/PS system for different indenter radius R_i .

Figures 3.6(a)-3.6(c) plot the large indentation response of the finite element studies, showing the effects of film thickness, substrate modulus, and film modulus. The results showed that the large displacement response was described reasonably well by the following scaling relation,

$$\frac{P}{Kh_f} = C \left(\frac{\delta}{h_f} \right)^{1.5} \quad (3.3)$$

where K is the effective stiffness in eq. (3.1), and C is a scaling coefficient. This result was consistent with the dimensional analysis of the nonlinear plate bending model of [53]. This indicates the effect of flexure of the stiff film remains significant for displacements up to ten times the thickness. The coefficient C showed a weak dependence on the film thickness, but little dependence on the substrate and film moduli for very stiff films, $E_f/E_s^* > 1000$.

Figure 3.6(d) plots the indentation response of the 100 nm W/PS system for different indenter radius R_i . For $\delta < h_f$, the indenter radius had a small but noticeable effect on the force-displacement relationship. As expected, a smaller R_i produced better agreement with the linear plate-bending model, which assumed a concentrated applied force. The indenter radius had a larger effect on the force-displacement curves for $\delta > h_f$, and a larger indenter produced a stiffer response.

3.4 Summary

The elastic indentation response of compliant substrate systems were characterized using nanoindentation experiments and finite element simulations. The experiments and simulations investigated the effects of rate-dependence, residual film stresses, film thickness, film and substrate compliance, and indenter radius. The main conclusions of the chapter were:

- The elastic indentation response in the bending dominated region were not sensitive to the residual stress in the film.
- The linear plate bending model of Ramsey et al. [53] accurately reproduced the indentation force-displacement response of the W/PS systems and finite element models for small displacements, $0.1h_f < \delta < h_f$, and for large ratios of film to substrate moduli, $E_f/E_s^* > 100$.
- For large displacements, $\delta > 4.0h_f$, the force-displacement relationship was described reasonably well by the scaling relationship $P/(Kh_f) = C(\delta/h_f)^{1.5}$. For very stiff films, $E_f/E_s^* > 1000$, the scaling parameter C displayed a slight dependence on the film thickness but was insensitive to the film and substrate moduli.
- The indenter radius had a slight effect on the small displacement response. A smaller indenter radius produced better agreement with the linear plate bending model. The indenter radius exerted a more pronounced effect for larger displacements $\delta > h_f$, and a larger indenter radius produced a stiffer response.

It is interesting to note that the $n = 1.5$ scaling exponent calculated for large displacements also describes the force-displacement relationship of the Hertzian spherical

contact problem. In the limit of a vanishingly thin film, one would expect to recover the Hertzian contact response. However, the large displacement response conformed to the dimensional analysis of plate bending model. This indicated that the effects of film bending remained dominant even for the largest displacements examined. For most applications, large displacements would be accompanied by film fracture and/or viscoelastic/plastic deformation in the substrate. The effects of both are currently being investigated.

4. Fracture Process Modeling

E. D. Reedy, Jr., E. Corona

4.1 Introduction

Two plane strain, thin film interfacial cracking problems were examined in this study: 1) cracking from the root of a channel crack when the thin film is subjected to tensile residual biaxial stress (Fig. 4.1a), and 2) buckle-driven interfacial cracking when the thin film is subjected to compressive residual biaxial stress (Fig. 4.1b). A cohesive zone modeling approach was used for both types of problems. A cohesive zone separation model is computationally attractive for simulating interfacial failure since crack growth is a natural outcome of the solution. A cohesive zone model is typically implemented as a zero-thickness surface element that is inserted along the interface between two materials. The interface element's constitutive model determines how interfacial stresses depend on relative displacements across the interface (defined by an effective traction-separation relationship). The particular cohesive zone formulation used in this study, which is based upon a traction potential, is similar to that used by Tvergaard and Hutchinson [63]. For the sake of simplicity the effective traction-separation relationship was taken to be triangular with a steep loading segment. The key parameters defining this traction-separation relationship are the interfacial strength $\hat{\sigma}$ and the work of separation/unit area of interface, which is also referred to as the interfacial toughness Γ . Note that Γ is independent of loading mode mixity and is associated with the area under the traction-separation relationship while $\hat{\sigma}$ determines the peak stress transferred across the interface as well as the length of the cohesive zone. Since the cohesive zone model includes a length scale, calculated results are mesh-independent provided that the mesh is fine enough to resolve the cohesive zone --- the region of interfacial softening behind the crack tip.

4.2 Channel Cracks

4.2.1 Channel crack modeling techniques

Simulating delamination from the root of a channel crack in a thin film subjected to a residual biaxial tensile stress is reasonably straight-forward provided that the film is stiff relative to the substrate. In this case, a monotonically increasing tensile film stress causes stable interfacial crack growth until the steady-state limit is reached (where crack growth proceeds without a further increase in film stress). Only a vanishingly small pre-existing interfacial defect is required to initiate crack growth. This follows from the fact that for a prescribed film tensile stress, the energy release rate monotonically decreases with increasing delamination length and is unbounded as the interfacial crack length approaches zero [64].

4.2.2 Channel crack results

The effect of substrate compliance on interfacial cracking from the root of a channel crack was investigated (i.e., Fig 4.1a geometry). The film is subjected to a monotonically increasing residual biaxial tensile stress σ_o . Results are for a 0.1- μm thick tungsten film ($E=410$ GPa) on four different substrates (Al_2O_3 with $E=370$ GPa, polycrystalline silicon with $E=160$ GPa, fused silica with $E=70$ GPa, and PMMA with $E=3$ GPa). Test calculations showed that in the case of the very compliant PMMA substrate, the substrate must be $5000h$ thick and wide to minimize substrate size effects. The interface was assumed to have an intrinsic interfacial toughness $\Gamma = 0.5 \text{ J/m}^2$. Figure 4.2 plots film stress σ_o (normalized by $\sigma^* = \sqrt{2\bar{E}_1\Gamma/h}$) vs. interfacial half-crack length a (normalized by h). Note that σ^* is the biaxial film stress associated with the long crack limit for an interface with toughness Γ . In the steady-state (long crack) limit, crack growth increases rapidly with a small increase in σ_o . The amount of crack growth required to approach the steady-state limit increases with increasing substrate compliance. Most notably, the low compliance PMMA substrate needs over $500h$ of crack growth to approach the steady state limit while the other substrates require crack growth of less than $10h$. Note that in the steady-state limit, interfacial toughness simply equals $\Gamma = \sigma_o^2 h / (2\bar{E}_1)$.

Since the PMMA substrate can yield, the effect of such yielding on interfacial crack growth was also investigated. The PMMA was idealized as an elastic-perfectly plastic material with a shear yield strength $\tau_y = 46$ MPa. Results plotted in Fig. 4.3 are again for a 0.1 μm -thick W film on a $5000h$ thick and wide substrate, where $\Gamma=0.5 \text{ J/m}^2$. The value of the interfacial strength $\hat{\sigma}$ is varied from 25 to 60 MPa. When $\hat{\sigma} < \tau_y$, there is little or no yielding at the crack tip as the crack propagates. When $\hat{\sigma} > \tau_y$, the PMMA substrate yields and the crack blunts with increasing $\hat{\sigma}$. Blunting occurs because yielding limits the magnitude of the interfacial stress that can be generated in this primarily Mode II loading to a value of τ_y , thus limiting the interfacial stress to a value less than that needed to cause separation.

4.3 Buckle-driven interfacial cracking

4.3.1 Buckle-driven interfacial cracking modeling techniques

Analytic results for a thin elastic film on a rigid substrate are well established for the case of a one-dimensional blister [65-68]. The analysis of this particular problem is simplified by the fact that there is no substrate deformation and the ends of the buckle can be considered to be fixed. There are also a number of published results for the case of a compliant substrate [37,38], however, results for a very stiff film on a very compliant substrate have not been fully determined (e.g., W/PMMA). Furthermore, there appears to be little work aimed at understanding the effect of substrate yielding when the thin film is relatively stiff and elastic.

