



Adhesion of polymer thin-films and patterned lines

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Abstract. The adhesion of interfaces in thin-film structures containing ductile polymer blanket films and patterned lines is reported. The intent of the study was to demonstrate that both the film thickness and the aspect ratio of patterned lines have a significant effect on the interfacial fracture energy of interfaces adjacent to the ductile polymer. In particular, there is currently limited understanding of the effect of dimensional constraint in the plane of the film on local plasticity and associated interfacial fracture energies. Accordingly, the interfacial adhesion of patterned structures containing arrays of polymer/SiO₂ lines with varying aspect ratios was investigated. Macroscopic adhesion values were determined by measuring the critical strain energy release rate, G_c , for debonding of the patterned interface. The yield properties of the polymer films as a function of film thickness was also investigated. Decreasing aspect ratio of the polymer lines was found to significantly increase interface fracture energy and is rationalized in terms of the effect of stress state on the extent of plastic deformation in the polymer line.

1. Introduction

Adhesion of thin-film interconnect and device structures is a pervasive problem in the manufacture of complex microelectronic devices, and often significantly limits the integration of new materials (Lane et al., 2000a; Dauskardt et al., 1998; Ma et al., 1997; Volinsky et al., 2002; Maidenberg et al., 2002). Similarly, resistance to subcritical debonding associated with moisture exposure during CMP and other processing operations is of major technological concern (Hohlfelder et al., 2001; Ritter et al., 1998; Kook and Dauskardt, 2002; Lane et al., 1999). In the present study, these challenges are addressed with specific regard to understanding the effect of patterned device structures as opposed to blanket films. The intention is to develop a more fundamental understanding of the adhesive and cohesive fracture properties of interconnect structures with regard to their feature size and aspect ratio. Such an understanding may ultimately form the basis for developing technologically relevant *design guidelines* that govern the reliability of patterned thin-film structures. The basic hypothesis that we wish to investigate is that the feature size and geometry of ductile materials may affect the adhesive properties of adjacent interfaces.

Recent studies of the adhesion of blanket thin-film interconnect structures have established that the fracture resistance, G_c , of the typically weak dielectric/barrier interface (e.g. SiO₂/TaN) may be significantly increased through plastic energy dissipation in thin Al-Cu or Cu films present in the structure (Dauskardt et al., 1997; Lane et al., 2000b). The metal film is generally separated from the debonding interface by a thin elastic barrier layer. In the case of Cu films, the yield stress, σ_{ys} , and the associated plastic energy contribution to the interface fracture resistance, G_{pl} , have been shown to be strongly dependent on the thickness of the ductile Cu layer (Lane et al., 2000b; Wei and Hutchinson, 1997; Tymiak et al., 2000).

Other salient parameters of blanket thin-film structures containing metal films that markedly effect G_{pl} include the inherent fracture resistance, G_0 , of the interface (or ‘work-of-adhesion’) and the elastic layer thickness (Lane et al., 2000b; Wei and Hutchinson, 1997; Tymiak et al., 2000; Evans et al., 1999). More recent multi-scale simulations of interconnect adhesion have revealed that the residual stress state in the Cu layer may also effect the plasticity contribution (Strohband and Dauskardt, 2002).

Microelectronic devices, however, do not generally employ blanket thin films, but rather utilize complex wiring interconnections where metal lines have lateral dimension comparable to their thickness. The lines are typically encased with a thin (< 100 nm) elastic diffusion barrier layer and supported mechanically by adjacent dielectric materials. Traditional dielectric materials are SiO₂ glasses, which are brittle and behave elastically. However, several new classes of organic materials, including spin-on polymers, have been developed to replace SiO₂. These polymers behave in a ductile fashion, allowing plastic energy dissipation during debonding similar to metal layers. The basic premise of the present study is that the aspect ratio of lines made of ductile materials will effect the elastic constraint and stress state in the line, and hence change the plasticity contribution to the fracture resistance of interfaces that are close to the line. In the case of bulk metals and polymers, the effect of stress state and specimen thickness on fracture resistance is well known (Kinloch and Young, 1983; Knott, 1971; Irwin, 1960). However, these studies have generally been conducted on large fracture specimens compared to the length scales relevant to microelectronic devices. Indeed, there are currently no reported studies that elucidate the role of the size and geometry of features patterned in ductile materials on plastic energy dissipation and associated interfacial fracture resistance in device structures.

