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Fracture Toughness Measurements of Amorphous Diamond Thin Films using Acoustic Emission-Sensing Nanoindentation

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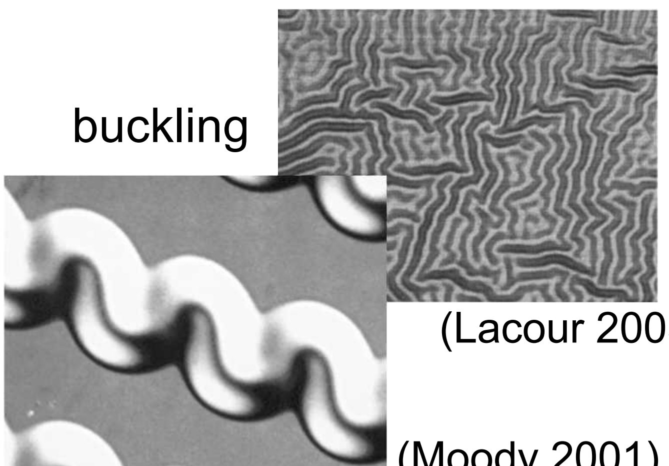
Coating Lifetime and Performance

- **Determination of mechanical properties is critical in designing modern IC and MEMS devices**

- **Understanding adhesion and fracture behavior allows for increased reliability of nanostructured materials and devices.**

wrinkling

buckling



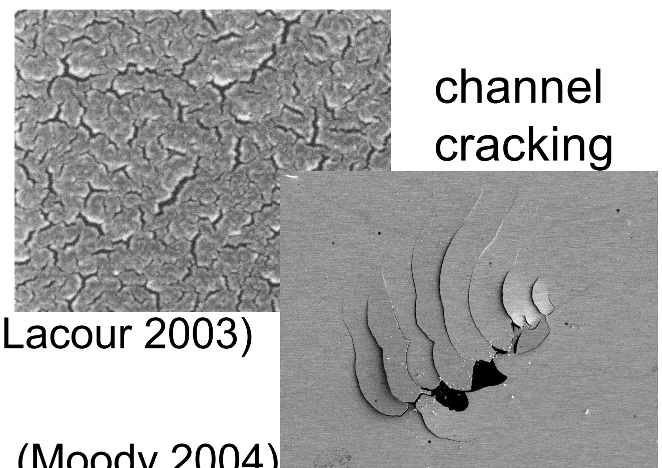
(Lacour 2003)

(Moody 2001)

delamination

mudflat cracking

channel cracking

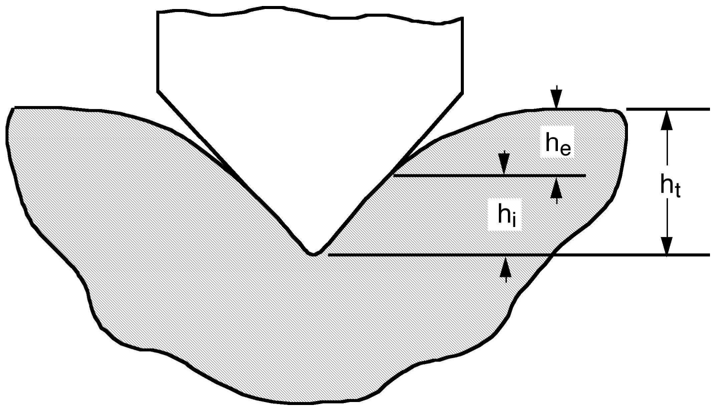


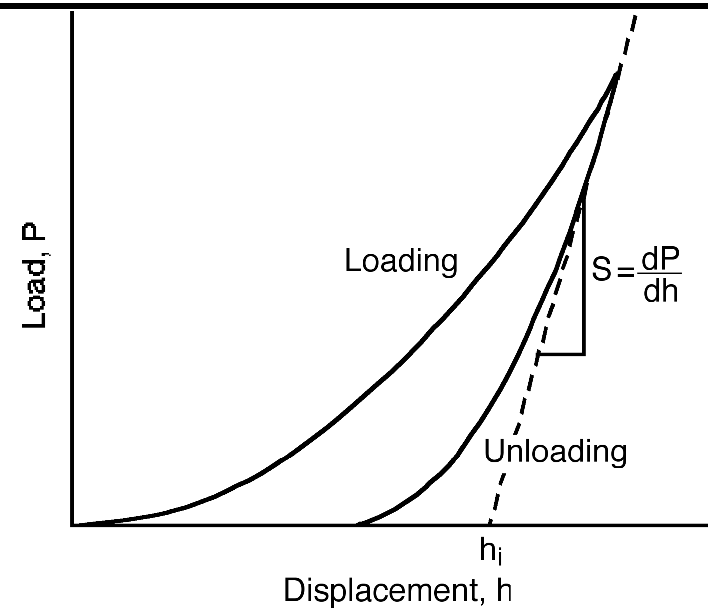
(Lacour 2003)

