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Predicting Fracture in Micron-scale Polycrystalline Silicon MEMS Structures

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Discovery at the Interface of Science and Engineering: Science Matters!

National Nuclear Security Administration

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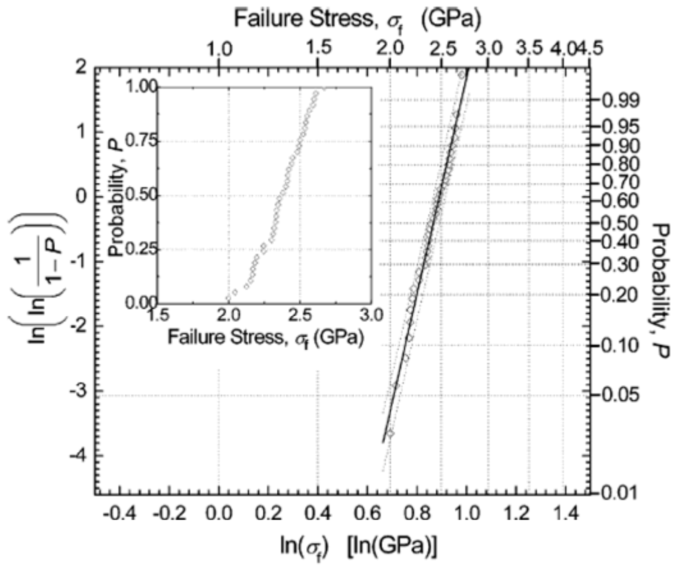
LABORATORY DIRECTED RESEARCH & DEVELOPMENT

Some Background

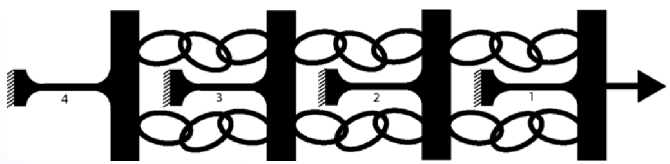


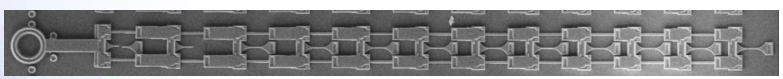
- State of the art: 10's of nominally identical MEMS tensile bars tested.
- For example: 37 tests of 2 x 2.25 x 150 µm tensile bars using "pull-tab" method (Boyce, et. al JMEMS 16, 2007).
- Significant variation in strength.
- Limited information on distribution tails.

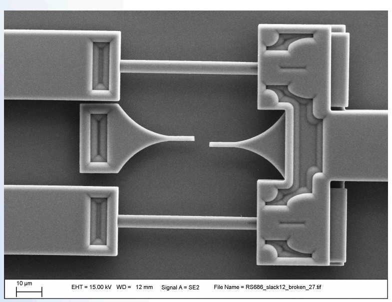
- Strength depends on size of highly stressed region.
  - Chasiotis, and Knauss, JMPS 51(2003), 1551-1572.
- Accuracy of a Weibull failure analysis appears limited.
  - Sharpe, Jadaan, Beheim, Quinn and Nemeth (2005), JMEMS14 (2005), 903-913.
  - McCarty and Chasiotis, Thin Solid Films 515 (2007), 3267-3276.
- SUMMiT V^{TM} side-wall flaws can be up to 90-nm deep and presumably linked to the strength distribution.
  - Boyce, Grazier, Buchheit and Shaw, JMEMS 16 (2007) ,170-190.

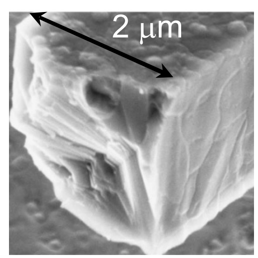
Developed Two High Throughput MEMS Tensile Test Methods

•Boyce’s Slack-Chain, Sequential Tensile Test method.







