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Effect of Nano-scale Patterned Interfacial Roughness on Interfacial Toughness

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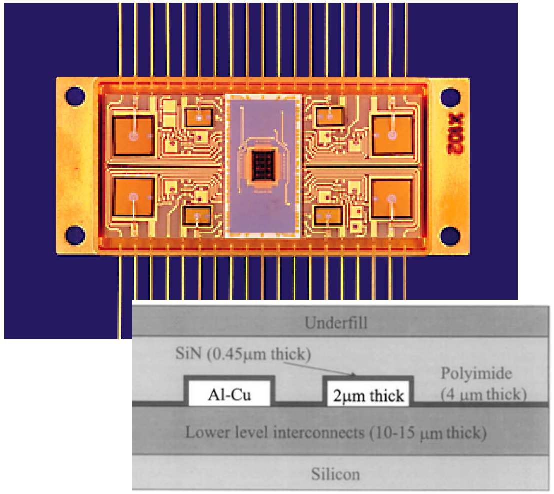
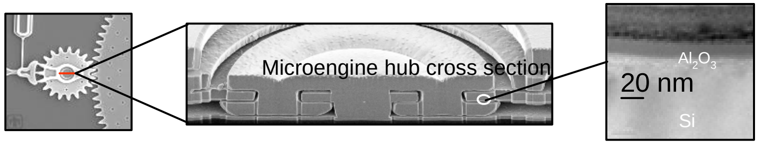
Problem

- The performance and the reliability of many devices are controlled by interfaces between thin, brittle films.
- The toughness of such interfaces is often quite low because there is little energy dissipation in the surrounding bulk materials.
- Need a method to engineer thin film interfaces with improved toughness to enhance thin film performance and reliability.



Thin films in microelectronics



Tribological coatings on MEMS

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Objectives and Approach

Aluminum adherends bonded together with an epoxy adhesive

(SAND2000-1042)

Surface Roughness (microns)	G_c (J/m^2)	Standard Deviation (J/m^2)
0.2	22	3
1.0	31	7
5.0	126	11
7.0	168	5

- Well known that surface roughness increases interfacial toughness.
- Mechanisms that can increase interfacial toughness include:
  - –increase in surface area
  - –mechanical interlock
  - –increased crack path tortuousity
  - –stick/slip crack growth
  - –mixed-mode crack-tip loading
  - –enhanced plastic dissipation

Objective:

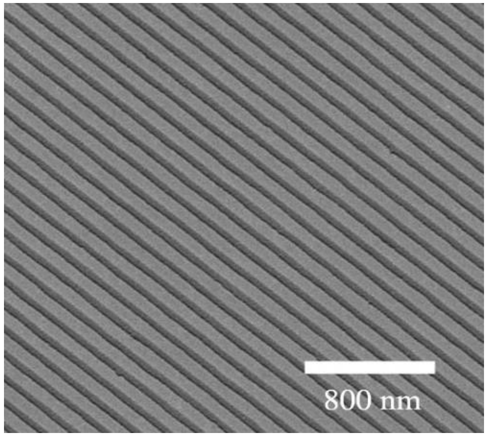
- Determine if patterned nm-scale interfacial roughness can be used to increase the interfacial toughness of brittle, thin-film material systems

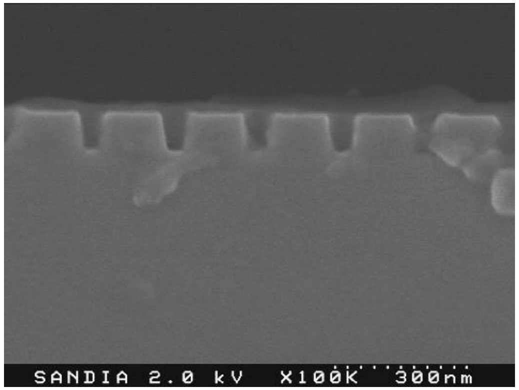
Approach:

- Well defined experiments to demonstrate how patterned nanoscale roughness affects the toughness of brittle thin-film material systems.
- Detailed finite element analysis to understand how crack growth along a nano-patterned interface between brittle, thin films dissipates energy and as a consequence affects apparent interfacial toughness.