(Moody 2004)

decohesion

Nanoindentation Technique





Stiffness is calculated from the elastic unloading curve:

$$S = \frac{dP}{dh} = \frac{2E_r\sqrt{A_i}}{\sqrt{\pi}}$$

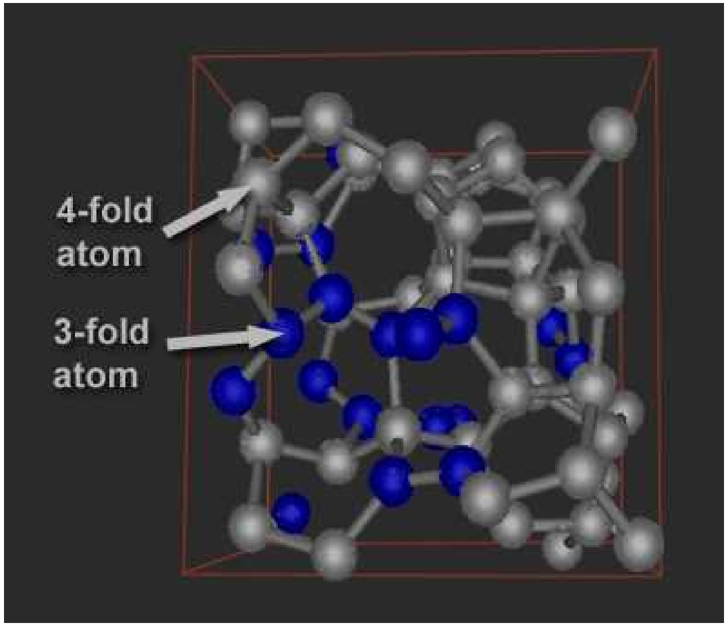
from which the sample elastic modulus can be determined as follows.

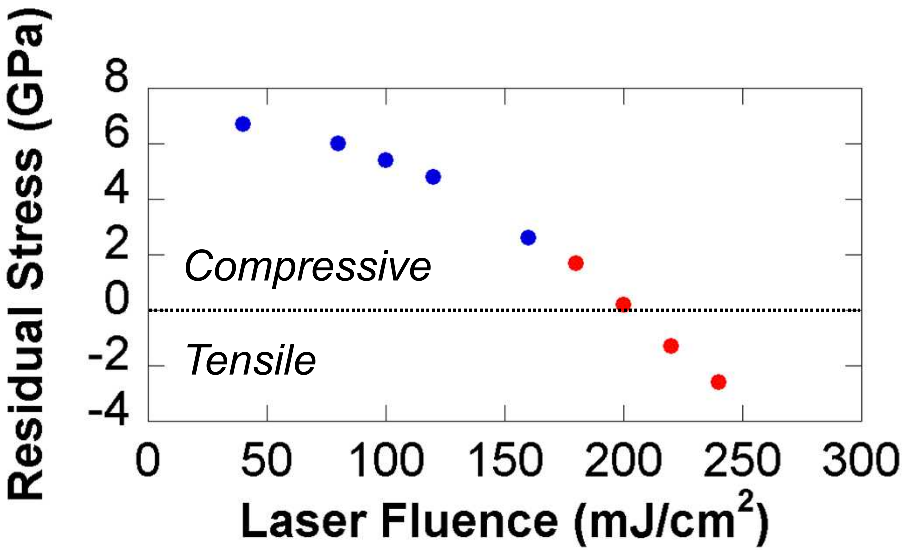
$$\frac{1}{E_r} = \frac{\left(1 - \nu_s^2\right)}{E_s} + \frac{\left(1 - \nu_i^2\right)}{E_i}$$