The intent of the present study was to examine the adhesion or interface fracture resistance of structures containing arrays of polymer lines with varying aspect ratio. Macroscopic adhesion values were determined by measuring the critical strain energy release rate, G_c , for debonding of a selected interface adjacent to a polymer line. Similar to behavior observed in thin Cu films (Lane et al., 2000b; Wei and Hutchinson, 1997; Tymiak et al., 2000), plastic energy dissipation in the thin polymer layer was initially demonstrated by measuring the interface fracture resistance as a function of the blanket polymer film thickness. Nanoindentation techniques were used to measure the yield strength of each polymer film.

Subsequently, the effect of patterned polymer lines with aspect ratios varying from 2 to 12 on interfacial adhesion was investigated and compared to values measured using blanket films. The aspect ratio of the line was found to have a pronounced effect on the fracture resistance of the interface adjacent to the line. Adhesion values were increased by more than 50% for lines with the smallest aspect ratio compared to large aspect ratio lines that were wide with respect to their thickness. These results clearly support the hypothesis that macroscopic adhesion values of interfaces in interconnect structures may be significantly influenced by the geometry and size of ductile features adjacent to, or near to, the interface of interest. The study also suggests that the extent of plasticity can be manipulated in patterned structures by constraining the dimensions of the ductile layer in either one or two dimensions.

2. Experimental procedures

2.1. SPECIMEN PREPARATION

2.1.1. Blanket polymer films

The ductile polymer selected for study was a modified poly(arylene)ether, PAE (SiLK™, Dow Chemical, Midland, MI) that is being considered as a low dielectric constant interlayer material. Using standard deposition and curing techniques, polymer layers with a thickness of 0.25 μm , 0.5 μm , 1.0 μm , 2.12 μm and 5.25 μm were spun onto 200 mm diameter Si wafers (Townsend et al., 1997). A 0.2 μm thick blanket film of SiO₂ was then deposited on top of the polymer by low temperature PECVD. In order to manipulate the interface chemistry, one group of substrates was exposed to an UV/ozone clean prior to the SiO₂ deposition, while the control group remained untreated. Another set of specimens containing pure poly(arylene)ether polymer layers with thicknesses of 50 nm and 100 nm were fabricated from a similar ductile polymer material (FLARE™, Honeywell Corporation, Santa Clara, CA) to assess the effect of even thinner polymer layers. These samples were deposited onto wafers previously coated with 50 nm of SiN and then capped with an additional 500 nm of SiN.

To produce specimens for four-point bending adhesion measurements, 35 mm square samples were cleaved from the wafers and the top of the thin-film structure was bonded to a similar sized bare Si substrate using a strong Cu diffusion bond as previously described (Dauskardt and Lane, 1998). Individual four-point bend specimens were machined from the sandwiched wafers using a high speed dicing saw with a diamond tipped blade such that dimensions of each beam were 3 mm wide and 35 mm long with a central notch machined to within $\sim 100 \mu\text{m}$ of the thin film interface.

2.1.2. Patterned polymer lines

Blanket films of 1.0 μm modified PAE (SiLK™) followed by 0.25 μm SiO₂ were deposited on Si substrates to fabricate simple patterned line structures to investigate the effect of line aspect ratio on interfacial adhesion. Standard lithographic techniques were employed to expose and develop 1.0 μm of photoresist deposited over the SiO₂ using a vacuum contact exposure mask that was fabricated to contain the selected line geometries. A CHF₃/CF₄ reactive ion etch chemistry was used to transfer the patterns to the SiO₂ layer. Using the oxide as a hard mask, the etch chemistry was changed to an O₂/N₂ plasma to pattern the polymer and clear the remaining photoresist. The final structures consisted of five different arrays of polymer/SiO₂ lines processed on the same wafer. Each array was 25 \times 35 mm in size and contained lines with an identical width. Lines widths of 12 μm , 6 μm , 4 μm , 3 μm and 2 μm were fabricated. As observed in Figure 1, the width of the trench in between each line was fixed at 1.5 μm . In order to provide a baseline for blanket film adhesion, another region on the wafer was not patterned.

