Combinatorial (Multivariant) Approach to Measuring Thin Film Adhesion

Motivation: Provide a fast, practical and reliable thin film adhesion test method to industry

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NIST Combinatorial Methods Center (www.nist.gov/combi)

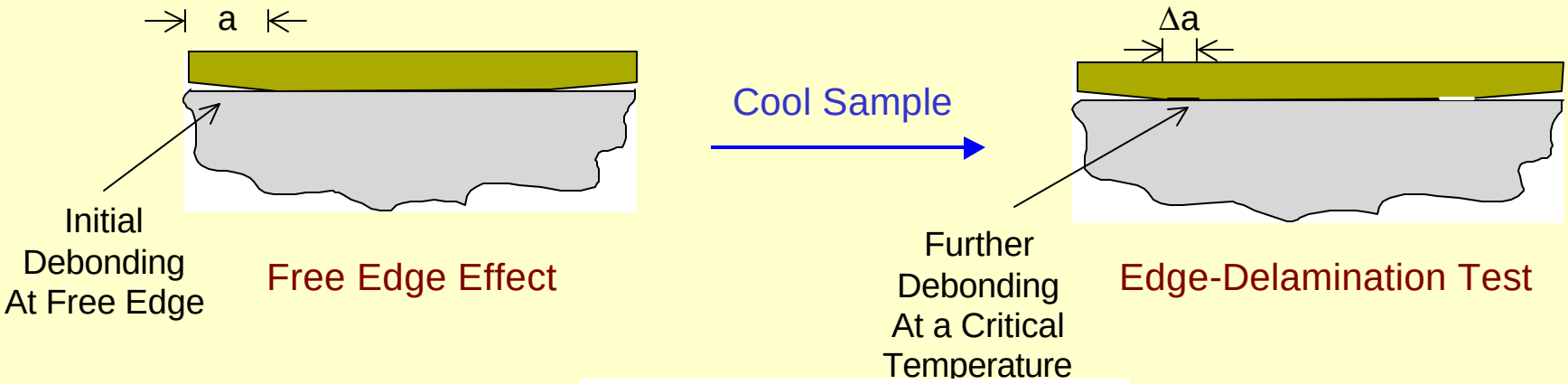
Combinatorial (Multivariant) Approach to Measuring Thin Film Adhesion

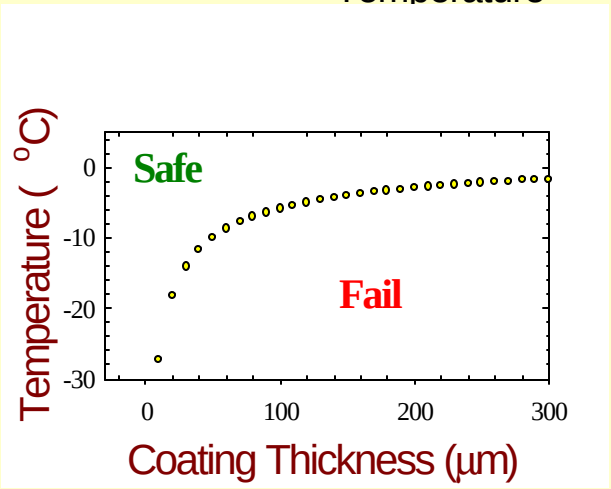
- Objective: Develop combinatorial approach to the edge-delamination test for thin film adhesion ---- as a function of temperature and film thickness

Outline

- Introduction: the Edge-Delamination Test and Current Industry Practice
- Combinatorial Approach to the Edge-Delamination Test
- Issues for Valid Test Results: Finite Element Analysis and Simulation
- Issues in Experiments and Initial Experimental Results
- Summary and Conclusions