Patterned interface experiments

- Using thermal nanoimprint lithography (NIL, Nanonex) to create well-formed, nano-scale patterns.
  - NIL allows rapid nano-patterning of a large area using a high precision mold.
- Initial work uses a 200-nm pitch, rectangular-wave pattern.
  - Master created using laser interference lithography.
  - Thermal NIL used to transfer pattern to the polymer resist.
  - Plasma etch (RIE) used to pattern the Si wafer.
- Sputter tungsten layer (~250 nm) on patterned Si wafer.
  - Deposited so to introduce high (~ 3 GPa) compressive stress.
  - Chose Si/W bimaterials to minimize the contribution of bulk yielding to measured toughness.





Nanoimprint lithography creates 200-nm rectangular-wave pattern with channels that are ~ 60 nm wide by 90 nm deep.

Methods to measure interfacial toughness

<u>Stressed Overlayer Test</u>

-Compressively stressed overlayers generate buckles.

-Measured buckle shapes can be combined with mechanic's models to infer interfacial toughness.

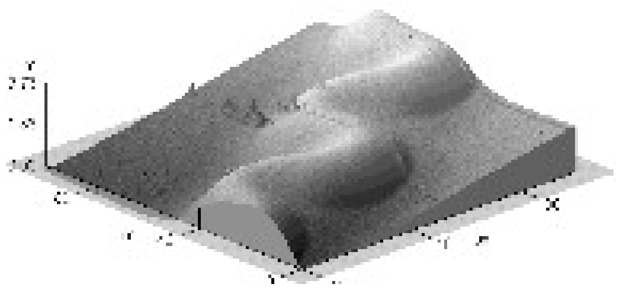
-Imposes a nominally mode II-like shear loading.

-Can perform many tests on a single, relatively small sample (10 mm square).

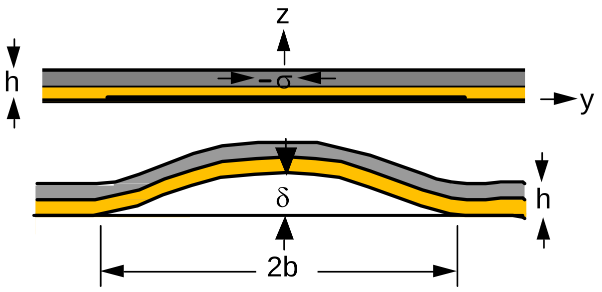
-Issues include need to iteratively 'tune in' the overlayer thickness to match interfacial toughness and generating crack propagation along the desired interface.

-On a patterned surface, buckles will tend to grow in direction of lowest toughness.

Compressive stress in W is are easily controlled during DC magnetron sputtering deposition.

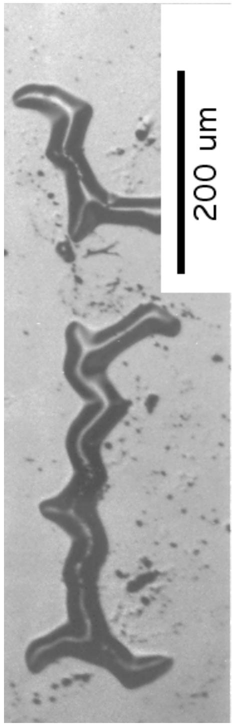


Generated telephone cord blisters.