Furthermore, hardness can be determined as

$$H = \frac{P_{max}}{A_i}$$

Amorphous Diamond System

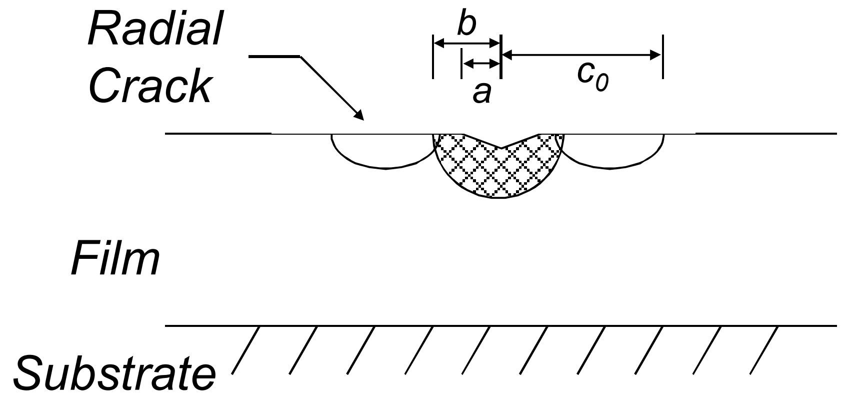




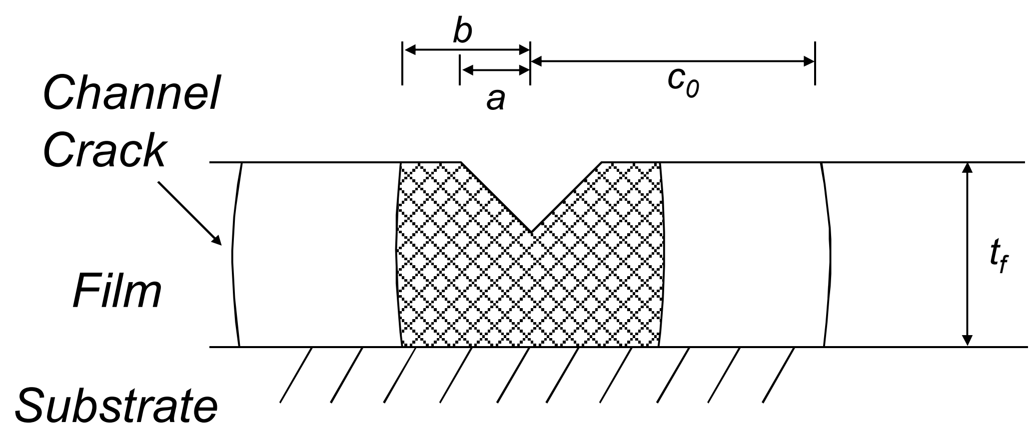
- 110 nm thick films
- 80% sp^3 (4-fold) bonding
  - E ~ 750 GPa
  - H ~ 43 GPa
- σ_R = 1.7 GPa compressive to 2.6 GPa tensile
- Cube-corner probe