Introduction: the Edge-Delamination Test and Current Industry Practice





Current Industry Practice

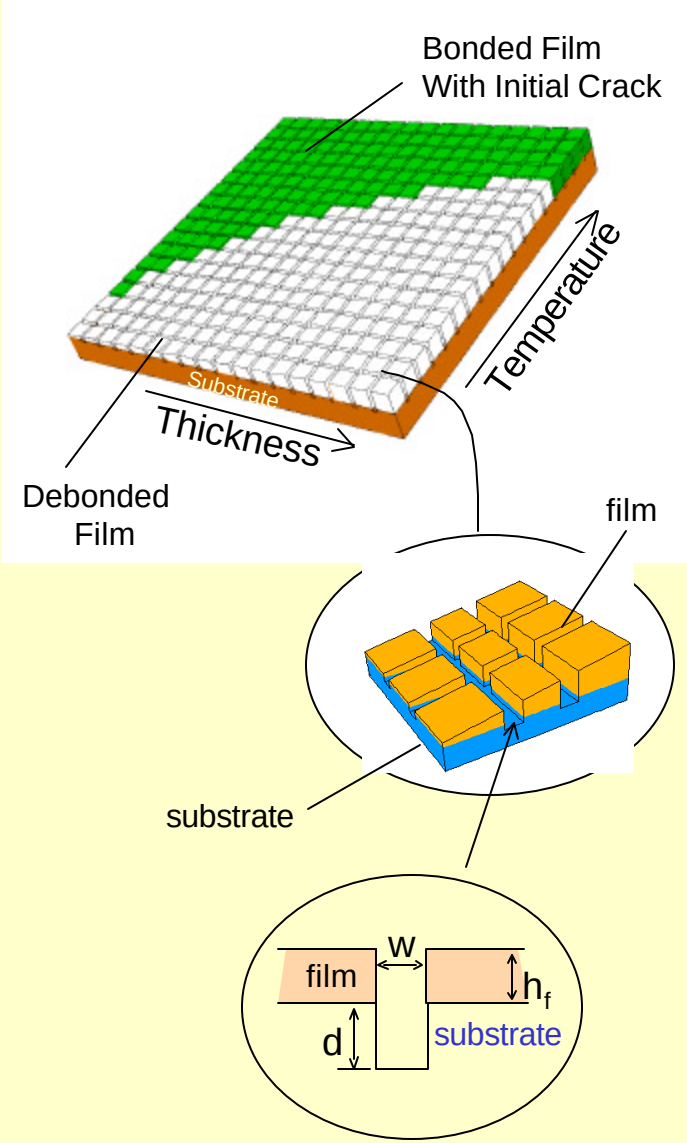
Combinatorial (Multivariant) Approach to the Edge-Delamination Test

Combinatorial Measurements

- Multivariable Approach
  (film thickness, temperature, composition, etc.)
- High-throughput Data Collection

Procedure

- Coat a film with thickness gradient on a substrate
- Dice into square samples
- Apply cooling with temperature gradient orthogonal to thickness gradient
- Monitor interfacial debonding events using image analysis
- Construct failure map
- Quantify the adhesion of a film to substrate by using critical energy criterion



Combinatorial (Multivariant) Approach to the Edge-Delamination Test

Alternate Multivariant Approach to Edge-Delamination Test:

To extend the method to brittle films or
films that do not generate sufficient thermal deformation to induce debonding