Measured toughness of patterned interface ~50% higher

250nm W on smooth Si

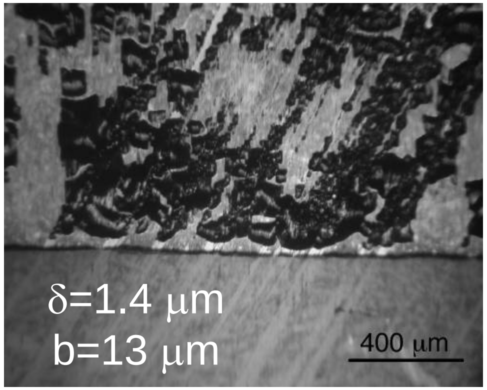


δ=1.4 μm   b=14 μm

Inferred Toughness of 2.7 ± 1.1 J/m^2 at a mode mixity of -90^o

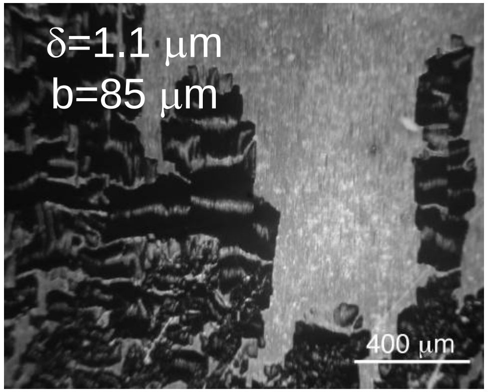
Telephone cord buckles

280nm W on patterned Si



Inferred Toughness of 4.3 ± 1.0 J/m^2 at a mode mixity of -90^o

Small telephone cord buckles formed by alternating segments running parallel and perpendicular to channels



Inferred Toughness of 4.1 ± x.0 J/m^2 at a mode mixity of -90^o

Large straight-sided buckles formed perpendicular to channel pattern.

Finite Element Simulations of Crack Growth

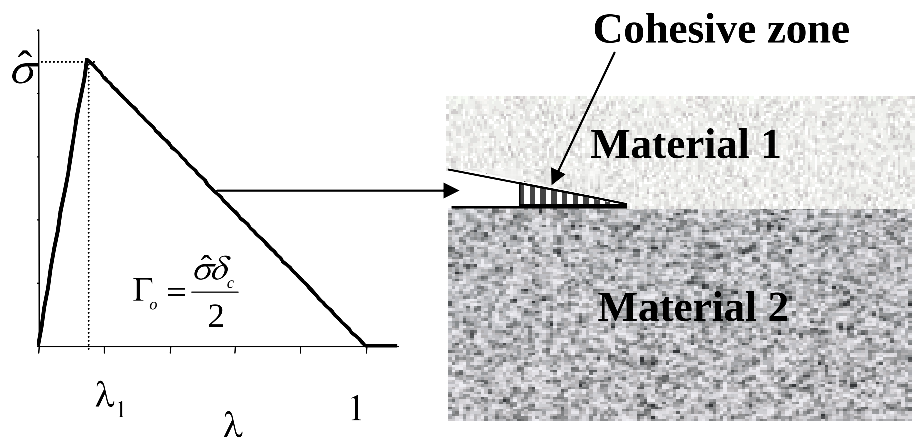
- Need to model subcritical crack growth.
- Crack growth may not be continuous or sequential --- portions of the interface might open up ahead of the main crack.
- Could have contact in regions a considerable distance behind an advancing the crack tip.
- Require accurate representation of the actual separation process.

Used an explicit, transient dynamics finite element code (PRESTO).

- Well suited for analyzing discontinuous processes with complex contact conditions.
- Often referred to as a wave code since solution resolves the stress waves within a body.
- Discretizes the equations of motion for a body and solves the resulting system of equations using a central difference time integrator that advances the solution from an initial state.
- Solution algorithm is conditionally stable requiring a time step that scales with the transit time of a dilatational wave over the shortest dimension of an element.

Cohesive Zone Model

- Material separation based on a specified traction-separation (σ–δ) relationship.
  - -Implemented in PRESTO within context of contact algorithm.
- Crack growth is a natural outcome of the solution.
- Key parameters are the cohesive strength $\hat{\sigma}$ and the work of separation/unit area Γ_o.
- Mesh-independent results.
- Defined in terms of a potential that depends on a scalar effective separation.
- Similar to model introduced by Tvergaard and Hutchinson (J.Mech. Phys. Solids, 1993).



Effective Separation $$\lambda = \sqrt{\left(\frac{\delta_n}{\delta_n^c}\right)^2 + \left(\frac{\delta_t}{\delta_t^c}\right)^2}$$

The traction-separation relation represents in a rudimentary way the atomistic separation process.