Indentation Fracture Mechanics in Thin Films

$$K = \xi \sqrt{\frac{E}{H}} \frac{P}{c^{3/2}} + \psi \sigma_f t_f^{1/2}$$

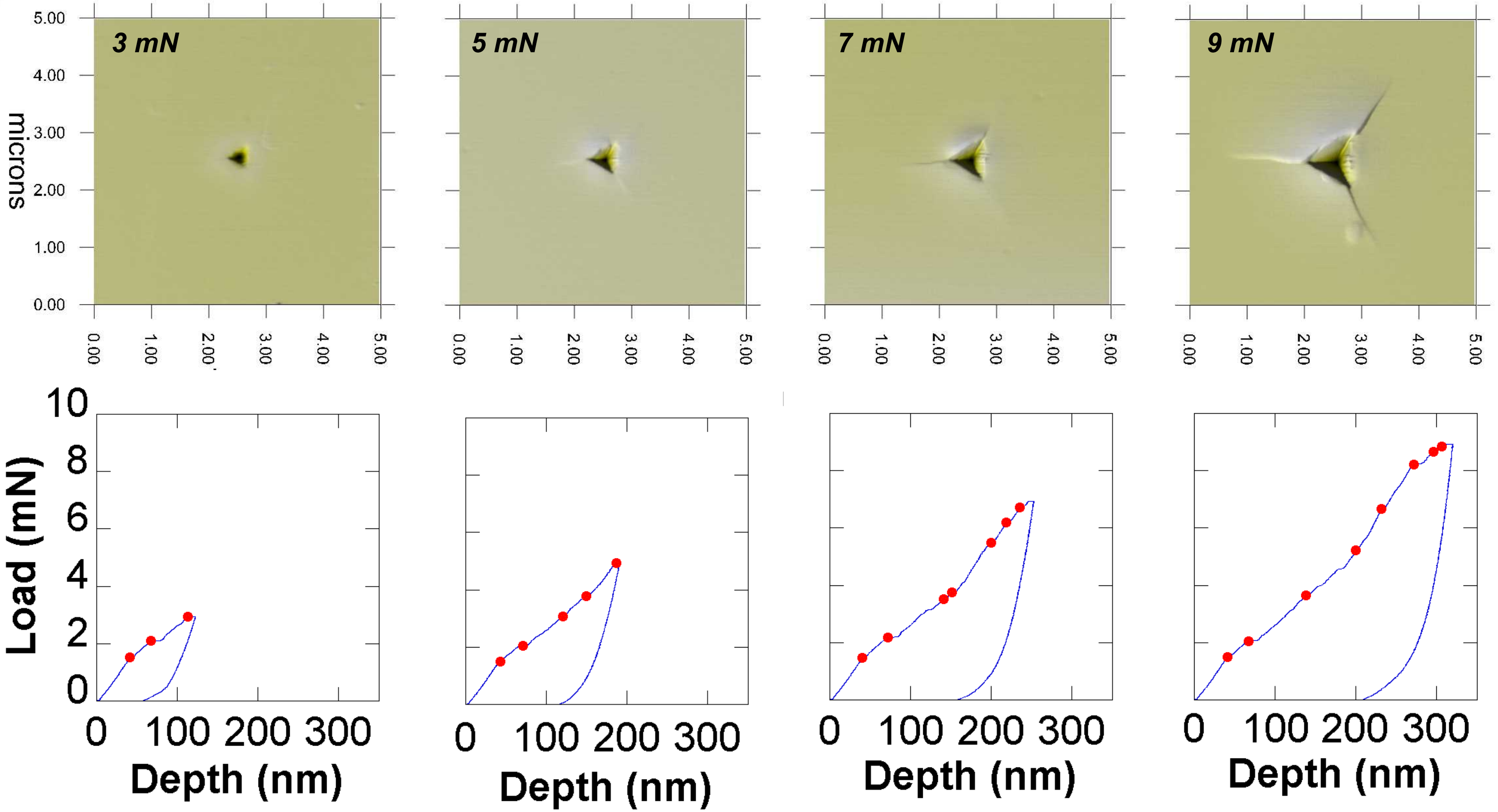


$$K = \lambda \left(\frac{E_f}{H_f} \right)^{1/3} \frac{1}{t_f} \frac{P}{c_0^{1/2}} + \psi \sigma_f t_f^{1/2}$$



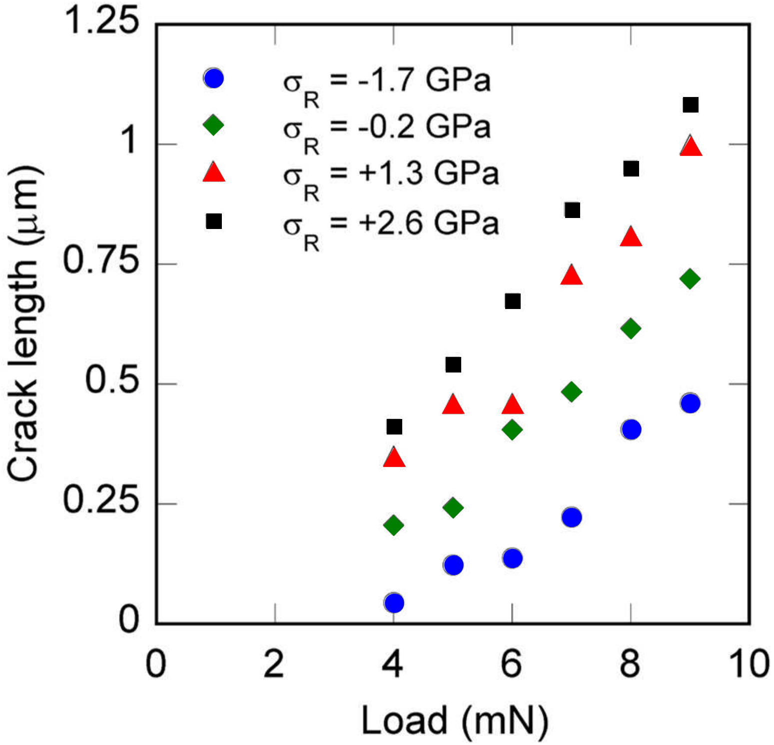
Lawn, Evans and Marshall, 1980
Thurn and Cook, 2004