- Coat a test film with thickness gradient on a substrate

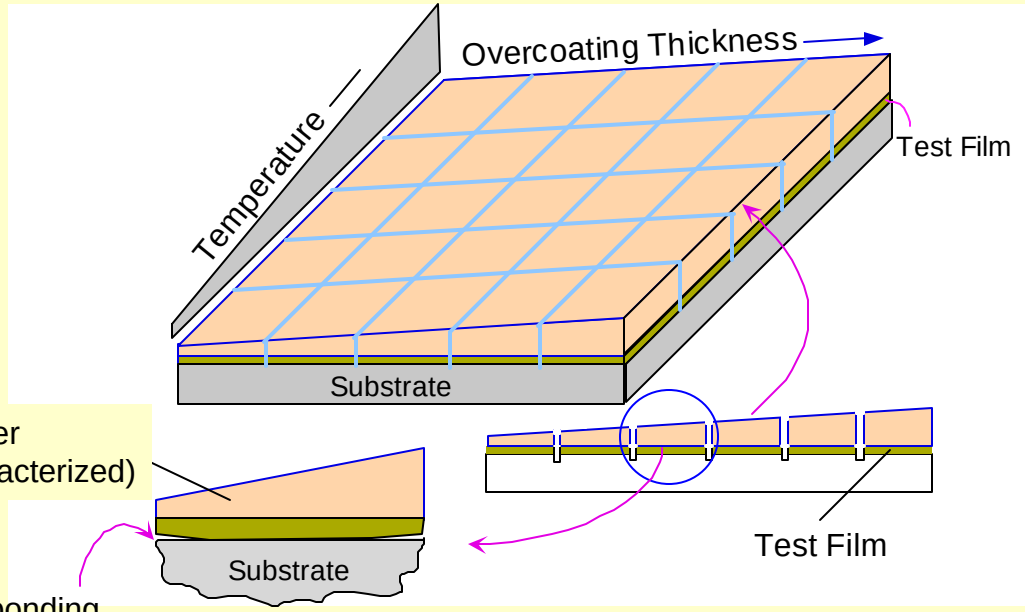
Replaced by →

- Coat a test film with constant (very small) thickness on a substrate
- Apply an overcoating layer with thickness gradient on top of the test film

Advantages:

Knowledge of the thermomechanical properties of test film is not required

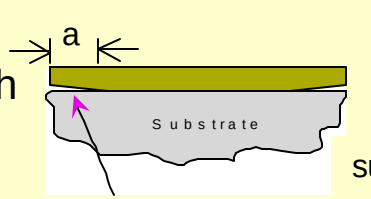
Only the thickness and thermomechanical properties of the overcoating need to be known



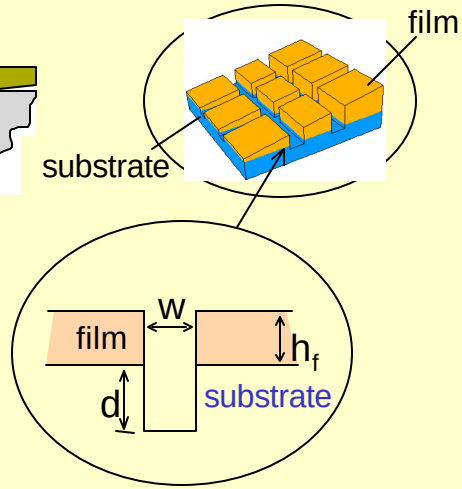
Issues for Valid Test Results: Finite Element Analysis

Issues:

- Stress state should be independent of initial crack length
- Each edge-delamination sample should be independent from the others