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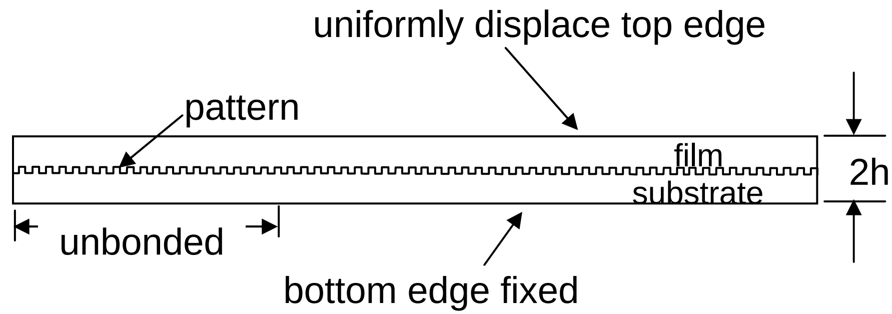
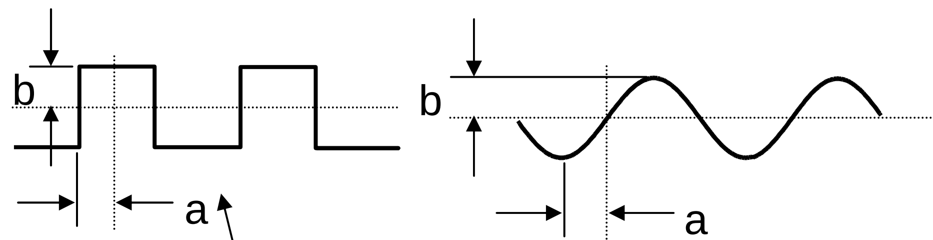
Fracture Analysis

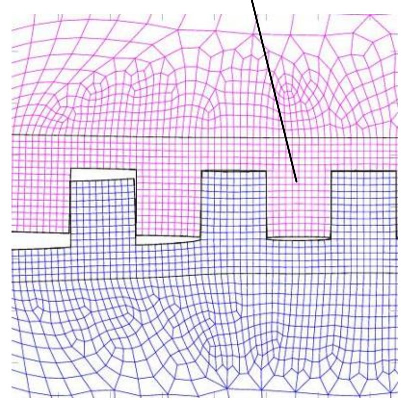
- Analyzed idealized problem of a thin, bimaterial strip loaded by displacing the top edge relative to the bottom edge.
- A plane strain analysis that defines parallel channels.
- Can generate any desired load mode mixity.
- There is a simple analytic solution for fixed-grip loading when interface is flat:

$$\Gamma_a = \frac{\sigma_n^{*2} h}{2\left(1/E_{u1} + 1/E_{u2}\right)^{-1}} + \frac{\tau^{*2} h}{2\left(1/G_1 + 1/G_2\right)^{-1}}$$

- Define the effective interfacial toughness Γ_a as the toughness that a similar but flat interface would have for crack propagation at the same critical far-field ligament stress.







W: E=410 GPa, ν=0.28
Si: E=161 GPa, ν=0.23
Γ_o = 0.3 J/m^2

Simulations to verify Γ_a calculations

Pattern type	Code	Interfacial element length (nm)	$\hat{\sigma}$ (MPa)	Γ_a (J/m^2)
flat	Presto	5.0	600	0.31
flat	Presto	5.0	1000	0.30
square	Presto	5.0	600	0.58
square	Presto	2.5	600	0.62
square	Presto	5.0	1000	0.88
square	Presto	2.5	1000	0.92
square	Tahoe	5.0	600	0.62
square	Tahoe	5.0	1000	0.92

- W/Si with h=250 nm
- interface pattern has a=b=25 nm
- Γ_o=0.3 J/m^2
- global mode I loading
- Loading rate slow so KE associated with loading is negligible.
- Tahoe code uses interface elements and quads with 4-point integration for the film and substrate.
- Calculated failure process also the same.