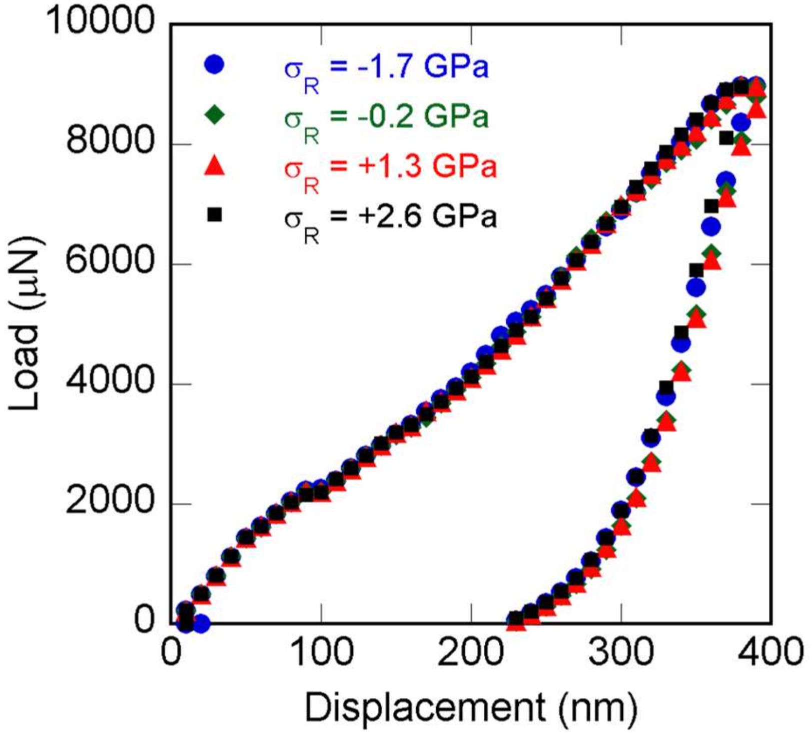
AE Indicates Fracture During Loading



- **Acoustic events imply cracking during loading**

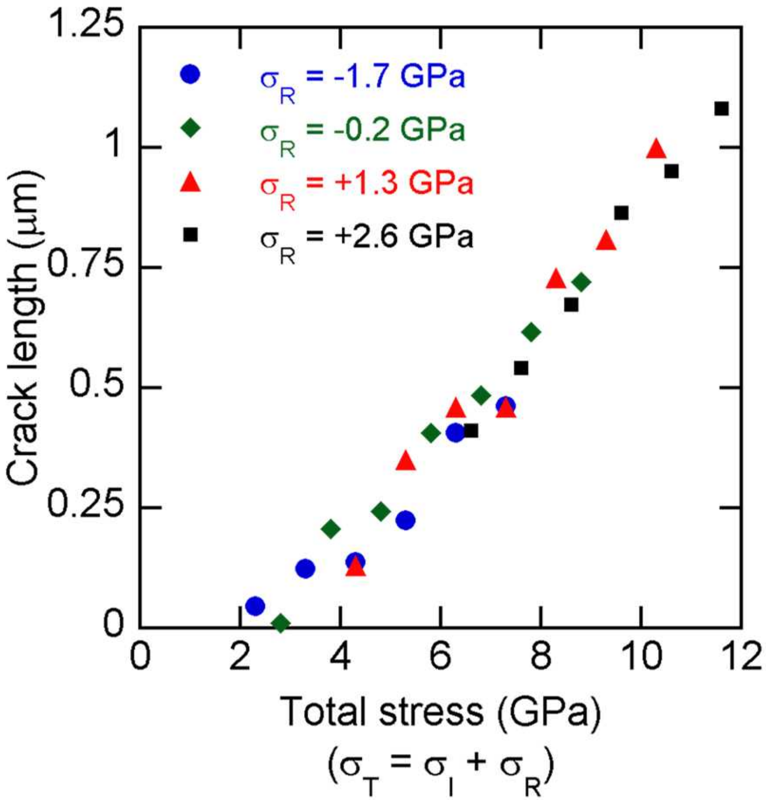
Indentation Crack Length - Load Relationship