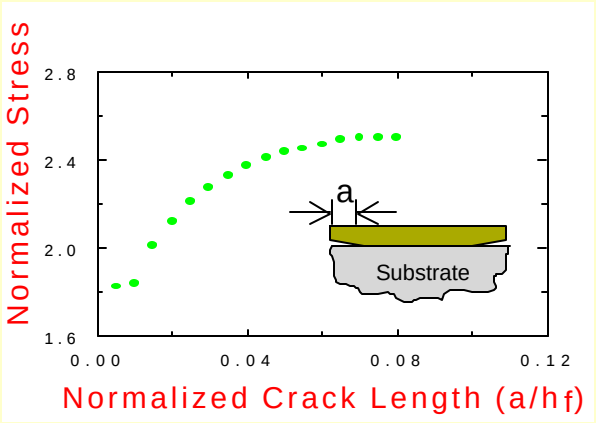
Initial Debonding
At Free Edge

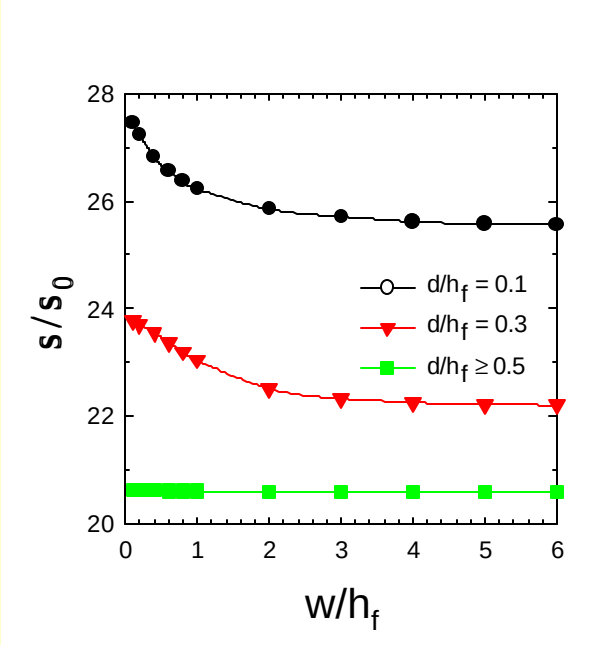


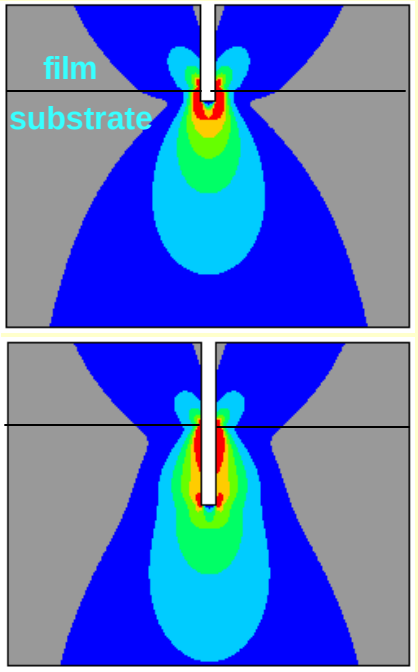
Two Questions Need to be Addressed:

- What is the minimum initial crack length such that the stress state is independent of crack length?
- What are the optimum depth (d) and width (w) of the cut to eliminate stress interactions?