Fig. 4.1. Analyzed a) growth of an interfacial crack that initiates from the root of a channel crack in a thin film subjected to tensile residual film stress, and 2) growth of a buckle-driven interfacial crack in a thin film subjected to a compressive residual film stress.

Figure 4.2. The effect of substrate compliance on interfacial crack growth from the root of a channel crack in a tungsten film for monotonically increasing tensile film stress σ_0 .

Fig. 4.3. The effect of interfacial strength on interfacial crack growth from the root of a channel crack in a tungsten film on an elastic-perfectly plastic PMMA substrate for monotonically increasing tensile film stress σ_o .

Buckle-driven interfacial cracking is a relatively complex phenomenon to simulate using the finite element method. An initial delamination as well as a geometric perturbation is required to enable buckling, and buckling, by its very nature, requires a large deflection analysis. The film compressive stress must exceed the critical buckling stress of the delaminated segment for the buckle to grow. For a pre-existing flaw and a prescribed film stress equal to the critical buckling stress, the initially zero energy release rate increases with increasing buckle (crack) length until reaching a maximum and then decreases monotonically. Depending on the level of the prescribed film stress, the initial flaw length, and the toughness of the interface, the delamination may or may not propagate, and if it does propagate it may or may not arrest. Based upon dimensional considerations,

$$\frac{b}{h} = f\left(\frac{\Gamma}{G_o}, \frac{\sigma_o}{\bar{E}_1}, \alpha, \beta\right) \quad (4.1)$$

where $G_o = \sigma_o^2 h / 2\bar{E}$ and $\bar{E}_i = E_i / (1 - \nu_i^2)$ with $i=1$ for the film and 2 for the substrate.

Dundurs' parameters α and β depend on the elastic properties of the film and substrate. In particular, $\alpha = (\bar{E}_1 - \bar{E}_2) / (\bar{E}_1 + \bar{E}_2)$ is often used to characterize the relative stiffness of the film and substrate ($\alpha=0$ for identical materials, -1 when the film is very compliant, and +1 when the film is very stiff). Based upon Eq. 4.1, one would like to determine the relationship between delamination length and interfacial toughness for a prescribed value of film stress (i.e., determine b/h as a function of Γ/G_o for prescribed values of $\sigma_o/\bar{E}_1$, α

and β). This was done in the following manner. The first step in this quasi-static finite element analysis is to generate a biaxial film stress equal to σ_o (e.g., prescribe a thermal strain increment). Note that the finite element model is constructed so as to contain an initial delamination with a length shorter than the critical buckling length. The next step in the finite element simulation is to displace the center of the pre-existing delamination upwards so as to define an increasing delamination height δ . The applied force associated with this applied displacement is monitored as function of δ . The applied force is first positive (need to pull on the delamination to open it up and make it grow), then turns negative (need to restrain the delamination because the buckle wants to grow unstably), and finally the force again turns positive (need to pull on an arrested buckle to make it grow longer). There are two δ values where the applied force equals zero and a free-standing buckle exists. This corresponds to the two buckle lengths that can exist for the prescribed Γ and σ_o values (the shorter buckle is unstable and will grow unless constrained while the longer buckle is arrested and stable). This analysis is repeated for a broad range of Γ values (σ_o remains fixed) to determine the relationship between delamination height (width) and interfacial toughness. The relationship between δ and b is also determined by this technique.

Unless indicated otherwise, all results reported below are for a 0.1- μm thick W-film on a 500- μm thick and wide PMMA substrate. As indicated above when channel cracks were discussed, test calculations showed that a substrate of this relatively large size is required to minimize substrate size effects (i.e., used a substrate that was $5000h$ thick and wide). Also note that all buckle-driven interface cracking calculations were performed using eight-node, plane strain elements since such 2nd order elements can accurately model bending when using only one element through the film thickness (as was done in the calculations presented here). The tungsten film has a Young's modulus $E=410$ GPa and a Poisson's ration $\nu = 0.28$ while the PMMA substrate has an $E=3$ GPa, and $\nu = 0.35$. For this combination of elastic properties, the values of Dundurs' parameters are $\alpha=0.985$ and $\beta=0.227$.

The accuracy and convergence of this simulation technique used in this study has been verified. Table 4.1 reports results for the case of PMMA thin film on a W substrate for $h=0.1$ μm , $\sigma_o=50$ MPa, $\Gamma = 0.04$ J/m², and $\hat{\sigma} = 15$ MPa. Since there is a two orders of magnitude difference in the film and substrate Young's modulus, the results of the finite element calculation should closely match those determined by using the analytic results for a compliant film on a rigid substrate [66]. The comparison of analytic and finite element results presented in Table 1 shows that there is indeed good agreement. Note that Δ is the characteristic length of the cohesive zone element, and calculated results are relatively insensitive to mesh refinement. The effect of mesh refinement on the calculated results for the W-film on a PMMA substrate problem of interest is reported in Table 4.2. These results are for $\sigma_o=1700$ MPa, $\Gamma=1.0$ J/m², and $\hat{\sigma} = 100$ MPa. Table 4.2 shows that the mesh is sufficiently refined to accurately determine buckle height δ and buckle width $2b$ when $\Delta=h=0.1$ μm . The choice for the value of the interfacial strength $\hat{\sigma}$ is not obvious for the case when the film and substrate are both linear elastic materials. For a fixed value of the interfacial toughness Γ , the primary effect of varying $\hat{\sigma}$ (or

equivalently the interfacial separation distance) is to change the length of the cohesive zone. Calculated results are mesh-independent provided that the mesh is sufficient to resolve the cohesive zone --- the region of interfacial softening behind the advancing crack tip. Table 4.3 presents results that illustrate the effect $\hat{\sigma}$ on calculated results ($\sigma_0=1700$ MPa, $\Gamma = 1.0$ J/m², and $\Delta = 0.1$ μ m). There is only a modest variation in the calculated half-buckle width b when the interface strength $\hat{\sigma}$ varies from 80 to 120 MPa, with the cohesive zone (CZ) length varying from $6h$ to $3h$. Note that b is measured from tip of cohesive zone, where the stress is equal to $\hat{\sigma}$. In the calculations discussed below, $\hat{\sigma}$ was chosen so as to maintain a CZ length that was within the range of ~ 3 - $6h$. Note that although this analysis technique generates accurate results for energy release rate and buckle width and height, it cannot accurately determine crack-tip mode mixity. A direct determination of crack-tip mode mixity would require a much finer mesh and a much smaller CZ length; mode mixity is associated with the complex crack-tip stress intensity factor and the region of dominated by the associated stress singularity is a small fraction of the thin film thickness. The present analysis is analogous to the use of beam theory to calculate energy release rates for beam-like specimens (e.g., DCB specimens).

Table 4.1. Results for a PMMA thin film on a W substrate.

	Baseline mesh $\Delta=0.1$ μ m	Refined mesh $\Delta=0.05$ μ m	Rigid substrate theory
δ/h	5.0	5.1	5.0
b/h	34.7	35.1	33.2

Table 4.2. Effect of mesh refinement on calculated results for a W film on a PMMA substrate.

	Baseline mesh $\Delta=0.1$ μ m	Refined mesh $\Delta=0.05$ μ m
δ/h	6.8	6.8
b/h	44.7	44.6

Table 4.3. Effect of the value of interfacial strength $\hat{\sigma}$ on calculated results for a W film on a PMMA substrate when interfacial toughness is fixed at $\Gamma=1.0$ J/m².