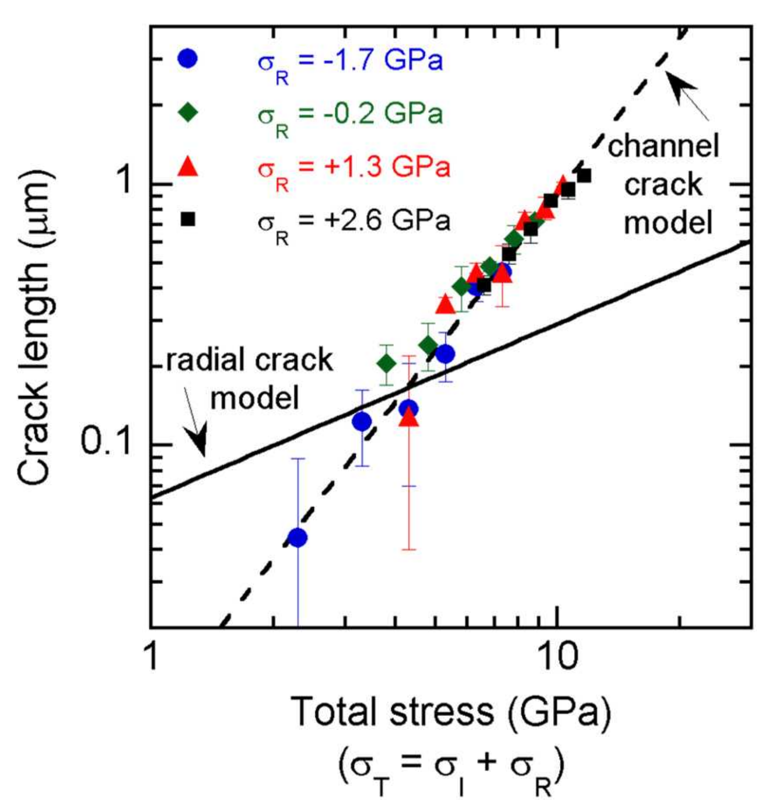




- **Induced crack length is a function of both applied load and residual stress**
- **Load-displacement response is residual stress invariant**
  - **Produce a relationship between applied load and indentation stress at crack tip**

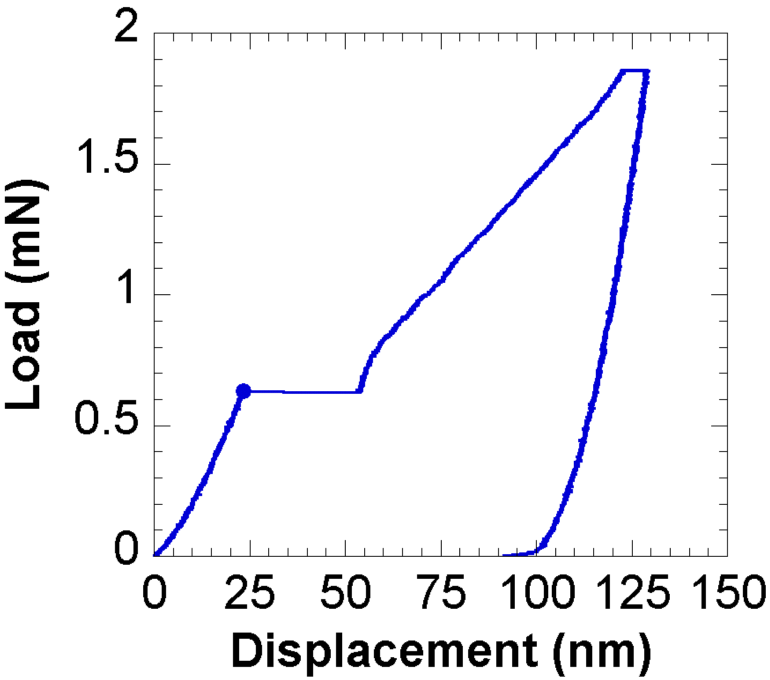
Crack Length Normalization and Model Result

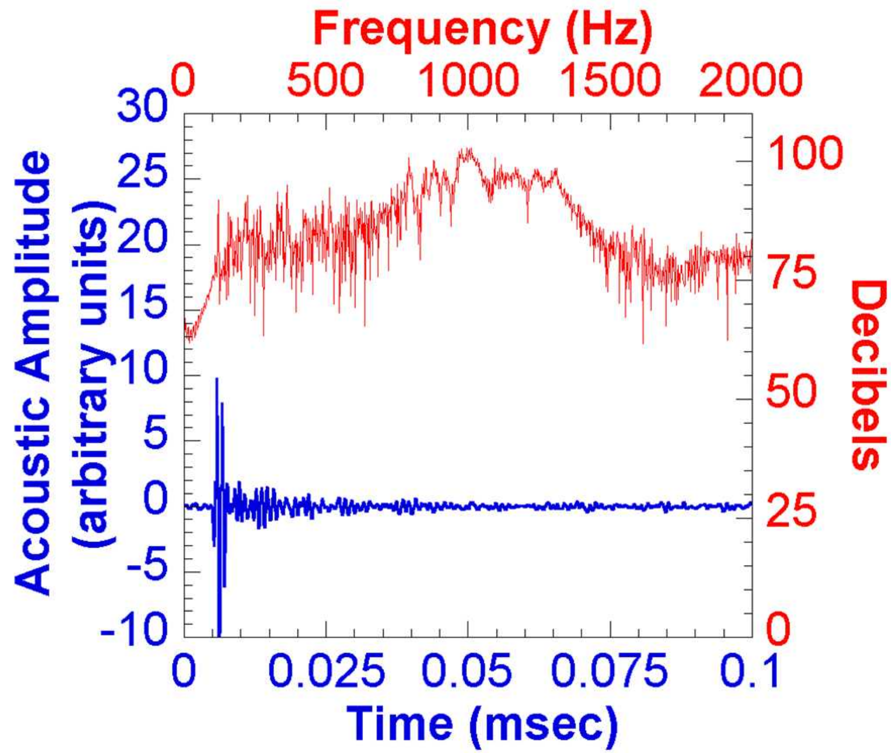




- **If 1 mN = 1 GPa, then the data collapses onto a single curve**
- **From this curve, a good fit of the data is obtained with the channel crack model**