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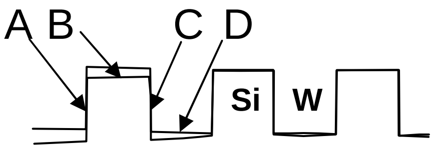
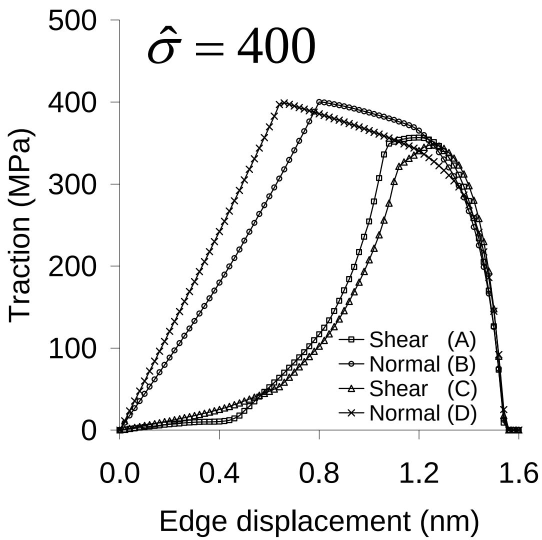
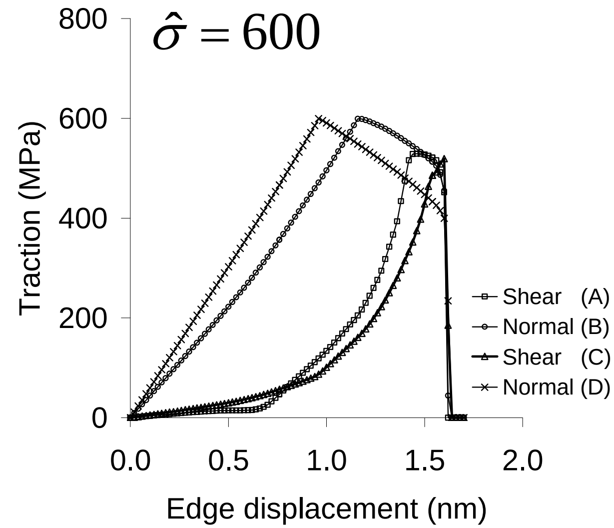
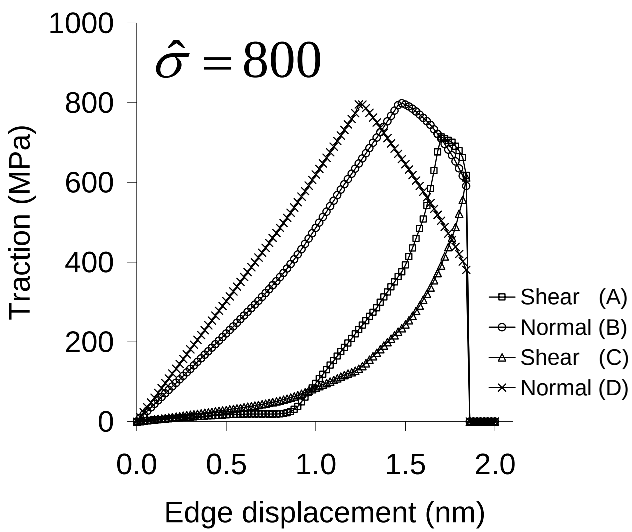
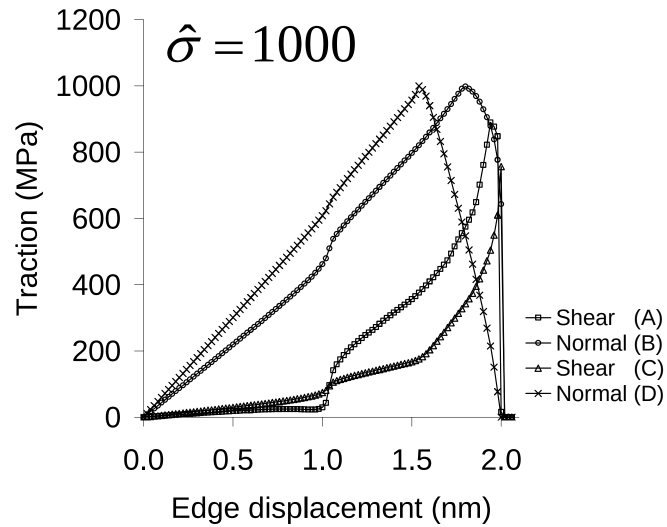
Vary interfacial strength $\hat{\sigma}$





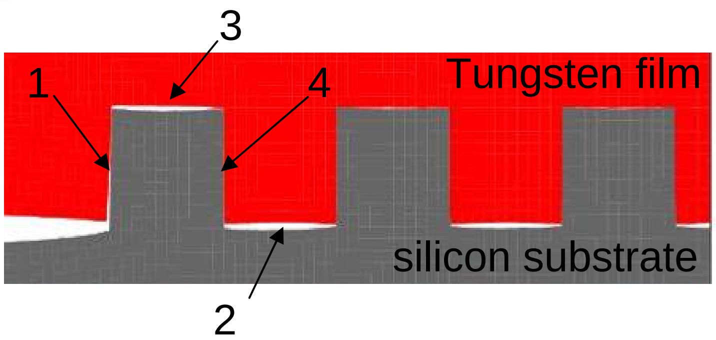






W/Si, *h*=250 nm, square channel interface pattern with *a*=*b*=25 nm, Γ_o=0.3 J/m^2, global mode I loading, vary δ_c to maintain Γ_o