	$\hat{\sigma} = 50$ MPa	$\hat{\sigma} = 80$ MPa	$\hat{\sigma} = 100$ MPa	$\hat{\sigma} = 120$ MPa
δ/h	6.9	6.8	6.8	6.8
b/h	48.0	45.3	44.7	44.3
CZ length/ h	9	6	4	3

4.3.2 Buckle-driven interfacial cracking results

Figure 4.4 plots the calculated calibration relating normalized interfacial toughness Γ to normalized buckle half width b for a 0.1 μm tungsten film on either a PMMA or a rigid substrate ($\sigma_o = 1.7$ GPa). For this choice of σ_o , G_o , which is equal to the long crack energy release rate, has a value of 0.325 J/m² and $\sigma_o / \bar{E}_1 = 0.0038$. Figure 4.4 clearly illustrates the significant effect of substrate compliance. If one were to use the rigid substrate calibration to determine the interfacial toughness from a measurement of the buckle width when the substrate was actually a rather compliant PMMA substrate, then one would significantly underestimate interfacial toughness. For a $b/h=20$, there is more than a factor of 5 difference in Γ/G_o values for PMMA and rigid substrates. Figure 4.5 plots normalized buckle height versus buckle half width. For a given value of b/h , buckles on a PMMA substrate are much higher than those on a rigid substrate. A comparison of experimentally determined buckle height versus buckle width results with the calculated Fig. 4.5 results permits an assessment of the fidelity of the analysis. Differences in the measured and calculated relationship between buckle height and buckle width suggests that either that the parameters used in the analysis were not chosen properly (e.g., σ_o , which is not measured directly) or the underlying assumption used in the analysis are violated (e.g., of purely elastic film and substrate response).

Fig. 4.4. Calibration relating interfacial toughness Γ to buckle half width b for the case of a 0.1 μm tungsten film on either a PMMA or a rigid substrate ($\sigma_o = 1.7$ GPa).

Fig. 4.5. Calibration relating buckle height δ to buckle half width b for the case of a 0.1 μm tungsten film on either a PMMA or a rigid substrate ($\sigma_o = 1.7 \text{ GPa}$).

The results plotted in Figs. 4.6 and 4.7 illustrate the effect that residual biaxial compressive film stress has on buckle-driven interfacial cracking ($\sigma_o = 0.85, 1.7$ and 3.4 GPa). The dependence of Γ/G_o on $\sigma_o/\bar{E}_1$ has been previously discussed by Yu and Hutchinson [38] and is expected based upon dimensional considerations (Eq. 4.1). The greater deformation associated with higher values of $\sigma_o/\bar{E}_1$ generate higher peak Γ/G_o values. Figure 4.7 also shows that higher values of $\sigma_o/\bar{E}_1$ also generate larger buckle heights at a given buckle width. Results such as those plotted in Fig. 4.7 could be used in conjunction with the measured δ/h versus b/h values to deduce the value of σ_o that is actually present in a thin film. Naturally this assumes that all other parameter choices, as well as the underlying assumption of purely elastic film and substrate response, are valid.

An alternate method for modeling buckle-driven interfacial cracking was also developed. In this approach, instead of fixing the value of σ_o , σ_o is monotonically increased (i.e., the finite element calculation imposes an increasing thermal film strain). Consequently $\sigma_o/\bar{E}_1$ increases with increasing σ_o . The calculation starts with a small preexisting delamination, and a load is applied to open the delamination. This load is fully released prior to any delamination growth so that it does not affect crack growth. The purpose of this loading is to create an initial “imperfection” that enables lateral deflection upon the

Fig. 4.6. Calibration relating interfacial toughness Γ to buckle half width b for the case of a 0.1 μm tungsten film on a PMMA substrate for $\sigma_o = 0.85, 1.7$ and 3.4 GPa.

Fig. 4.7. Calibration relating buckle height δ to buckle half width b for the case of a 0.1 μm tungsten film on a PMMA substrate for $\sigma_o = 0.85, 1.7$ and 3.4 GPa.

Fig. 4.8. Comparison of results for a monotonically increasing film stress to those that have a fixed film stress (0.1 μm tungsten film on a PMMA substrate).

application of the monotonically increasing compressive film stress (another approach would be to simply introduce a geometric imperfection directly into the mesh). Figure 4.8 presents results for an interface with an initial delamination of 3 μm ($b/h=30$) and an interfacial toughness of $\Gamma = 0.5 \text{ J/m}^2$. Results for a monotonically increasing film stress, where $\sigma_o / \bar{E}_1$ varied from 0.0012 to 0.0037, are compared those with a fixed film stress, where $\sigma_o / \bar{E}_1 = 0.0019$ or 0.0038 (from Fig. 4.6). Figure 4.8 shows that the two approaches yield consistent results, with the curve with varying $\sigma_o / \bar{E}_1$ transitioning between those with fixed values of $\sigma_o / \bar{E}_1$. Note that calculations where σ_o is fixed, G_o is fixed while Γ is varied, while in the calculations with monotonically increasing σ_o , G_o varies while Γ is fixed.

As discussed above, substrate yielding was found to have a significant effect on interfacial cracking from the root of a channel crack (Fig. 4.3). That is also true for buckle-driven interfacial cracking. As before, the PMMA was idealized as an elastic-perfectly plastic material with a shear yield strength $\tau_y = 46 \text{ MPa}$. The interfacial toughness was fixed at 0.5 J/m^2 , and the interfacial strength $\hat{\sigma}$ was varied from 40 to 70 MPa. In these calculations the buckle width was determined as the compressive film stress σ_o was monotonically increased. Figure 4.9 plots film stress σ_o (normalized

Fig. 4.9. The effect of interfacial strength on buckle width for a 0.1 μm tungsten film on an elastic-perfectly plastic PMMA substrate for case of monotonically increasing compressive film stress σ_0 .

Fig. 4.10. The effect of interfacial strength on buckle width vs. height relationship for a 0.1 μm tungsten film on an elastic-perfectly plastic PMMA substrate for case of monotonically increasing compressive film stress σ_0 .

by $\sigma^* = \sqrt{2E_I\Gamma/h}$, the biaxial film stress associated with the long crack limit for an interface with toughness Γ vs. interfacial half-buckle width b (normalized by h). The results are qualitatively similar to those for channel cracks. When $\hat{\sigma} > \tau_y$, there is substrate yielding and the crack blunts with increasing $\hat{\sigma}$. Yielding limits the interfacial shear stress in this primarily Mode II loading to a value of τ_y , thus preventing interfacial separation. Figure 4.10 shows the associated buckle width vs. height relationship. As the crack blunts, the buckle height increases for a given buckle width.

Experiments on a 0.1-micron thick, tungsten film on a commercial PMMA substrate generated spontaneous delamination buckles. The buckles appeared to fall into two groups; referred to as small and large buckles. The tungsten film had been deposited in such a way that it was highly compressed, with a residual biaxial film stress σ_o of -1.7 GPa. The buckle heights δ and half widths b (from Fig. 2.18a) were measured and are plotted in Fig. 4.11. AFM images of the substrate showed apparent localized plastic deformation at the buckle edges, with the small buckles having a significantly greater residual deformation. Also shown on this plot is the calculated δ/h versus b/h relationship for a linear elastic PMMA substrate ($\sigma_o = -1.7$ GPa). As anticipated the calculated results are a lower bound to the experimental results; as shown in Fig. 4.10 plastic yielding increases buckle height.

Fig. 4.11. Comparison of data for a 0.1 μ m tungsten film on commercial PMMA substrate with finite element results for an elastic PMMA substrate ($\sigma_o = 1.7$ GPa).

4.4 Summary

There is an increasing interest in the development of flexible, thin film structures for applications that include flexible displays and tactile sensor arrays. Interfacial cracking is a potentially important factor limiting the use of such systems. This has motivated a fracture analysis of thin films on a compliant substrate. The analysis has focused on the extreme case of a 0.1-micron thick tungsten film ($E=410$ GPa) on a thick PMMA ($E=3$ GPa) substrate. Interfacial cracking from the root of a thru-the-film channel crack can occur when the film stress is tensile. Buckle-driven interfacial cracking can occur when the film stress is compressive. The finite element analysis techniques that were developed to analyze both of these problems use a cohesive zone model to simulate interfacial crack growth. The analysis indicates that substrate compliance can have a significant effect on the calculated crack growth. Analyses that include substrate yielding suggest that yielding can greatly increase apparent interfacial toughness

4.5 Appendix

The objective of this appendix is to compare the calculated buckle widths for different values of interfacial fracture toughness for a film under plane strain biaxial compressive stress (see Fig. 4A1(b)) obtained with the procedure outlined in Section 4.3 against the energy release rate vs. buckle width curves calculated by Yu and Hutchinson [8]. In the present analysis the buckle width is grown quasi-statically by prescribing the transverse displacement at the center of the buckle while monitoring the required transverse point force. When the buckle width is growing the energy release rate is equal to the interfacial toughness. It follows that at the instants when the transverse point force is zero this equality occurs with the only applied force being the biaxial compression in the film. Therefore, the calculated buckle widths when the transverse point load is zero should fall on the energy release rate vs. buckle width curves shown in [37].