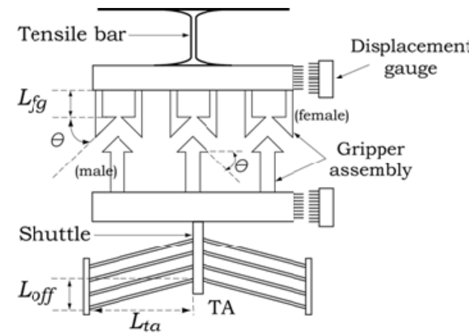


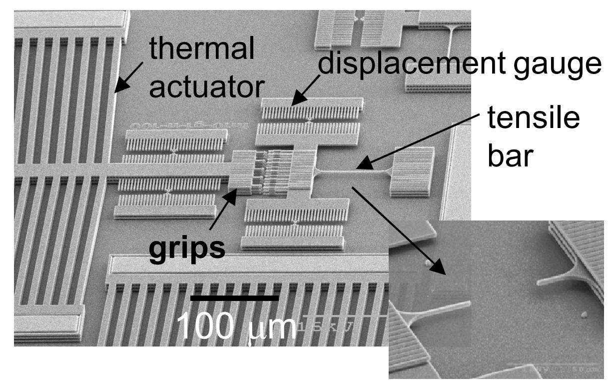
•20-µm gage length

•Measure breaking force.

(Boyce, Exp. Mech., 2010).

• De Boer’s On-Chip Tensile Tester





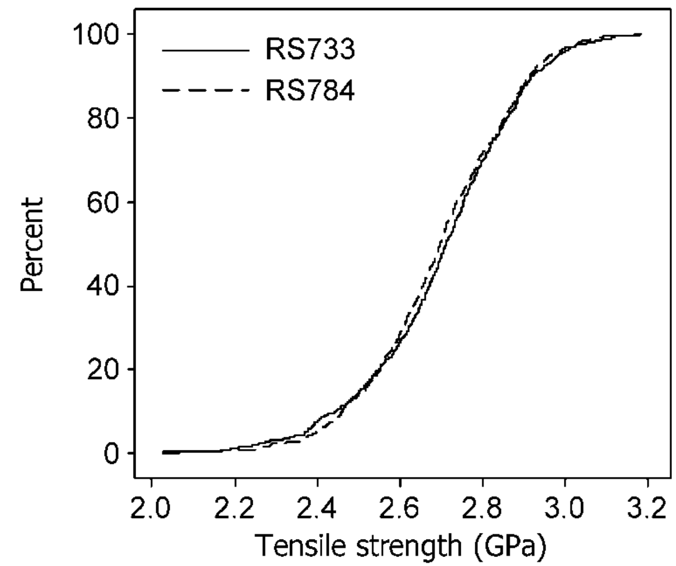
• Measure breaking end displacement.

• 70-µm gage length

(Hazra, Baker, Beuth, and deBoer, J. Micromech. Microeng, 19, 2009).

Slack-Chain RS733 and RS784 Poly3 Tensile Tests

- CDF of measured RS733 (n=616) and RS784 (n=671) tensile strengths suggest consistency of test method.
  - The two-sample Kolmogorov-Smirnov test (KS2) indicates that the null hypothesis that measured RS733 and RS784 tensile strengths are from the same population cannot be rejected at the 5% significance level.
  - Suggests Summit processing generates a consistent strength distribution.
  - Strength ranged from 2.0 to 3.2 GPa.



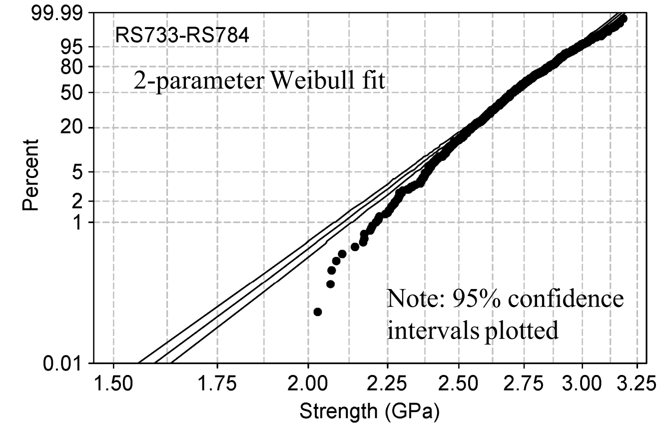
Note: all RS733 samples have a VSAM surface treatment while roughly half of the RS784 have a have a SCOO2 treatment instead.