The structures were sandwiched between two silicon wafers by diffusion bonding a similar thickness Si wafer on top of the structure using the Cu diffusion bonding method described above. The gaps in between the polymer/SiO₂ lines remained open except for a thin Cr/Cu layer less than 220 nm thick on the sidewalls that resulted from the Cu diffusion bonding process. Four-point adhesion test specimens were diced from the sandwiched wafers. The features were orientated along the length of the adhesion test specimen so that the debond front was perpendicular to the features and the debond propagated in the direction of the patterned lines.

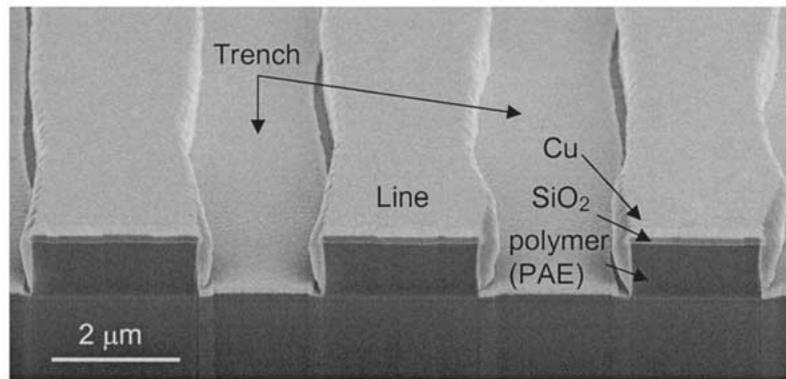


Figure 1. SEM image of a FIB sectioned polymer/SiO₂ patterned structure after blanket Cu deposition in preparation for diffusion bonding. Features with aspect ratios (width/height) of 2, 3, 4, 6 and 12 were fabricated with a 1.5 μm trench separating each line.

2.2. NANOINDENTATION

The hardness and yield stress of the selected polymer films were investigated using nanoindentation techniques. Measurements were made with a nanoindenter (XP, MTS, Minneapolis, MN) fitted with a dynamic control module (DCM) head and a Berkovich indenter using the continuous stiffness measurement (CSM) technique (Oliver and Pharr, 1992). The CSM technique, used in conjunction with the DCM head, is particularly useful for testing of compliant materials since it allows detection of initial surface contact with the indenter. Quasi-constant strain-rate experiments were performed, using a load-rate/load equal to 0.05 s⁻¹.

2.3. ADHESION TESTING

Thin-film adhesion testing was conducted under mixed mode loading conditions using the four-point bend technique (Dauskardt et al., 1998; Ma et al., 1995). Specimens were loaded under displacement control in an articulating four-point bending fixture. The four-point bend adhesion measurements were conducted with a crosshead displacement rate of 0.3 μm s⁻¹. Interfacial debonds from five different beams were propagated along the selected blanket or patterned interface for each experimental condition. Multiple measurements of adhesion were made on each beam by changing the position of the loading pins. Following adhesion measurements, the resulting fracture surfaces were characterized by x-ray photoelectron spectroscopy (XPS), atomic force microscopy (AFM), and optical microscopy to determine the location of the debond path. For the blanket film specimens, debonding occurred at the polymer/barrier layer interface. In the patterned line structures, debonding occurred near the patterned polymer/SiO₂ interface along the length of the line.