Calculations are conducted for four elastic mismatches given by the Dundurs' parameters $\alpha = 0, 0.5, 0.9$ and 0.99 , $\beta = 0$ and two values of normalized pre-stress, $\sigma_o / \bar{E}_1 = 1\%$ and 0.1% where the subscript 1 denotes the film and 2 will denote the substrate. The Young's moduli are given by E_i and $\bar{E}_i = E_i / (1 - \nu_i^2)$, where ν_i represents the Poisson's ratios. Yu and Hutchinson considered two ways of calculating the energy release rate vs. buckle width curves. One is based on the expressions for the stress intensity factors while the other is based on a more direct energy calculation. Interestingly, the two approaches yielded different results, especially for very compliant substrates and high film pre-stress. They attributed the difference mainly to geometric nonlinearities in the problem. The finite element formulation utilized here accounts for geometric nonlinearities, so it would be interesting to see whether the predictions fall closer to one or the other set of results shown in [37].

The procedure to generate the results is the same as described in Section 4.3 and utilized the same dimensions, mesh, element type, interface model, boundary conditions and loading conditions. The material properties were different in order to accommodate the variation in α and to keep $\beta = 0$ as was done in [37]. The elastic properties for the film were kept fixed while those of the substrate were varied as shown in Table 4A1. In addition, the initial delamination length was adjusted depending on the value of pre-stress. A length $b_o = 0.3 \mu\text{m}$ was used when $\sigma_o / \bar{E}_1 = 1\%$ and $b_o = 1 \mu\text{m}$ when $\sigma_o / \bar{E}_1 = 0.1\%$.

The results obtained are presented in Tables 4A2 and 4A3 for $\sigma_o / \bar{E}_1 = 1\%$ and 0.1% respectively. As in the results presented in Section 4.3, two buckle half-widths, b_1 and b_2 for which the transverse point force is zero were found in most cases. The buckle half-width was measured from the tip of the cohesive zone. The fracture toughness Γ is normalized by the steady-state energy release rate $G_o = \frac{1}{2} \frac{\sigma_o^2 h}{E_1}$ while the buckle half-width is normalized by the half-width of a buckle on a film clamped at its edges and

subjected to the same value of $\sigma_o/\bar{E}_1$, given by $b_* = \frac{\pi h}{2\sqrt{3}} \sqrt{\frac{\bar{E}_1}{\sigma_o}}$. The value of the interface toughness, Γ , is set by choosing an appropriate combination of peak stress $\bar{\sigma}$ and total decohesion displacement δ_c (the same value was chosen for normal and shear modes). In general, for a given value of Γ , decreasing δ_c (and correspondingly increasing $\bar{\sigma}$) leads to shorter cohesive zones, but makes convergence of the nonlinear solver more difficult. With this in mind, a large enough value of $\bar{\sigma}$ to keep the length of the cohesive zone (l) as short as possible, typically in the range $l/h < 6$, and that allowed convergence was chosen for each case. The values of $\bar{\sigma}$ and approximate values of l/h in each case are also listed in Tables A2 and A3. The entries with the highest values of Γ for each combination of α and $\sigma_o/\bar{E}_1$ do not list results. In these cases the sign changes of the transverse point force could not be identified and therefore indicate that the interface toughness is high enough to prevent buckle-driven delamination.

The results obtained using the present analysis are compared to those in [37] in Figs. 4A1 (a)-(d) for each value of α . The comparison shows that the results obtained tend to give slightly larger buckle widths in many cases. The largest differences occur in the cases with $\alpha = 0$, $\sigma/\bar{E}_f = 1\%$ and large Γ where they are in the order of 12%. In many other cases the differences are much smaller, under 5%. All of the results of the present analysis fall much closer to the solid lines corresponding to the calculations in [37] obtained from the stress intensity factor calculations than to the dashed lines corresponding to the energy calculation.

Since the half-width of the buckles is measured from the tip of the cohesive zone, the measurements must necessarily depend on the value of $\bar{\sigma}$ used. Figure 4A2 shows the measured buckle half width as function of the value of $\bar{\sigma}$, when all other parameters are fixed. Interestingly, the longer buckle width (b_2) is more sensitive. As $\bar{\sigma}$ increases b_2 becomes smaller, and so does the cohesive zone. The trend shows that the results are converging as $\bar{\sigma}$ increases. This figure shows the importance of using values of $\bar{\sigma}$ that are as high as possible. Interestingly, subtracting the length of the cohesive zone from each of the results in Fig. 4A2 yields a nearly constant value.

Table 4A1: Film (1) and substrate (2) elastic properties used in the calculation of buckle widths as function of the interfacial fracture toughness ($\beta = 0$).

α	E_1 , MPa	ν_1	E_2 , MPa	ν_2
0.99	410,000	0	1,547.82	0.49874
0.90	410,000	0	16,471.8	0.48649
0.50	410,000	0	114,800	0.4
0.00	410,000	0	410,000	0

Table 4A2: Calculated buckle widths and approximate cohesive zone lengths for each value of interface fracture toughness. The values of the peak stress in the cohesive model is also included. ($\sigma/\bar{E}_f = 1\%$, $G_o = 2.05 \text{ Pa} \cdot \text{m}$, $b_* = 0.9069 \mu\text{m}$)

α	Γ/G_o	$\bar{\sigma}$, MPa	b_1/b_*	b_2/b_*	l_1/h	l_2/h
0	1.0	1500	1.189	-	2	-
0	1.2	1500	1.30	3.33	2	2
0	1.3	1000	1.362	2.807	2	3
0	1.4	1500	1.442	2.109	2	2
0	1.5	1500	-	-	-	-
0.5	1	600	1.140	-	2	-
0.5	1.25	800	1.189	3.697	2	3
0.5	1.375	800	1.251	2.919	2	3
0.5	1.5	1000	1.300	2.363	2	2
0.5	1.625	1000	1.412	2.029	2	2
0.5	1.70	1000	-	-	-	-
0.9	1.3	400	0.967	5.698	2	4
0.9	1.5	400	1.029	4.191	2	2
0.9	2.1	400	1.078	2.634	2	4
0.9	2.5	400	1.140	2.140	3	3
0.9	3	400	1.300	1.665	4	4
0.9	3.3	400	-	-	-	-
0.99	3	200	*	4.49	-	4
0.99	6	200	.776	2.515	4	5
0.99	8	200	0.917	2.079	5	6
0.99	10	200	1.029	1.696	6	5
0.99	12	300	1.109	1.251	4	4
0.99	14	-	-	-	-	-

* To achieve convergence $b_o = 1 \mu\text{m}$ was used in this case.

Table 4A3: Calculated buckle widths and approximate cohesive zone lengths for each value of interface fracture toughness. The values of the peak stress in the cohesive model is also included. ($\sigma/\bar{E}_f = 0.1\%$, $G_o = 0.0205$ Pa - m, $b_* = 2.868 \mu\text{m}$)

α	Γ/G_o	$\bar{\sigma}$, MPa	b_1/b_*	b_2/b_*	l_1/h	l_2/h
0	1.0	60	1.22	-	3	-
0	1.2	70	1.315	3.183	3	4
0	1.3	60	1.420	2.485	4	5
0	1.35	60	1.490	2.197	3	5
0	1.45	60	-	-	-	-
0.5	1.15	40	1.160	4.209	3	8
0.5	1.25	40	1.280	3.138	3	8
0.5	1.375	50	1.360	2.371	3	5
0.5	1.45	50	1.439	2.048	3	5
0.5	1.5	50	-	-	-	-
0.9	1.25	25	1.106	4.37	3	7
0.9	1.5	40	1.14	2.85	3	5
0.9	2	40	1.300	1.803	2	4
0.9	2.2	-	-	-	-	-
0.99	2	15	0.951	4.314	3	10
0.99	4	20	1.021	2.197	3	3
0.99	6	20	1.16	1.524	4	4
0.99	7	20	-	-	-	-

(a)

(b)

Fig. 4A1: Comparison of buckle widths as function of the interfacial fracture toughness, for different elastic mismatches and pre-stress values as obtained by the present analysis to the results for the energy release rate as a function of buckle width calculated by Yu and Hutchinson (Y/H). (a) $\alpha = 0$ and (b) $\alpha = 0.5$.