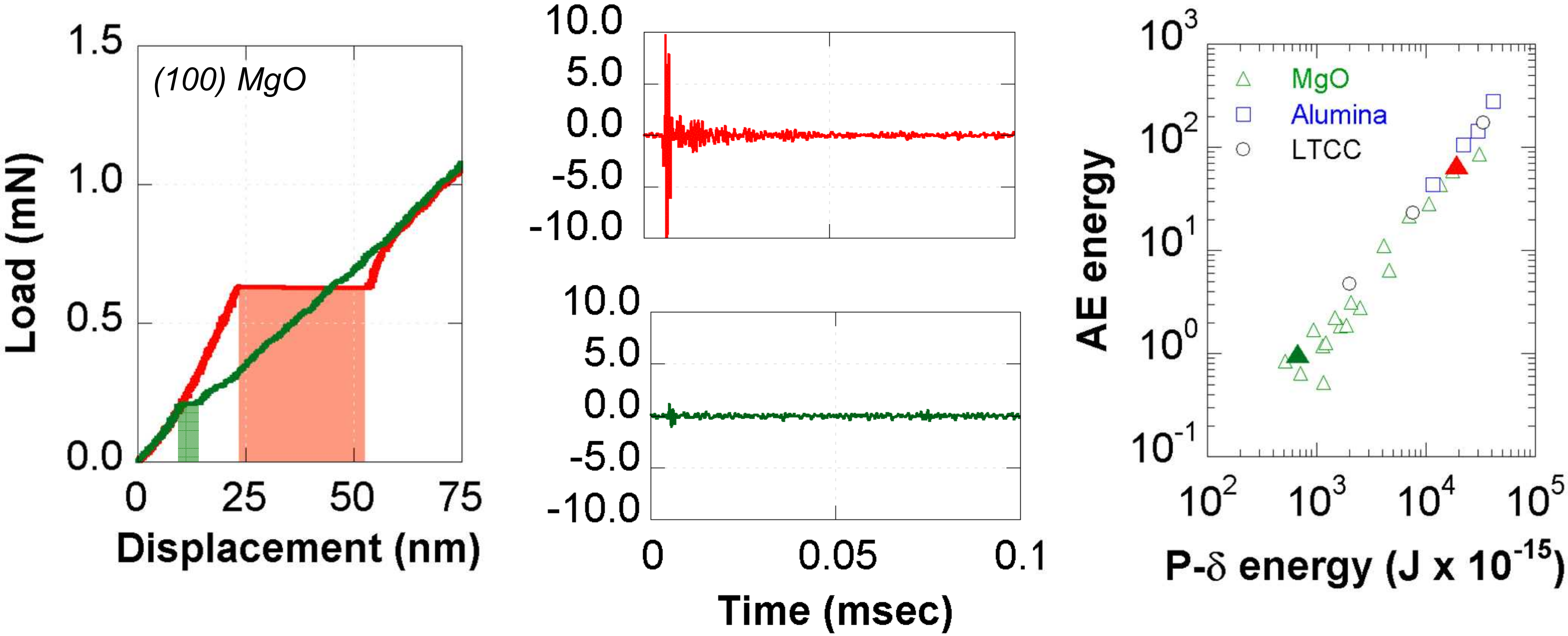
Indentation Acoustic Emission





- Indentation "pop-in" or "pop-out" transients are occasionally observed
- Transients usually correspond to mechanical events
- Acoustic waveform and frequency distributions as alternate characterization