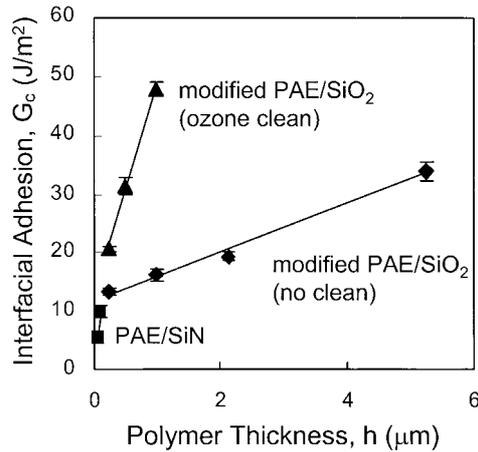


Figure 2. Polymer/barrier interfacial adhesion as a function of film thickness. The extent of plasticity in the polymer layer is related to the thickness of the layer and the intrinsic fracture resistance, G_o , of the interface.

3. Results and discussion

3.1. BLANKET POLYMER FILMS

Blanket film adhesion values for the polymer/barrier interface as a function of the polymer layer thickness are presented in Figure 2. A significant increase in the measured macroscopic adhesion with increasing polymer layer thickness for the three selected polymer/barrier interfaces is clearly apparent. The adhesion of the thinnest PAE polymer film (50 nm) to the adjacent SiN barrier was $\sim 5 \text{ J m}^{-2}$ which increased to $\sim 10 \text{ J m}^{-2}$ for the 100 nm film thickness. The lowest measured adhesion for the thinnest (0.25 μm) modified PAE layer to the adjacent SiO₂ layer was $\sim 12 \text{ J m}^{-2}$. This fracture energy is larger than the bulk fracture resistance of SiO₂ which is generally considered to be $\sim 5\text{--}8 \text{ J m}^{-2}$ (Ritter et al., 1998; Lucas et al., 1995). This suggests that there is a plastic energy contribution, G_{pl} , to the total interfacial energy, G_c , for even the thinnest modified PAE polymer films.

As reported for debonding in other thin-film structures containing ductile layers, G_{pl} increases with the thickness of the ductile layer (Lane et al., 1999, 2000a, b; Volinsky et al., 2002; Wei and Hutchinson, 1997). For the present polymers, simple estimates suggest that the plastic zone that would form in a bulk polymer specimen would be larger than the polymer layer thickness for similar values of G_c . The effect of the elastic constraint provided by the adjacent elastic layers has been shown to locally increase the debond tip stress fields and hence the size of the plastic zone ahead of the debond tip (Varias et al., 1991). Using this analysis, the plastic zone in the present modified PAE polymer films was estimated to be 10 μm for G_c values of 5 J m^{-2} . Therefore, even at relatively low values of applied G , the plastic zone is expected to fully engulf the polymer layer. These estimates are similar to those reported for a thin polymer film with similar adhesive and yield properties (Snodgrass et al., 2002).

Assuming that the ductile film thickness is fully engulfed by the plastic zone, G_c has been shown to scale with the ductile film thickness, h (Strohband and Dauskardt, 2002). This behavior was generally observed for the present specimens where macroscopic adhesion values, $G_c = G_o + G_{pl}$, increased linearly with polymer layer thickness as shown in Figure 2. A simple strength of materials model for the fracture resistance of an interface adjacent to an elastic-perfectly plastic ductile layer, suggests that G_{pl} should scale with $h \ln(h)$ (Volinsky

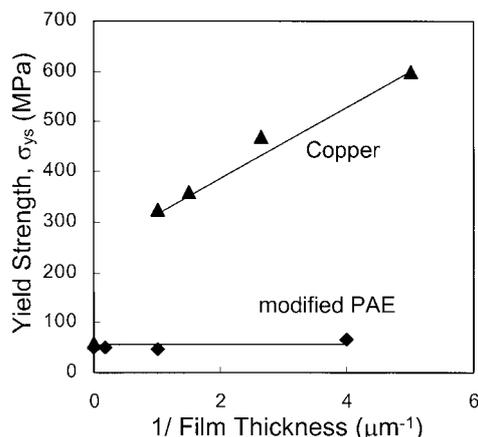


Figure 3. Yield stress of modified PAE (SiLKTM) and Cu (Vinci et al., 1995) films as a function of film thickness. The modified PAE polymer films are not susceptible to strengthening mechanisms that operate on thin metal films.

et al., 1999). One of the many simplifying assumptions of this model is that the polymer yield stress is independent of the film thickness. This was validated for the present polymer films using nanoindentation measurements. These were employed to estimate the yield strength of the polymer films as shown in Figure 3. Included in the figure for comparison are similar results reported for thin Cu films (Vinci et al., 1995).