(c)

(d)

Fig. 4A1: Continued. (c) $\alpha = 0.90$ and (d) $\alpha = 0.99$.

Fig. 4A2: Calculated buckle widths as a function of peak stress in the cohesive zone model. The equation solver diverged for values of 1400 MPa or larger.

5. Molecular Dynamics Simulation of Delamination of a Model Tungsten Film from a Silicon Substrate

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5.1 Introduction

Experiments have indicated that W films deposited onto Si substrates can delaminate at the interface through buckling and cracking [36]. However, the atomic mechanisms of the deformation and fracture of the films have not been well studied. Here we have applied molecular dynamics (MD) simulations to study the delamination of a model W crystalline film from a Si substrate due to tensile loading. Atomic mechanisms, such as dislocation nucleation, multiplication, and interaction are identified. The various defects that form prior to fracture are discussed and related to the mechanical properties of the system.

5.2 Method

5.2.1 Interatomic Potentials

The interactions between the Si atoms were modeled by a Tersoff potential using the parameters given by Tersoff for good elastic constant predictions [69,70]. The interactions between the model W atoms were modeled using the analytical embedded atom method approach [71-73] with the parameters listed in Table I. A Morse potential, $\phi(r) = 0.8\{\exp[-4(r-3)] - 2\exp[-2(r-3)]\}$, for r in Å and ϕ in eV, was used for the cross interaction between the W and Si atoms.

Table 5.1. Potential parameters for the model W.

$r_e(\text{\AA})$	f_e	$r_e = r_s$	a	b	A (eV)	B (eV)
2.740840	3.487340	37.234847	8.900114	4.746728	0.882435	1.394592
κ	λ	F_{n0} (eV)	F_{n1} (eV)	F_{n2} (eV)	F_{n3} (eV)	F_0 (eV)
0.5	1.0	-4.946281	-0.148818	0.365057	-4.432406	-4.96
F_1 (eV)	F_2 (eV)	F_3 (eV)	η	F_e (eV)	μ	ν
0	0.661935	0.348147	0.582714	-4.961306	0.85	1.15

For our applications, the material properties such as the lattice constants, the cohesive energies, the elastic constants, the surface energies, and the increases in energy during phase transformations are all important. The lattice constants affect lattice mismatch. The cohesive and surface energies are key parameters regarding fracture strength. The elastic constants determine the relative compliance of the materials and the dislocation energy (which in turn affects the elastic and plastic behavior). Finally, the increases in energy

due to the phase transformations determine both the deformation path (e.g., the phase transformation deformation mechanism of shape-memory alloys) and the stability of the (bcc) phase. The predicted values of these properties for the Si potential used have been previously published [36,69,70]. The values for the W potential were calculated here. All of the relevant properties predicted by both potentials are listed in Table II and III, along with experimentally determined values for Si [74,75]. The lattice constant, cohesive energy, elastic constants, and surface energies of the model W closely match with the experimental values [76-80]. In addition, the model for W correctly predicts an increase in energy due to a bcc $\rightarrow$ fcc (face-centered-cubic) phase change. The cross interaction was tuned to correctly predict the pressure profiles experimentally observed in W on Si films [81].

Table 5.2. Lattice constant a , cohesive energy E_c (eV/atom), elastic constants C_{11} (GPa), C_{12} (GPa), and C_{44} (GPa), and energy increase $\Delta E_{\text{bcc} \rightarrow \text{fcc}}$ (eV/atom) during bcc $\rightarrow$ fcc phase transformation. Calculated values of Si and experimental values (in parenthesis) are from reference [74].

element	a	E_c	C_{11}	C_{12}	C_{44}	$\Delta E_{\text{bcc} \rightarrow \text{fcc}}$
W	3.165	-8.76	522.5	204.2	160.8	0.16
Si	5.43 (5.43)	-4.63(-4.63)	143(167)	75(65)	69(81)	--

Table 5.3. Surface energies γ (J·m⁻²) for various surfaces. Calculated values of Si are from reference [74] and experimental values (in parenthesis) from reference [75].

element	{010}	{011}	{111}
W	2.98	2.56	3.32
Si	2.31(2.13)	1.57(1.51)	1.29(1.23)

5.2.2 Molecular Dynamics Model

Direct MD simulations of the deposition of W on a {010} Si surface [81] indicated that W grows into a {010} textured bcc crystal with its $\langle 100 \rangle$ directions parallel to the $\langle 101 \rangle$ directions of the {010} Si surface. This type of interface was selected for study in this work. Our system assumes that the interface between the two materials lies on the global x-z plane, and the tensile loading is applied in the y-direction corresponding to the [010] direction in both materials. Accordingly, the x-, y-, and z- axes of the global system are aligned with the [101], [010], and $[\bar{1}01]$ directions of the Si, and with the [100], [010], and [001] of the W. The system size in the x- and z- directions was chosen to be about 126.75 Å. This closely fits both 33 repeat units of Si {101} planes and 40 repeat units of W {100} planes, with a low mismatch strain in the W of about -0.00069. Periodic boundary conditions were imposed in the x- and z- directions. The film was given a finite thickness in the y- direction with the atoms in the outermost 15 Å on both free surfaces being held at perfect bulk lattice positions.

A reasonable interfacial structure was created by first positioning the W crystal above (positive y-direction) the Si crystal with a gap between the two materials slightly larger than the expected atomic spacing. Next, a molecular statics simulation was carried out to minimize the system energy and relax the interfacial structure. Finally, the interface was

further allowed to relax through the use of an MD simulation at the simulated temperature with no applied loads. In this work, the x- and z- dimensions were held constant and a Nose/Hoover thermostat [82] was used to control the temperature. An example of the simulated W/Si system containing a relaxed interface is shown in Fig. 5.1. We found that following the relaxation process, rippled structures developed at the interface, similar to what was previously observed in Ni/Si and Al/Si systems [83]. The rippling occurred due to W and Si atoms at the interface moving to fill the gaps produced by the mismatch between the two lattices.

Fig. 5.1. Front view of the simulated system.

To apply a tensile strain to the system, constant temperature MD simulations were performed where the bottom 15 Å of Si atoms were fixed and the top 15 Å of W atoms were constrained to move at a constant rate. A gauge length was specified within the

specimen prior to the test and all atoms within that section were identified. The change in length during the test was determined by finding the maximum and minimum y-coordinate for the atoms that were contained within the initial gauge length. The applied stress was calculated as the average Virial stress [84] over all of the gauged atoms. All simulations were performed using the LAMMPS software [85].

5.3 Results

5.3.1 Stress vs. Strain Relationships

The simulations were carried out at accelerated strain rates in the range $10^8 - 10^9 \text{ s}^{-1}$. To explore the effects of these accelerated strain rates and the small sample dimensions of the simulation systems, stress vs. strain curves were calculated for simulation runs with varying strain rates and sample thicknesses at a fixed temperature of 300 K. The results are shown in Fig. 5.2. Fig. 5.2(a) compares the behavior of two systems with different sample thicknesses both subject to the same strain rate of $2.88 \times 10^9 \text{ s}^{-1}$, while Fig. 5.2(b) shows the effect of two different strain rates at the same sample thickness ($\sim 87 \text{ \AA}$ W and $\sim 107 \text{ \AA}$ Si). The oscillation during elastic loading seen in Fig. 5.2 is known to result from a shockwave created during accelerated tensile loading [83]. The effect of the shockwave is effectively eliminated by using the relatively low strain rate of $2.88 \times 10^9 \text{ s}^{-1}$ as seen Fig. 5.2(b). It can be seen that the results, especially the elastic deformation and initial yielding, are not sensitive to the system dimension and strain rate within the explored ranges.

Fig. 5.2. (a) Thickness and (b) strain rate effects on stress vs. strain curves.