Key Observations



<u>Crack growth is discontinuous</u>

1. Crack stalled by high energy needed to kink 90°.
2. Initiation of interfacial segment cracking at ends of W teeth.
3. Initiation of interfacial segment cracking at ends of Si teeth.
4. Side wall fails in shear at stalled crack tip.
5. Crack propagates rapidly

- Postulate that crack propagation depends on the ratio of interfacial strength $\hat{\sigma}$ to the stress σ_c characterizing the tendency to open an interfacial crack segment.
- If stress needed to nucleate segment cracking, $\hat{\sigma}$, exceeds that needed open the segment, σ_c, the segment will open rapidly --- generating KE.
- Rough estimate of σ_c from solution of a finite crack between to half-planes.

$$\sigma_c = \sqrt{\frac{E^*\Gamma_o}{\pi a}}$$

where

E^* is a bimaterial modulus

Γ_o = intrinsic interfacial toughness

a = the characteristic length scale of the pattern

Apparent toughness Γ_a

Vary interfacial strength $\hat{\sigma}$
W/Si bimaterial

$\hat{\sigma}$ (MPa)	σ_c (MPa)	$\hat{\sigma}/\sigma_c$	Γ_a (J/m^2)	$\Gamma_a/(A_r\Gamma_o)$
400	970	0.41	0.42[1]	0.70
600	970	0.62	0.58	0.97
800	970	0.83	0.78	1.30
1000	970	1.03	0.88	1.47
1200	970	1.24	0.90[1]	1.50

[1] Maximum applied load does not coincide with crack propagation.

Tungsten film
Vary substrate material
$\hat{\sigma} = 600\,\text{MPa}$

	E (GPa)	ν	α	σ_c (MPa)	$\hat{\sigma}/\sigma_c$	Γ_a (J/m^2)	$\Gamma_a/(A_r\Gamma_o)$
tungsten	410	0.28	0.00	1300	0.46	0.47	0.79
silicon	161	0.23	0.45	970	0.62	0.58	0.97
aluminum	69	0.32	0.71	710	0.85	0.68	1.13
graphite	17	0.30	0.92	370	1.62	0.89[1]	1.48

[1] Maximum applied load does not coincide with crack propagation.

h=250 nm, square channel interface pattern with a=b=25 nm, Γ_o=0.3 J/m^2, global mode I loading