Approach: Fracture mechanics with 3D finite element analysis



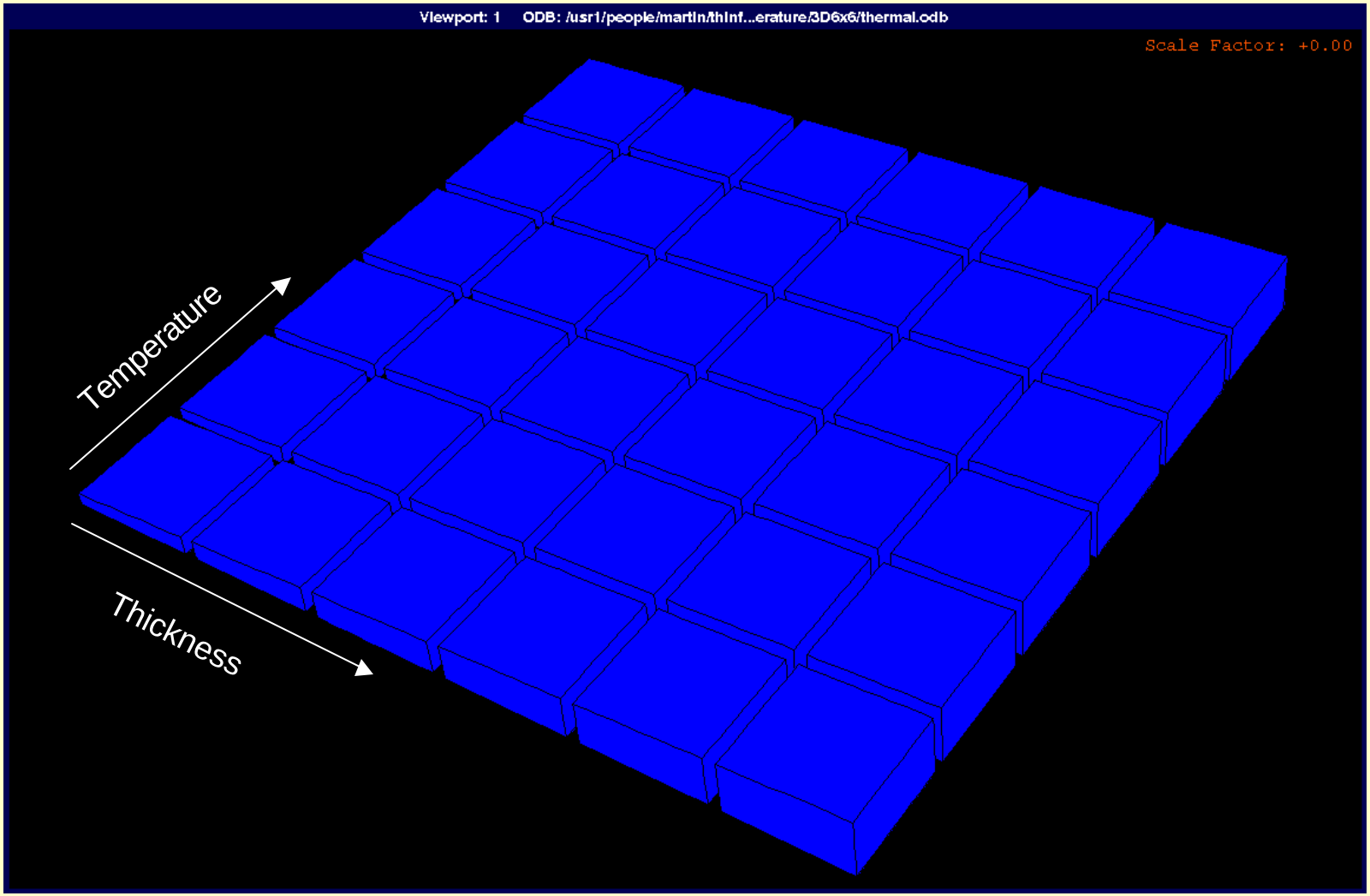




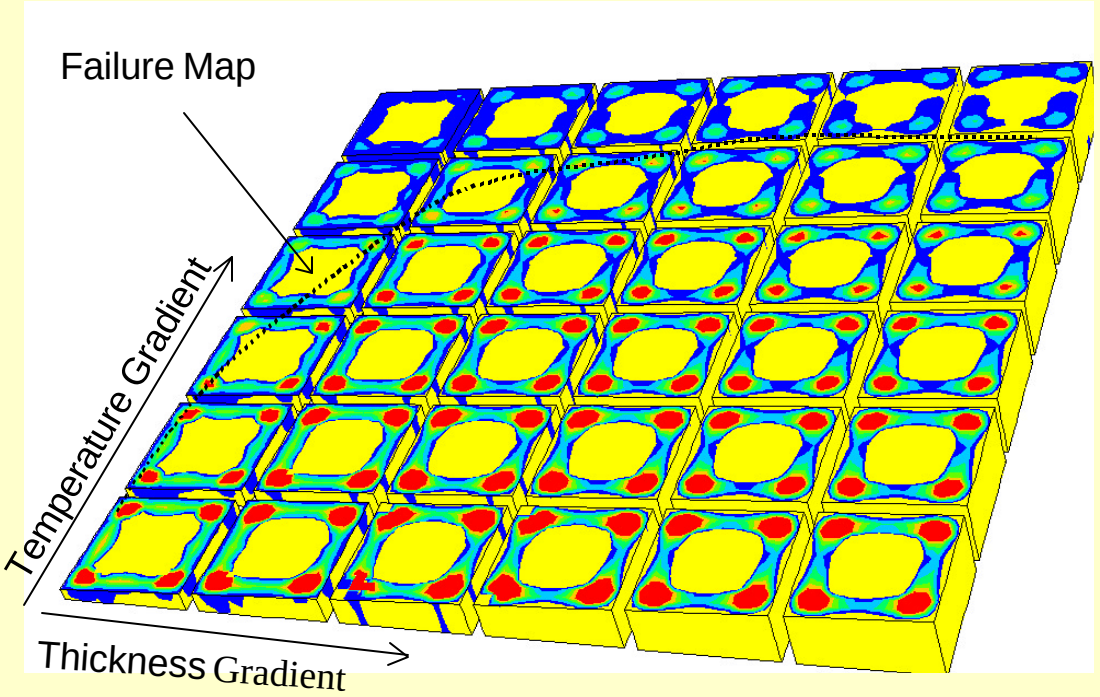
d/h_f=0.1
w/h_f=0.1

d/h_f=0.5
w/h_f=0.1

Finite Element Simulation



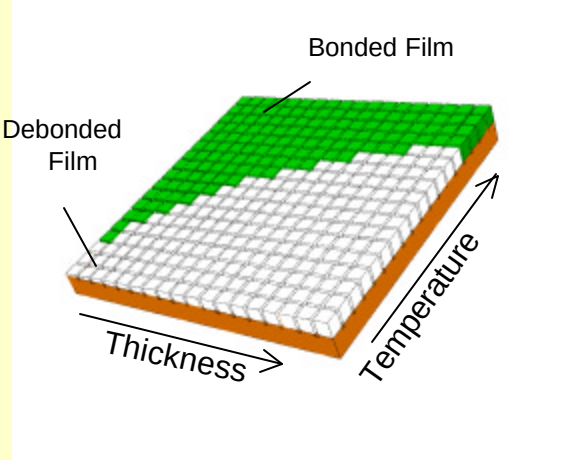
Finite Element Simulation

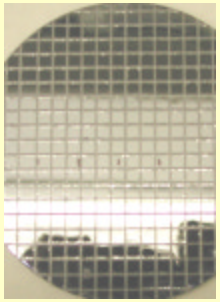


Initial Experimental Results

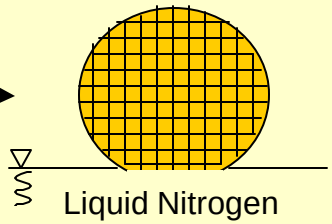
Issues:

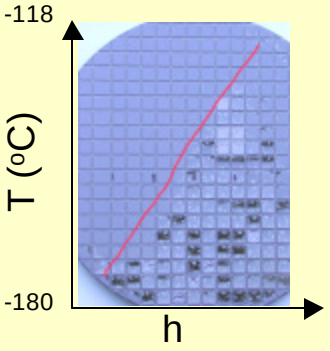
Thickness Gradient Control
Dissolving of Test Film
Sample Dicing
Temperature Control
Other Variables