The stress vs. strain curves were also measured at two additional low temperatures of 20 K and 50 K with a fixed strain rate of $2.88 \times 10^9 \text{ s}^{-1}$ and a fixed sample thickness of $\sim 87 \text{ \AA}$ W and $\sim 107 \text{ \AA}$ Si. It was found that this change in temperature also did not affect the initial yielding.

Fig. 5.2 shows that upon loading, the specimen initially deformed elastically before yielding at a strain of approximately 0.1. Strain hardening can be seen following the yield up until fracture occurred at a strain of ~ 0.25 . To isolate the deformation behavior occurring in each material, two additional gauge lengths were specified: one containing only Si atoms and the other only W atoms. These smaller gauge lengths allowed for stress vs. strain plots to be developed for each component material in the composite system. Two examples of the stress vs. strain plots, measured respectively for the W and Si, are shown respectively in Figs. 3(a) and 3(b). As indicated in Fig. 5.3(a), the W shows three expected stages of behavior: elastic deformation, extensive yielding, and strain hardening. In contrast, Fig. 5.3(b) shows that the Si behaves elastically over the entire strain range explored, although the elastic behavior deviates from linearity at large strains. The plastic yielding within the W will be further explored through analysis of dislocation motion.

Because no shockwave was observed at the slower strain rates, and temperature and sample thickness have little effect on the overall behavior over the range explored, the remaining analysis will focus solely on one tested case: sample thickness of ~ 87 Å W and ~ 107 Å Si, strain rate of $2.97 \times 10^8 \text{ s}^{-1}$, and temperature of 300 K.

Fig. 5.3. Isolated (a) W and (b) Si behavior.

5.3.2 Fracture Observation

The evolution of atomic scale configurations around the time of fracture has been examined and one example is shown in Fig. 5.4. Analysis of the images showed that cracks initiated at one point along the interface and then proceeded to expand outward along the interface. This is consistent with the previous observations for Al/Si interfaces [83] that cracks initiated at the highly strained sites of the rippled interface. As the crack propagated along the interface, Fig. 5.4(b) and Fig. 5.4(c), Si developed a wavy appearance as the regions that had separated from the W attempted to relax to their unstrained positions while other regions remained attached. This is best seen in the diagonal view of the system, Fig. 5.4(c). It further confirms that Si is relatively more

compliant than W. The complete fracture, Fig. 5.4(d), resulted in the formation of an atomically clean W and Si surfaces. However, while the fractured Si surface remained atomically flat, the W surface appeared to contain many steps characteristic of dislocation motion.

5.3.3 Slip mechanisms at a small strain (< 0.12)

To more clearly show the defects that formed within the W during loading, parameters that distinguish defects from perfect lattice are needed. Here we use the slip vector parameter developed previously [86]. The slip vector is defined

as $\vec{S}_i = -\frac{1}{N_s} \sum_{j \neq i}^N (\vec{R}_{i,j} - \vec{R}_{i,j}^0)$, where N is the total number of nearest neighbors to atom i ,

N_s is the number of neighbors that are on an adjacent slip plane to atom i (e.g., $N_s = 3$ if slip occurs on an $\{111\}$ fcc lattice), $\vec{R}_{i,j}^0$ is the vector from atoms i to its neighbor j at an initial reference configuration, and $\vec{R}_{i,j}$ is the corresponding vector at the current configuration. By finding the relative displacement of the nearest neighbor atoms j with respect to a given atom i , it can be determined if a plane neighboring atom i has slipped and in what direction. Dividing by $-N_s$ scales the vector's magnitude so that it will be equal to the Burgers vector of the dislocation that caused the slip.

Fig. 5.4. Interfacial fracture process. (a) prior to crack formation; (b) during crack propagation; (c) another view of the crack propagation; and (d) after fracture.

The deformation mechanisms present during the simulations have been explored by mapping the magnitude of the slip vector to the atom configurations. This analysis revealed no defects within the Si and therefore confirmed that Si remained elastic prior to fracture. In sharp contrast, significant defects were seen in W. Here, we first demonstrate the defects formed in W at relatively small strains. One typical example is shown in Fig. 5.5 for a strain of 0.089. To improve the quality of the demonstration, the image was made after an energy minimization simulation was applied to relax the atom positions while under the applied strain. In Fig. 5.5, all Si atoms are colored white, whereas W atoms are colored according to the magnitude of the slip vector with the color scaled so that atoms with slipped distances outside of the 0.4 - 0.8 Å range are invisible. Fig. 5.5 clearly reveals slipped planes that formed during the early stage of loading. The slip occurred due to the emission of dislocations into the W from the interface primarily at the rippled sites where the mismatch stress was the highest. The onset of dislocation emission corresponded well with the yielding of the system. Fig. 5.5 shows that the magnitude of the slip vector is ~ 0.45 Å. If the crystallographic orientations are referenced with respect to the bcc W, then it can be seen that slip occurred on a $(\bar{1}21)$ plane, and that the in-plane slip vector is $(1/12)[11\bar{1}]$. This type of slip has a Schmid factor of 0.471. It is therefore a favorable system for slip, and is expected in bcc systems [87,88]. In addition, it can be seen that the slip planes are wavy and ill-defined, which is also consistent with the bcc lattice where slip planes can be a local combination of $\{110\}$ and $\{112\}$ planes [89].

Fig. 5.5. Slip configuration of system at a relatively small strain of 0.089. For W, only atoms with slip magnitude between 0.40 and 0.80 Å are shown.

5.3.4 Slip mechanisms at a large strain (>0.12)

A similar analysis of the slip vector has also been used to study the defects in W at large strains. As an example, Fig. 5.6 shows the slip configuration of the system at a strain of ~ 0.128 , where atoms with slipped distance outside of the 0.89 - 1.61 Å range are invisible. Fig. 5.6 clearly reveals extensive slipped planes that formed at the late stage of the tensile loading. Examinations indicated that the slip planes are of the $\{110\}$ type. Non-parallel $\{110\}$ planes interact to form a network of rectangular pipes along the $\{111\}$ direction. It can be seen that the yielding observed in Fig. 5.2 corresponds well with the extensive slip seen in Fig. 5.6.

Analyses indicated that during the loading, the leading partial dislocations are nucleated at the interface and then quickly swept through the crystal. If the strain is not significant enough to nucleate voids, the trailing partial dislocations can be pinned at the interface. This resulted in the formation of the observed structure of the slipped planes. To further examine the slipped nature and the Burgers vector of the partial dislocations, four consecutive $(\bar{1}10)$ planes in a circular region indicated in Fig. 5.6 are shown in Fig. 5.7 with the $[\bar{1}10]$ direction coming out of the image and the gray, white, blue, and (small) red circles ordered in the same direction. As a reference for comparing the two images, note that the gray and white atoms shown in Fig. 5.7 are invisible in Fig. 5.6 as they have relatively small slip distances, and only the blue and red atoms shown in Fig. 5.7 are also visible in Fig. 5.6.

Fig. 5.6. Slip configuration of system at a relatively large strain of 0.128. For W, only atoms with slip magnitude between 0.89 and 1.61 Å are shown.

Fig. 5.7. A projection of four consecutive $(\bar{1}10)$ planes in the circular region shown in Fig. 5.6. The top two planes (big blue and small red) have slip magnitude between 0.89 and 1.61. The dash circles indicate the “correct” sites if the red atoms did not slip.

Fig. 5.7 reveals several interesting phenomena: the symmetry of the planes is hexagonal; the ratio of the double plane spacing to in-plane atom spacing is about 1.633; and the three consecutive planes (gray, white, and blue) are stacked in an “ABC...” sequence. Note that the large slip distance calculated for the blue and the red atoms is due to the relative shift between the blue and red atoms and that no significant shift has occurred between other atoms. The stacking of the gray, white, and blue atoms is therefore representative of the majority of regions where shift is not significant. The observations made above are indicative that these regions have transformed to an fcc structure.