Apparent toughness Γ_a

Vary intrinsic toughness Γ_o
h=250 nm
a=b=25

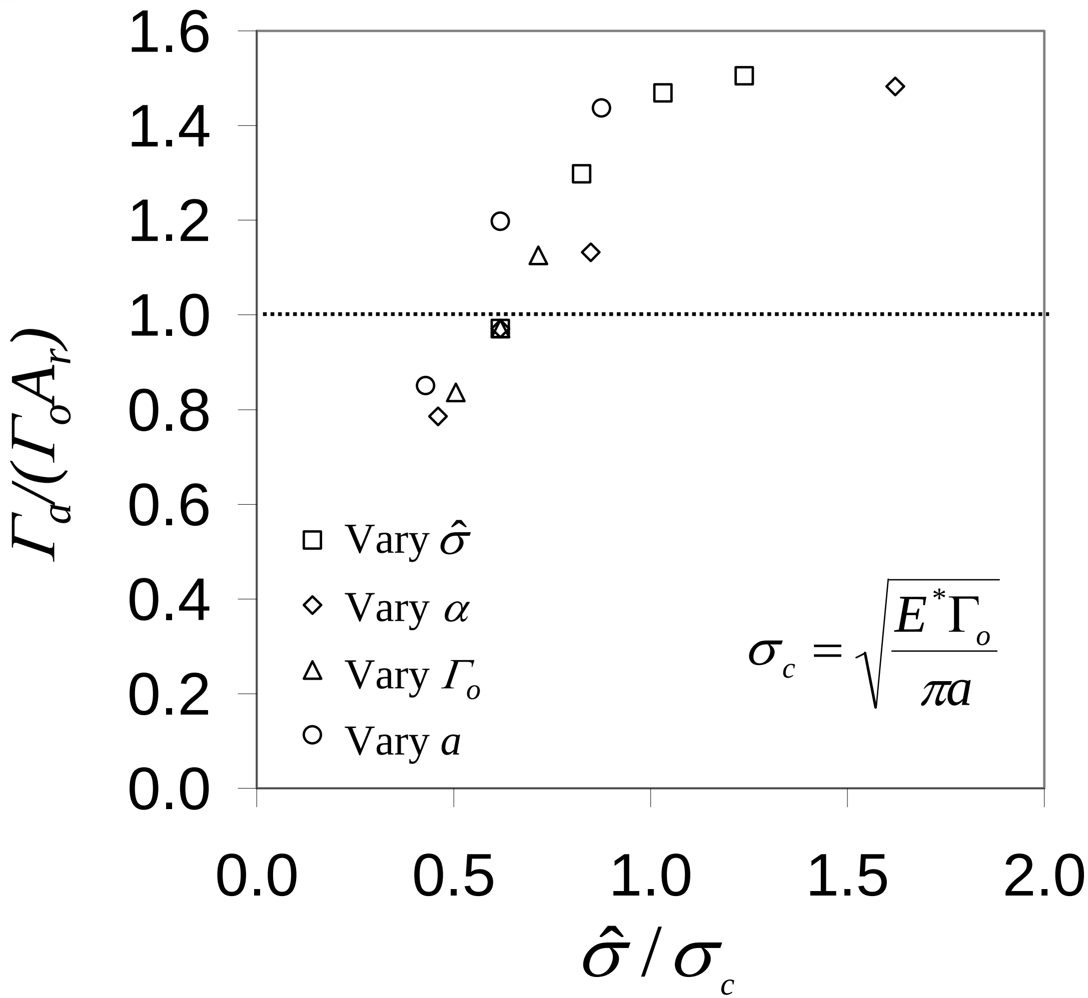
(J/m^2)	$\hat{\sigma}$ (MPa)	σ_c (MPa)	$\hat{\sigma}/\sigma_c$	Γ_a (J/m^2)	$\Gamma_a/(A_r\Gamma_o)$
0.2	400	790	0.51	0.34	0.84
0.3	600	970	0.62	0.58	0.97
0.4	800	1120	0.71	0.90	1.13

Vary characteristic length scale a
a=b
h=500 nm
Γ_o=0.3 J/m^2
$\hat{\sigma} = 600\,\mathrm{MPa}$

a (nm)	σ_c (MPa)	$\hat{\sigma}/\sigma_c$	Γ_a (J/m^2)	$\Gamma_a/(A_r\Gamma_o)$
12	1400	0.43	0.51	0.85
25	970	0.62	0.72	1.20
50	690	0.88	0.86	1.44

W/Si bimaterial, square channel interface pattern with a=b nm, global mode I loading

Apparent toughness Γ_a



- Γ_a increases with $\hat{\sigma}/\sigma_c$
- A more "dynamic" fracture process with increased energy dissipation associated with kinetic energy (which is ultimately transformed to thermal energy).
- Not without bound –could have change in failure process (e.g.voids in ligament growing dynamically without stalled crack growing).

Apparent toughness Γ_a

Vary pattern aspect ratio *b/a*
Square channel pattern
a=25 nm
global mode I loading

b/a	A_r	σ_c (MPa)	$\hat{\sigma}/\sigma_c$	Γ_a (J/m^2)	$\Gamma_a/(A_r\Gamma_o)$
0.5	1.5	970	0.62	0.40	0.89
1.0	2.0	970	0.62	0.58	0.97
1.5	2.5	970	0.62	0.83	1.10