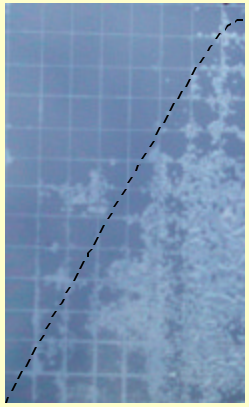
Thickness Gradient
40 - 220 mm

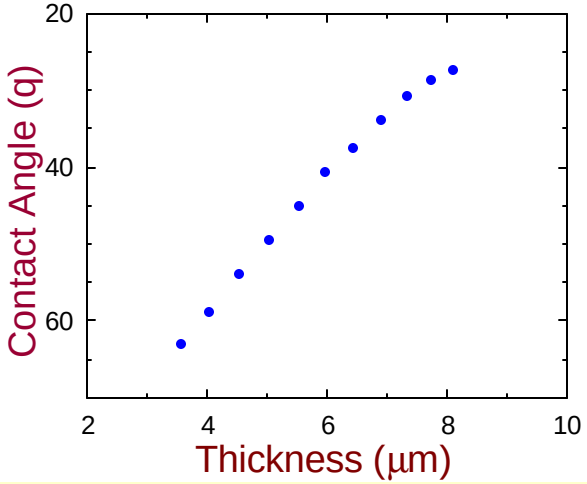


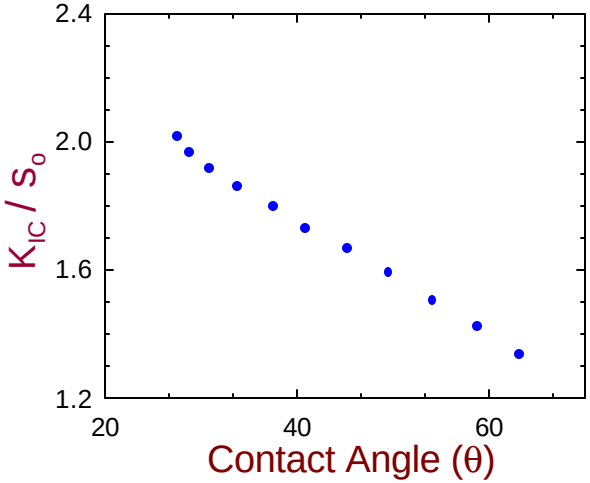


Other Variables

Thickness – Contact Angle → Thickness – Temperature for Each Contact Angle

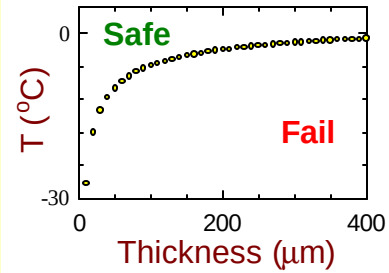




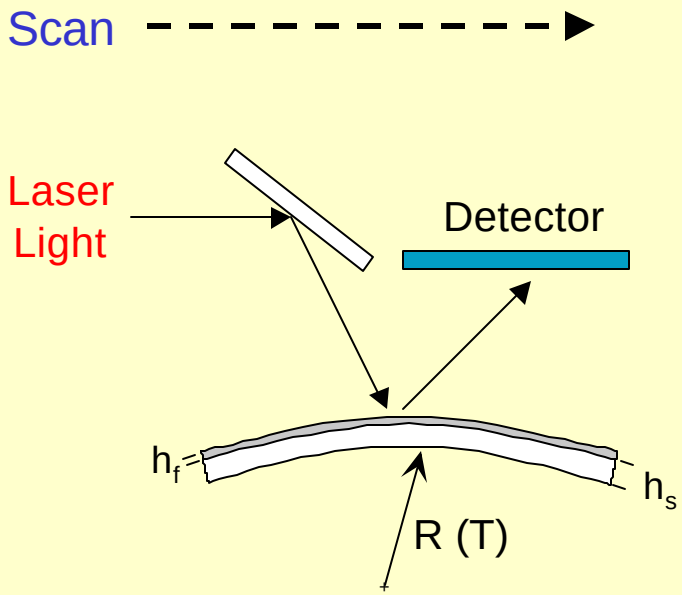


$$\frac{K_I}{s_o} = f(h) \quad + \quad s_o = s_o(T)$$

for each contact angle →



Stress -Temperature Relationship



$$s_o = \frac{\bar{E}_s h_s^2}{6 R h_f}$$ Stoney Eq.

stress-temperature relationship

Summary and Conclusions

- The Edge-Delamination Test and Current Industry Practice
- Multivariant Approach to Edge-Delamination Test
- Valid Test Results: Finite Element Analysis and Simulation
- Initial Experimental Results

- Successfully used finite element modeling to design the experimental protocol for the combinatorial adhesion test which shows the method will work

- Initial tests prove the concept of using thicker overcoating to generate necessary stress in a thin brittle film