The dashed circles in Fig. 5.7 mark the positions where the red atoms should be for an fcc structure had slip not occurred between the blue and the red atoms. By comparing the position of the red atoms with dashed circles, we find that the red atoms have slipped in the $[\bar{1}\bar{1}0]$ direction in reference to the original bcc structure. Note that the Schmid factor for the critical resolved shear stress upon the $(\bar{1}10)$ plane is the highest in the observed $[\bar{1}10]$ slip direction with a value of 0.5. The Schmid factor is reduced to 0.408 in the bcc close-packed direction $[111]$. By accounting for the phase change and converting to the crystallographic orientations of the fcc structure, the slip plane is seen to essentially be $\{11\}$, and the Burgers vector of the slip is seen to correspond to $(1/6)\langle 11\bar{2} \rangle$ partial dislocations. These observations are related to the Bain relation shown in Fig. 5.8, where

we can see that the bcc lattice can be represented as a face-centered-tetragonal (fct) lattice that can transform to an fcc lattice during a uniaxial extension in the y- direction. In particular, we can see that the bcc $(\bar{1}10)$ plane is essentially a (transformed) fcc $\{111\}$ plane, and the bcc $[\bar{1}\bar{1}0]$ direction is essentially an fcc $\langle 11\bar{2} \rangle$ direction. The observed behavior therefore agrees with the $\frac{1}{6}\langle 11\bar{2} \rangle \{111\}$ slip systems being commonly encountered in the fcc crystals.

Fig. 5.8. bcc to fcc transformation (Bain) mechanism.

The finding obtained in the present work suggests an interesting deformation mechanism that may not have been known previously. When a bcc material is subject to a high strain, the lattice may be elastically distorted towards an fcc lattice. The $\frac{1}{6}\langle 11\bar{2} \rangle \{111\}$ types of fcc slip systems can then be activated and contribute to the plastic deformation. When the stress is removed, the lattice can elastically recover to bcc. However, dislocations created during the slip are retained. These dislocations in the bcc lattice have apparent slip planes of $\{110\}$ and apparent slip direction $\langle 1\bar{1}0 \rangle$. Interestingly, such slip systems have been observed [90].

The present work is based on a few assumptions: (a) the system prior to the loading is dislocation free; (b) the model element W is characterized by a bcc $\rightarrow$ fcc energy increase of 0.16 eV/atom; (c) the top and bottom layers of the simulated system are treated as rigid, which prevents dislocations from sweeping through; and (d) the system size is limited. Under these conditions, interesting phenomena such as the formation of slipped pipes and a bcc $\rightarrow$ fcc transformation assisting further plastic deformation were observed. While further studies are needed to understand the effects of boundary conditions,

loading algorithms, and pre-existing defect sources, it is particularly interesting to explore how the phase transformation deformation mechanism changes if one varies the energy difference between the bcc and fcc phases of the model element.

5.4 Conclusions

MD simulations have been carried out to study the delamination of a model W film from a Si substrate under tensile loading. The system was found to undergo elastic deformation, followed by extensive yielding behavior before finally fracturing at the interface. As the crack propagates during failure, the exposed Si exhibits wavy features indicative of compliant behavior as the fractured regions attempt to relax to the unconstrained positions, but are constrained by the unfractured regions that remain adhered to the film.

The yielding is accompanied by dislocation emission from the interface to the inside of the film, whereas no dislocations were observed inside Si. As expected in bcc systems, $\frac{1}{12}\langle 11\bar{1} \rangle \{ \bar{1}21 \}$ types of leading partial dislocations are seen to sweep through the crystal at the early stage of the loading, leaving behind slipped planes extending all the way to the interface. At late stages of the loading, however, the bcc film is seen to transform to an fcc lattice, and the leading partial dislocations become the $\frac{1}{6}\langle 11\bar{2} \rangle \{ 111 \}$ (reference to fcc lattice) types commonly seen in fcc systems. These dislocations in the bcc lattice have apparent slip planes of $\{110\}$ and apparent slip direction $\langle 1\bar{1}0 \rangle$.

6. References

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7. Workshops

J. A. Emerson

In order to get a broader perspective of our work in the context of the academic and industrial needs and overview of the activities outside Sandia, several workshops were organized. We progressed from small workshops totally run by Sandians to larger national/international organizational structures. Over the duration of the project we held four workshops.

The Macroelectronics Workshop was held on August 7, 2007 at Greater Albuquerque Chamber of Commerce. Seventeen were present from several institutions – Cornell, Columbia, Washington State, Clemson, University of Minnesota, University of New Mexico, and Sandia. Seven made presentations ranging from “printing organic circuits with ink jet printers”, “simulation of nanoindentation of viscoelastic substrate systems” to presentations on interfacial mechanics of thin films. Equal time was divided between presenting and discussion.

The next Macroelectronics Workshop was held before the Spring MRS Meeting on March 24, 2008 in the afternoon at the San Francisco Marriott. We intended this to be a small working group (by invitation) to discuss our current thoughts in the area of macroelectronics and flexible electronics. Eight were present from several institutions – Washington State, Clemson, University of Leoben, Stanford, Johns Hopkins University, and Sandia. This was an opportunity to present about 10-15 minutes what the attendees’ thoughts or what is bothering them. The motivation is to keep the working community informed and provide direction to a forth coming major workshop in October.

The next step is for a larger activity evolving several organizing groups. Thus, First Flexible Electronics Workshop, hosted by The Center for Advanced Microelectronics Manufacturing (CAMM), in conjunction with Sandia National Laboratories, and Cornell University was held on October 2007. Over 65 participants attended. Large number attendees were from industry – Corning, GE, Endicott Interconnect, Kodak, and United States Display Consortium. A tour of the Center for Advanced Microelectronics Manufacturing (CAMM) in Endicott, NY was included. It was decided this workshop that IEEE Society Components, Packaging, and Manufacturing Technology (CPMT) would organize the series of workshops

Second Flexible Electronics Workshop similar to the first, hosted by The Center for Advanced Microelectronics Manufacturing (CAMM), in conjunction with Sandia National Laboratories, Cornell University, and the IEEE CPMT, on August 19, 2009 in Binghamton, New York. This year’s symposium will be held in conjunction with the Flex Tech Alliance’s Quarterly Flexible, Printed and Electronics Workshop. Leading researchers from academia, national labs, and industry in the fields of flexible electronics, functional printing, and emerging electronic materials reviewed and shared new research findings in critical technology areas and identified issues for the rapidly growing flexible electronics field. As part of the workshop, attendees toured the CAMM, a national research lab focusing on roll-to-roll flexible electronics.

8. Publications and Presentations

Publications

Journal

P. McNabb, J. Yeager, D. Bahr, M. Kennedy, Interfacial Fracture Testing of Thin Films on Compliant Substrates, Scripta Materialia (2008) in preparation.

L. M. Hale, X. W. Zhou, J. A. Zimmerman, N. R. Moody, R. Ballarini, and W. W. Gerberich, Molecular dynamics simulation of delamination of an elastically-hard, body-centered-cubic crystalline film from a Si substrate” J. Applied Physics, JR09-3440, accepted for publication

Thao D. Nguyen, J. Yeager, D. F. Bahr, D. P. Adams, N. R. Moody, Nanoindentation of Compliant Substrate Systems: Effects of Geometry and Compliance, Journal of Engineering Materials and Technology, accepted for publication

X. W. Zhou, N. R. Moody, R. E. Jones, J. A. Zimmerman, E. D. Reedy, Molecular-dynamics-based Cohesive Zone Law for Brittle Interfacial Fracture under Mixed Loading Conditions: Effects of Elastic Constant Mismatch, Acta Materialia, accepted for publication

Conference Proceedings

X. W. Zhou, N. R. Moody, R. E. Jones, J. A. Zimmerman, E. D. Reedy, Molecular Dynamics Study of Interfacial Cohesive Zone Law: Elastic Constant Effects, ICF12

E. D. Reedy, Jr. , D. P. Adams, N. R. Moody, M. J. Cordill, M. S. Kennedy, D. F. Bahr, Effect of Substrate Compliance and Yielding on Thin Film Delamination, Proceedings 32nd Annual Meeting of The Adhesion Society, 2/15-18/2009, Savannah, GA, pp 121-123.