Vary pattern type
a=*b*=25 nm
A_r=1.5 ripple
A_r=2.0 square

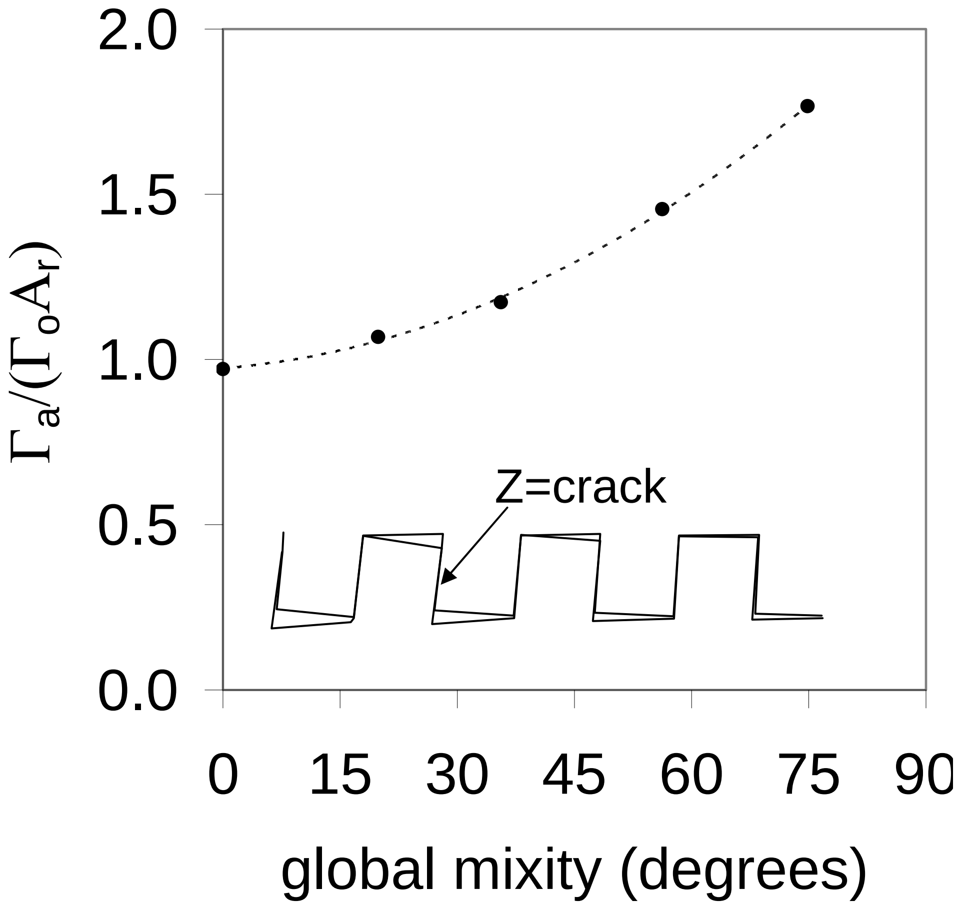
Pattern type	Global mode mixity (degrees)	$\hat{\sigma}$ (MPa)	σ_c (MPa)	$\hat{\sigma}/\sigma_c$	Γ_a (J/m^2)	$\Gamma_a/(A_r\Gamma_o)$
ripple	0	600	970	0.62	0.44	0.99
square	0	600	970	0.62	0.58	0.97
ripple	0	1000	970	1.03	0.62	1.40
square	0	1000	970	1.03	0.88	1.47
ripple	19	600	970	0.62	0.49	1.11
square	20	600	970	0.62	0.64	1.07

- Apparent interfacial toughness scales directly with ratio of true to nominal interfacial area, A_r.
- Not without bounds, could have change in failure mode (e.g., fracture tooth, etc.).

W/Si, *h*=250 nm, pattern with, Γ_o=0.3 J/m^2, $\hat{\sigma} = 600\,\text{MPa}$

Vary applied global mode mixity

- Global mode mixity defined as $\tan^{-1}(\tau^*/\sigma_n^*)$
- Apparent interfacial toughness Γ_a increases with applied global mode mixity.
- Rapid propagation of Z-cracks when mixity>35°(one side-wall remains bonded).
- Increased Γ_a associated with strain energy locked into the material by contact.



W/Si, h=250 nm, square channel pattern with a=b=25 nm, Γ_o=0.3 J/m^2, $\hat{\sigma}$ = 600 MPa

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Summary

- Experiments with nm-scale interfacial patterning suggest this is one approach for improving toughness of brittle, thin-film material interfaces.
- Have developed a fundamental understanding that will aid in the design of patterned interfaces with improved toughness.