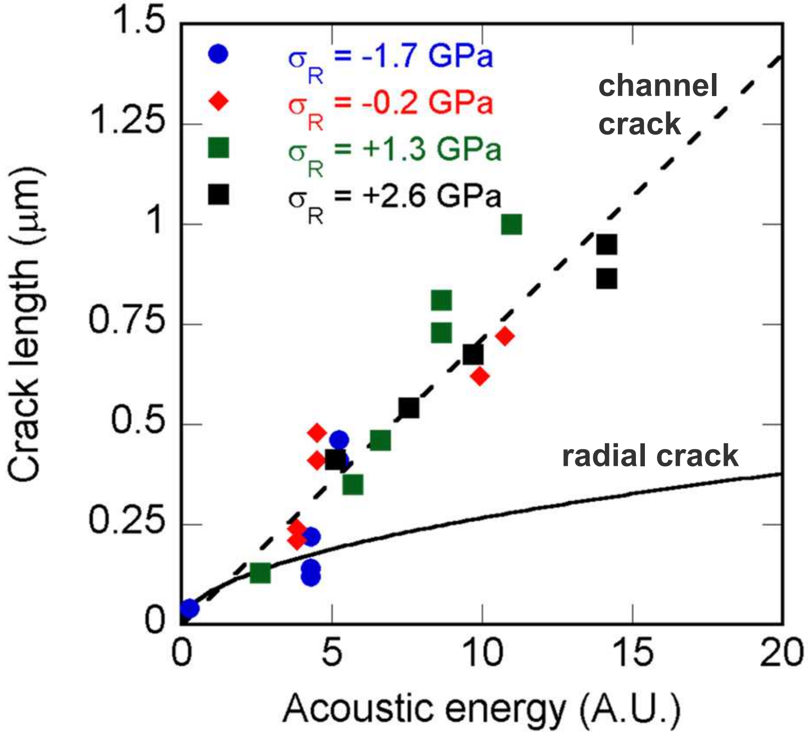
Acoustic Emission Relationships



- **Good agreement between AE magnitude and tip excursion on load-displacment curve**

- **Linear agreement between AE energy and *P-δ* energy**

Acoustic Energy and Fracture Toughness



$$K = \frac{\beta'' V^*}{t_f^{2/3}} \left(\frac{P^4}{\Sigma AE} \right)^{\frac{1}{3}} + \psi' \sigma_R t_f^{1/2}$$

- **Observe a good correlation between released acoustic energy and measured crack length**
- **Implies that AE methods may be used to approximate the crack length**

Measured Toughness Comparisons

Sample	Residual Stress (GPa)	K_{Indent} (MPa·m$^{1/2}$)	K_{AE} (MPa·m$^{1/2}$)
AD7	-1.7	3.7 $\pm$ 0.6	3.8 $\pm$ 0.2
AD8	-0.2	3.5 $\pm$ 0.2	3.0 $\pm$ 0.4
AD9	+1.3	3.3 $\pm$ 0.2	3.7 $\pm$ 0.1
AD12	+2.6	3.4 $\pm$ 0.2	3.4 $\pm$ 0.4

- Good agreement between measured fracture toughness and acoustically determined values

Summary and Conclusions

- Used nanoindentation to drive fracture in brittle thin films
- Related crack lengths with fracture toughness models to determine film toughness
  - ~3.5 $MPa \cdot m^{1/2}$
- Used acoustic monitoring of indentation process to measure released acoustic energy
- Related acoustic energy to film fracture toughness with good agreement

Sandia National Laboratories

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- Richard Nay

Others
- Jeremy Thurn (Seagate)

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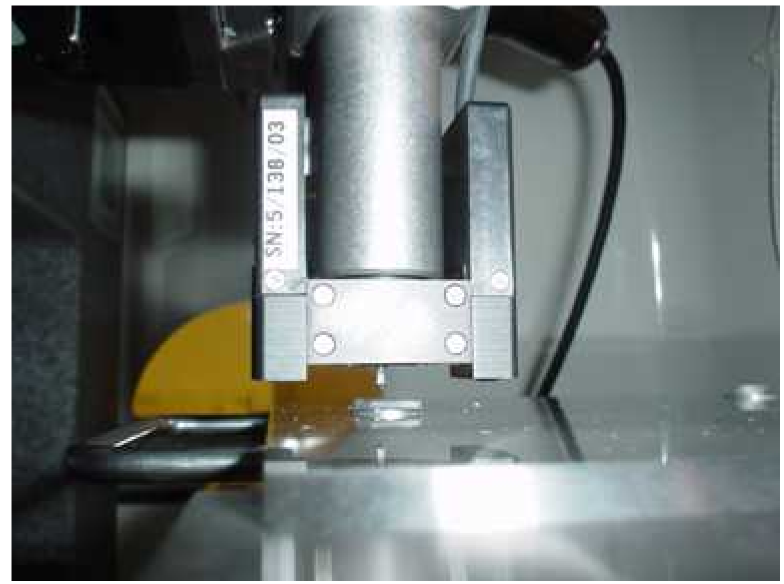
Hysitron TriboIndenter and TriboScope



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