Presentations

D.F. Bahr, J.D. Yeager, J.A. Youngsman, M.S. Kennedy, N.R. Moody, Interfacial fracture and failure in polymer - metal systems, Eighth International Conference on Fundamentals of Fracture (ICFF VIII), Hong Kong, January 4 (2008)

N. R. Moody, M. J. Cordill, M. S. Kennedy, T. D. Nguyen, D. P. Adams, J. A. Emerson, D. F. Bahr, E. D. Reedy, Jr., Effects of Substrate Compliance on Interfacial Fracture of Thin Hard Films, Symposium on Materials and Technology for Flexible, Conformable, and Stretchable Sensors and Transistors, 2008 MRS Spring Meeting, San Francisco, CA March 24-28, 2008.

T. D. Nguyen, N. R. Moody, Modeling the Indentation of Stiff Film-Compliant Substrate Systems, Symposium on Materials and Technology for Flexible, Conformable, and Stretchable Sensors and Transistors, MRS Spring Meeting, San Francisco, March 24-28, 2008.

M. Kennedy, R. McNabb, I. Rook, V. Klep, J. Yeager, D.F. Bahr, I. Luzinov, N.R. Moody, Alteration of Substrate Compliance and Effects on Deformation and Fracture of Thin Metallic Films, Symposium on Materials and Technology for Flexible, Conformable, and Stretchable Sensors and Transistors, MRS Spring Meeting, San Francisco, March 24-28, 2008.

N. R. Moody, M. J. Cordill, M. S. Kennedy, T. D. Nguyen, D. P. Adams, J. A. Emerson, D. F. Bahr, E. D. Reedy, Jr., Effects of Substrate Compliance on Delamination of Thin Hard Films, Symposium on Mechanical Instabilities in Polymer Films (Invited), Interfaces and Nanostructures, ACS Annual Meeting, New Orleans, LA, April 6-10, 2008

T. D. Nguyen, N. R. Moody, Modeling the Indentation of Stiff Film-Compliant Substrate Systems--Effects of Geometry and Film and Substrate Elasticity, Materials Science and Engineering Department Seminar, Johns Hopkins University, April 11, 2008.

N. R. Moody, M. J. Cordill, M. S. Kennedy, T. D. Nguyen, D. P. Adams, J. A. Emerson, D. F. Bahr, E. D. Reedy, Jr., Failure of Hard Nanoscale Films on Compliant Substrates (Invited), International Conference from Nanoparticles and Nanomaterials to Nanodevices and Nanosystems, Halkidiki, Greece, June 16-18, 2008.

N. R. Moody, M. S. Kennedy, M. J. Cordill, T. D. Nguyen, D. P. Adams, J. A. Emerson, D. F. Bahr, E. D. Reedy, Jr., Nanomechanics of Film Failure on Compliant Substrates (Invited), Hysitron Inc., Minneapolis, MN, October 20, 2008.

N. R. Moody, M. J. Cordill, M. S. Kennedy, T. D. Nguyen, D. P. Adams, J. A. Emerson, D. F. Bahr, E. D. Reedy, Jr., Substrate Deformation Effects on Fracture of Thin Hard Films on Compliant Substrates (Invited), 2008 ASME International Mechanical Engineering Congress and Exposition, Boston MA, October 31-November 6, 2008

N. R. Moody, M. J. Cordill, M. S. Kennedy, D. P. Adams, J. A. Emerson, D. F. Bahr, E. D. Reedy, Jr., Localized Plasticity Effects on the Delamination of Thin Hard films on Compliant Substrates, Symposium on Reliability and Properties of Electronic Devices on Flexible Substrates, MRS Fall Meeting, Boston, MA, December 1-5, 2008

M.S. Kennedy, N.R. Moody, D.C. Hurley, Investigation of Metal-Polymer Interfacial Cracking Using Contact Resonance Atomic Force Microscopy, Symposium on Reliability and Properties of Electronic Devices on Flexible Substrates, MRS Fall Meeting, Boston, MA, December 1-5, 2008

N. R. Moody, M. J. Cordill, M. S. Kennedy, T. D. Nguyen, D. P. Adams, J. A. Emerson, D. F. Bahr, E. D. Reedy, Jr., Substrate Deformation Effects on Delamination of Thin Hard Films (Invited), 15th International Symposium on Plasticity 2009, Frenchman's Reef and Morning Star Marriott Beach Resort, St. Thomas, Virgin Islands, January 2-9, 2009.

E. D. Reedy, Jr. , N. R. Moody, Effect of Substrate Compliance and Yielding on Thin Film Delamination, 2009 Annual Meeting of the Adhesion Society, Savannah GA, February 16-20, 2009.

N. R. Moody, M. S. Kennedy, M. J. Cordill, D. P. Adams, J. A. Emerson, D. F. Bahr, E. D. Reedy, Jr., Interfacial fracture of thin hard films on compliant substrates-test and simulation, Symposium on Mechanical Properties and Adhesion, ICMCTF, April 27-May 1, 2009, San Diego, CA.

N. R. Moody, J. Yeager, D. F. Bahr, M. J. Cordill, M. S. Kennedy, D. P. Adams, E. D. Reedy, Jr., Interfacial Fracture in Compliant Substrate Film Systems, Symposium on Fracture in MEMS, NEMS, and Small Volume Devices, ICF12, Ottawa, Ontario, Canada, July 12-17, 2009

N. R. Moody, E. D. Reedy, E. Corona, D. P. Adams, J. Yeager, M. S. Kennedy, M. J. Cordill, D. F. Bahr, Buckle Driven Delamination in Thin Film Compliant Substrate Systems-Tests and Simulations (Invited), Nanomechanical Testing in Materials Research and Development, Il Ciocco Hotel and Conference Center Barga (Tuscany), Italy, October 11-16, 2009.

N. R. Moody, E. D. Reedy, E. Corona, D. P. Adams, J. Yeager, M. S. Kennedy, M. J. Cordill, D. F. Bahr, Buckle Driven Delamination in Thin Film Compliant Substrate Systems-Tests and Simulations (Invited), Symposium on Mechanics of Integrated Material Structures in Advanced and Emerging Technologies, 2009 ASME International Mechanical Engineering Congress and Exposition, Lake Buena Vista, FL, November 13-19, 2009.

N. R. Moody, E. D. Reedy, Jr., E. Corona, M. S. Kennedy, M. J. Cordill, T. D. Nguyen, D. P. Adams, D. F. Bahr, Effects of Substrate Compliance and Yielding on Thin Film Delamination-Tests and Simulations, Symposium on Mechanical Behavior of Nanomaterials-Experiments and Modeling, 2009 MRS Fall Meeting, Boston MA, November 30-December 4, 2009

**Presentations at the Macroelectronic (Compliant Substrate)
Workshop, August 6-7, 2007, Albuquerque, NM:**

N. R. Moody, E. D. Reedy, Jr., T. D. Nguyen, D. P. Adams, A. A. Talin, J. A. Emerson, M. J. Cordill, M. S. Kennedy, D. F. Bahr, Nanomechanics of Films on Compliant Substrates to Enable New Flexible MEMS and NEMS Devices-Program Overview, Workshop on Macroelectronics.

N. R. Moody, E. D. Reedy, Jr., T. D. Nguyen, D. P. Adams, A. A. Talin, J. A. Emerson, M. J. Cordill, M. S. Kennedy, D. F. Bahr, Effects of Substrate Compliance on Deformation and Fracture of Thin Hard Films.

M. J. Cordill, Deformation and fracture of thin film buckles on compliant substrates.

E. D. Reedy, Jr., Simulating interfacial crack growth of an elastic film on a compliant elastic substrate under residual tensile stresses

T. D. Nguyen, Simulation of nanoindentation of compliant viscoelastic

D. F. Bahr, Deformation and fracture of gold films on Kapton

**Presentations at the Macroelectronic (Compliant Substrate)
Workshop, March 24, 2008, San Francisco, CA:**

N. R. Moody, M. S. Kennedy, M. J. Cordill, D. P. Adams, A. A. Talin, J. A. Emerson, D. F. Bahr, Nanomechanics of Thin Films on Compliant Substrates.

T. D. Nguyen, N. R. Moody, Modeling the Indentation of Hard Film-Compliant Substrate Systems.

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