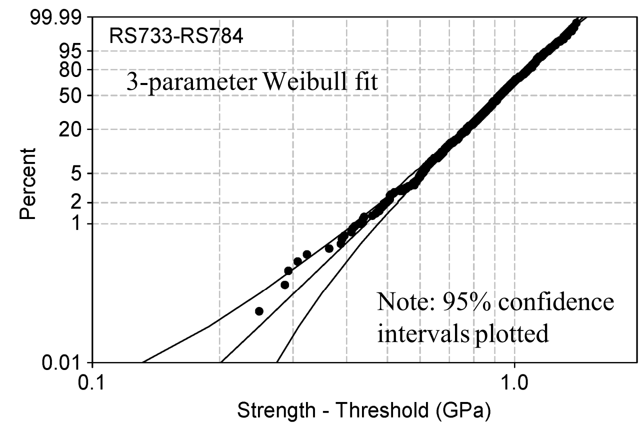
Slack-Chain RS733 and RS784 Poly3 Tensile Tests

- A 3-parameter Weibull distribution fits the tensile data better than a 2-parameter Weibull distribution.

$$P = 1 - \exp\left[-\left(\frac{\sigma - \sigma_u}{\sigma_\theta} \right)^m \right]$$

where σ is measured strength, m is the Weibull modulus, $\sigma_\theta + \sigma_u$ is the characteristic strength and σ_u is the threshold stress.

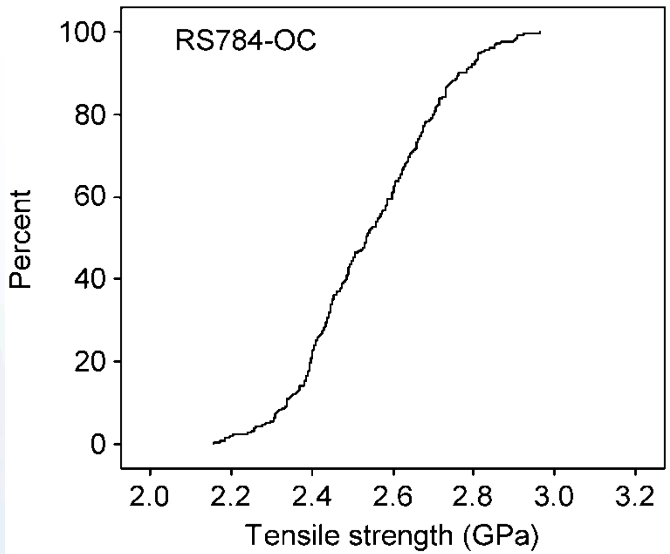


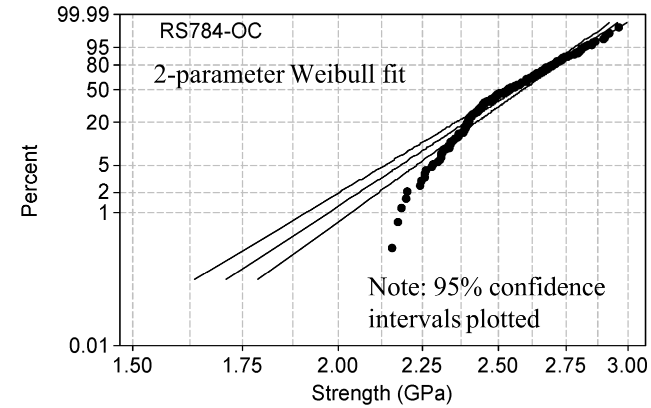


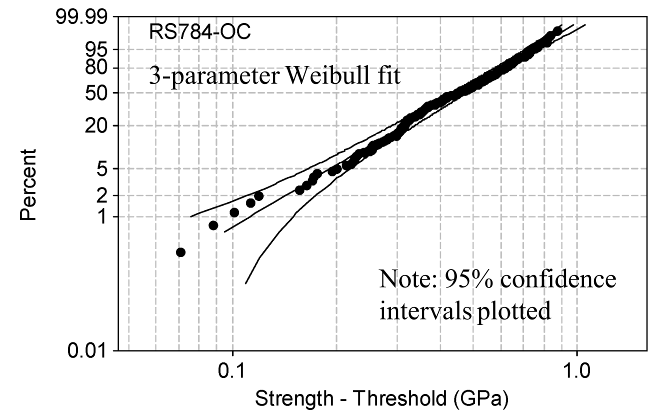
	n	m	m 95% CI	σ_θ (GPa)	σ_θ (GPa) 95% CI	σ_u (GPa)	σ_u (GPa) 95% CI
RS733	616	6.20	4.65-8.26	1.08	0.83-1.41	1.70	1.41-1.98
RS784	671	5.48	4.44-6.77	0.92	0.76-1.12	1.84	1.67-2.01
RS733-RS784	1287	5.78	4.85-6.88	0.99	0.84-1.16	1.78	1.62-1.94

On-Chip RS784 Poly3 Tensile Tests

- CDF and 3-parameter Weibull distribution fit.