While the polymer yield stress is constant at ~ 55 MPa and independent of film thickness, the initial flow stress for the Cu films is markedly increased with decreasing film thickness (Figure 3). For the Cu films, dislocation strengthening mechanisms are sensitive to the metal film thickness and generally inhibit plasticity contributions to interface fracture resistance for very thin ($< 0.25 \mu\text{m}$) films (Lane et al., 2002b; Wei and Hutchinson, 1997; Tymiak et al., 2000). However, in the case of the present polymer, plastic deformation is not sensitive to film thickness and plastic flow is consequently not inhibited. The effect of the polymer film thickness on adhesion is therefore primarily to physically restrict the size of the plastic zone to the layer thickness, rather than by inhibiting plastic flow through an increasing flow stress with decreasing film thickness. The effect was also found to persist to much smaller length scales compared to those reported for metals (Lane et al., 2000b; Wei and Hutchinson, 1997).

Adhesion of the polymer/SiO₂ interface was observed to markedly increase for interfaces formed after exposing the polymer to the UV/ozone clean before deposition of the SiO₂ layer (Figure 2). The UV/ozone clean removes residual surface contamination and functionalizes the polymer. These mechanisms are expected to increase the intrinsic fracture resistance, G_o , of the interface. Mechanics models for debonding of an interface directly adjacent to a ductile layer suggest that G_{pl} contributions from plastic energy dissipation in the ductile layer to G_c will be very sensitive to G_o (Wei and Hutchinson, 1997; Evans et al., 1999). This is consistent with the behavior observed in the present study and suggests that small improvements in G_o for an interface directly adjacent to ductile layer can result in large increases in G_c .

3.2. PATTERNED POLYMER LINES

The effect of the polymer line width on the interfacial fracture resistance of the polymer/SiO₂ interface is shown in Figure 4. With decreasing line width, a significant increase in the value of G_c was observed. The interfacial adhesion of polymer lines with an aspect ratio of 2 was found

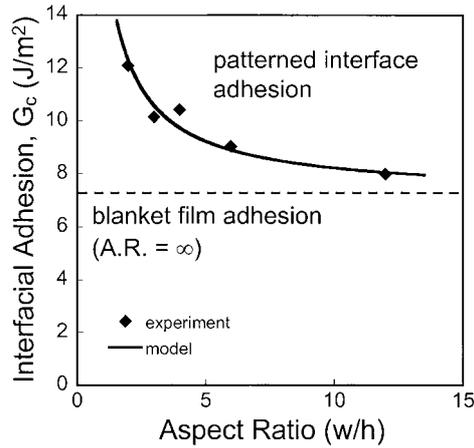


Figure 4. Polymer/SiO₂ adhesion as a function of feature aspect ratio. Increasing plastic contribution to G_c with decreasing aspect ratio was observed with a transition from plane strain to plane stress fracture.

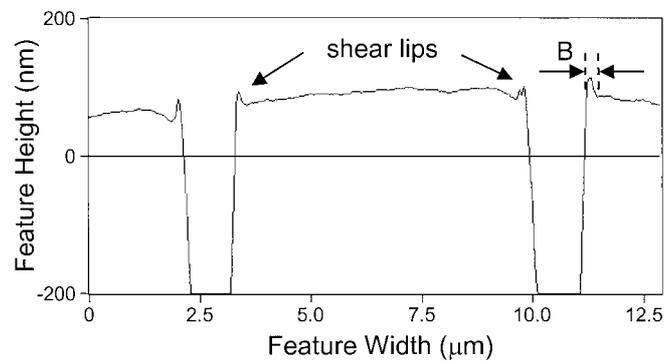


Figure 5. Tapping mode AFM cross-section revealed shear lips near the fracture edges of the polymer lines indicating a transition to plane stress.

to be $\sim 50\%$ larger than similar polymer/SiO₂ interfaces formed between blanket films. The increase in adhesion is associated with increasing contributions from plastic energy dissipation in the patterned lines. Due to decreasing lateral constraint of the lines with decreasing line width, the stress state in the polymer line becomes increasingly less triaxial. As a result, the polymer yields more readily as the stress state changes from plane strain, in the case of wide lines or the blanket film, to one of plane stress for the narrow lines. The observed fracture resistance behavior is similar to the transition from plane strain to plane stress fracture commonly found in bulk metals and polymers; however, the length scales involved in the present study are significantly smaller. For example, in the case of a bulk polymer with similar mechanical properties, the transition from plane strain to plane stress fracture is expected to occur at approximately $15 r_y \sim 1$ mm, where r_y is the plastic zone size (Braun and Srawley (1966)). The present transition is observed at length scales that are three orders of magnitude lower.

Several characterization techniques were used to identify the location of the debonded interface for the patterned specimens. Compositional analysis using XPS revealed characteristic high-energy satellites peaks in the C (1s) photoelectron spectrum on one side of the debond path. These $\pi - \pi^*$ shakeup transitions are commonly used to identify modified PAE and are consistent with the aromatic nature of such polymers (Rajagopal et al., 1999).

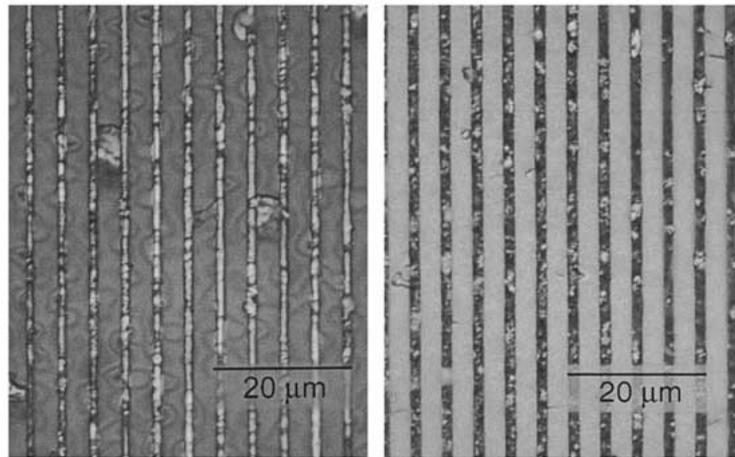


Figure 6. Optical microscopy of mating fracture surfaces from a specimens with 3 μm line widths. Images indicate the fracture path was located near the patterned interface.

The stoichiometry of the mating fracture surfaces was equivalent to the PECVD SiO_2 barrier layer, signifying polymer/ SiO_2 delamination. Tapping mode AFM cross-sections indicated the presence of surface roughness features with dimensions similar to the original lithographical patterned structures (Figure 5). Careful observations of the regions adjacent to the side of the debonded lines revealed a slanted region. These regions appear to be indications of shear lips that are commonly associated with plane stress fracture in ductile materials. In agreement with AFM and XPS measurements, optical microscopy also revealed features with the appropriate patterned line dimensions on both sides of the fracture surface (Figure 6). All of the surface characterization results therefore indicated that the debond in these structures propagated along the patterned polymer/ SiO_2 interface.

A model developed to predict the plane stress fracture resistance of ductile materials that exhibit slanted fracture surfaces was employed to estimate the contribution to G_c from energy dissipated through the formation of shear lips. Assuming elastic/perfectly plastic conditions, the shear lips provided a contribution to the fracture energy of $G_{\text{slant}} = 2\sigma_{ys}B$, where B is the width of the slanted ligament and σ_{ys} is the yield strength (Cottrell, 1963). Tapping mode AFM profiles of the patterned structure fracture surface indicated that the width of the slanted region, B , is $\sim 0.25 \mu\text{m}$, as depicted in Figure 5, and independent of the polymer feature width. Utilizing the nanoindentation results for σ_{ys} , the energy dissipated along the slanted fracture surface, G_{slant} , was determined to be $\sim 27.5 \text{ J m}^{-2}$. The fracture resistance of the remaining flat fracture surface was assumed to be that of the blanket polymer film, G_{blanket} , which for the current polymer/ SiO_2 interface was $\sim 7.2 \text{ J m}^{-2}$. The fracture resistance for the entire line should therefore be equal to the contribution from the slanted and blanket regions and given by:

$$G_c = \frac{2B \cdot G_{\text{slant}} + (w - 2B) \cdot G_{\text{blanket}}}{w}, \quad (1)$$

where w is the width of the patterned feature. The model predictions for G_c are shown in comparison to the experimentally measured values in Figure 4. The model is clearly consistent with the measured values and suggests that a continuing increase in fracture resistance may occur with decreasing polymer line width.

Similar toughening effects have been reported in structural Al-Li alloys (Rao and Ritchie, 1990). Rolled forms of these materials contain pancake grains with weak grain boundaries. During fracture, the grain boundaries normal to the fracture plane delaminate, causing plane stress fracture of the individual grains. On the resulting fracture surface, multiple 45° shear lips adjacent to the grain boundary delaminations were reported. Such plane stress fracture of the individual grains leads to significant increases in the fracture resistance of the material compared to the same material tested in conditions that do not promote grain debonding. It is interesting that in these materials, improvements in fracture resistance can therefore be obtained by engineering a microstructure that promotes a local state of plane stress within the plastic zone, while maintaining a macroscopic state of plane strain. Similar strategies are clearly possible in the case of ductile line structures. Similar to the fracture surface of the Al-Li alloy, pronounced shear lips were clearly apparent near the unconstrained lateral edges of the lithographically defined lines. The increased macroscopic adhesion can therefore be attributed to the increased fraction of the debond surface containing these features.

The present model predicts a maximum fracture resistance when the polymer line is equal to $2B \sim 0.5 \mu\text{m}$. At this width, the entire line fracture surface would consist of a slant fracture. It is interesting to note that this width corresponds to one half of the film thickness where a complete transition to plane stress in the polymer line is expected. The maximum achievable fracture resistance at this line width should be a factor of three higher than the blanket adhesion value. Clearly, these results demonstrate that significant increases in interfacial fracture resistance may be obtained adjacent to ductile lines where the width of the line is less than ~ 5 times the line thickness (Figure 4). The potential for nanofabrication techniques to produce such transitions in fracture resistance in device structures that would otherwise be observed at length scales orders of magnitude larger in bulk materials is also apparent.

4. Conclusions

The contribution from plastic energy dissipation to the macroscopic interfacial fracture resistance was measured for polymer/SiO₂ interfaces adjacent to blanket polymer films and patterned polymer lines. Plastic energy dissipation in the polymer thin-films was found to dominate macroscopic adhesion values and could be modulated by controlling the dimensions of the ductile film in either one or two dimensions. Results from blanket polymer films indicated that contributions from plasticity dramatically increase the interfacial fracture resistance of the polymer/SiO₂ interface above that of the SiO₂ glass. The plastic energy contribution was found to scale with the polymer layer thickness and the intrinsic fracture resistance of the interface. Lithographically patterned structures were fabricated to measure the effects of varying aspect ratios on interfacial adhesion. The interfacial fracture energy for the polymer/SiO₂ interface was dependent on the aspect ratio of the patterned line, increasing by as much as 50% for the narrowest lines. The resultant fracture surfaces exhibited slanted fracture surfaces near the edges of the patterned lines, similar to classical shear lips observed during fracture of bulk ductile materials. An estimate for the fracture energy of the slanted regions together with the interfacial fracture energy of blanket polymer films was used to predict the measured interfacial adhesion values of the patterned lines as a function of line width. The model was consistent with the measured values and suggests that the reliability of the interfaces relevant to the microelectronic industry could be significantly improved by considering the effects of ductile feature size and associated plastic energy dissipation on interfacial fracture resistance.

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