	n	m	m 95% CI	σ_θ (GPa)	σ_θ (GPa) 95% CI	σ_u (GPa)	σ_u (GPa) 95% CI
RS784-OC	231	3.03	2.39-3.84	0.52	0.43-0.62	2.08	1.99-2.17

Comparison of Slack-Chain and On-Chip Tensile Data

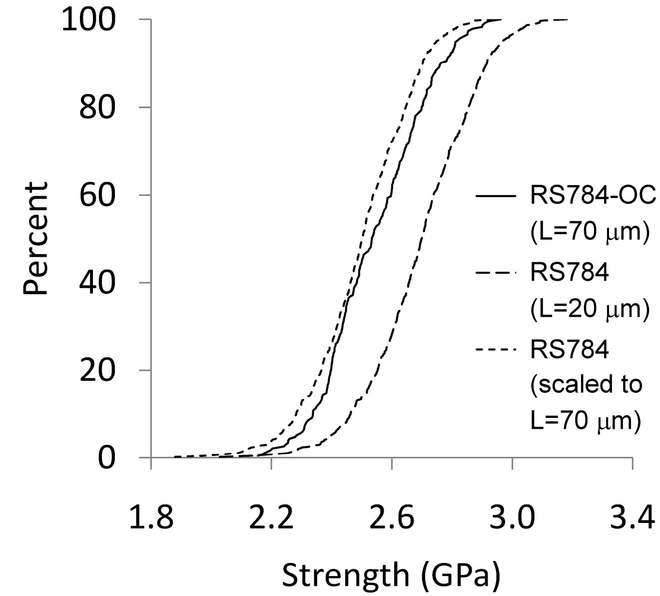
- Cannot directly compare slack-chain and on-chip tensile test results since they have different gage lengths, *L*.

- Within 2-parameter Weibull framework can estimate strength variation with gage sidewall area $A = 2Lh$ (location of strength controlling flaws; *h* is specimen thickness)

$$\frac{\sigma_2}{\sigma_1} = \left(\frac{A_1}{A_2}\right)^{1/m}$$

where σ_i is the tensile strength for specimen with sidewall area A_i, *m* is the Weibull modulus, and where both strength values are for the same percent of failures.

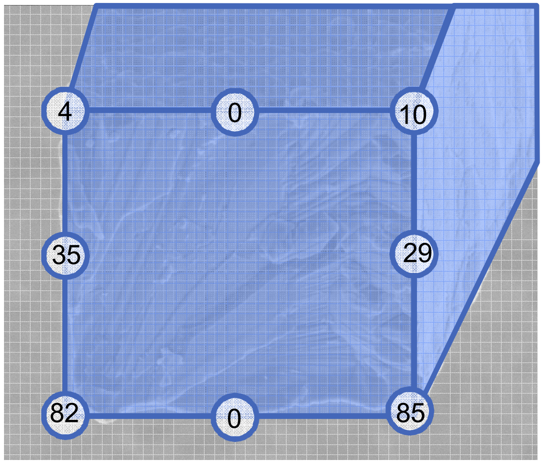
	n	m	L (μm)	avg (GPa)	st. dev. (GPa)	min (GPa)	max (GPa)
RS784	671	16.8	20	2.70	0.18	2.03	3.18
RS784-OC	231	16.1	70	2.54	0.17	2.15	2.96



Scaling RS784 CDF (L=20 μm) by the factor $(20/70)^{1/16.5}$=0.93 to estimate its CDF for L=70 μm brings into good agreement with RS784-OC CDF (L=70 μm) .

Location of Controlling Flaws

- Strength controlling flaws are limited to sidewall.
  - SEM of over 200 fractured tensile bars showed failures initiated along sidewalls, with a clear preference for origins at bottom corners.
  - no failures initiated in middle of the top or bottom surfaces.
  - AFM and TEM images indicate that sidewall edge flaws are V-notches and are associated with preferential etching of sidewall grain boundaries.
- Variation in edge flaw depth is presumed to be a major source of the variation in measured tensile strength.



Other Potential Sources of Variability in Tensile Strength

Line Width

- Line width *w* shows variability within a RS and between RS.
- Measurements suggest that a variation in *w* within and between RS could generate ~ 10% variation in strength when an average *w* is used to compute stress from failure load.

	n	min	max	avg	st dev
RS733	77	1.66	1.82	1.74	0.04
RS784	73	1.83	1.91	1.88	0.02

w (μm) based on 3-4 measures/sample

- Found to be associated with position of die within wafer.
- Must be accounted for when reduce Slack-Chain tensile test data.

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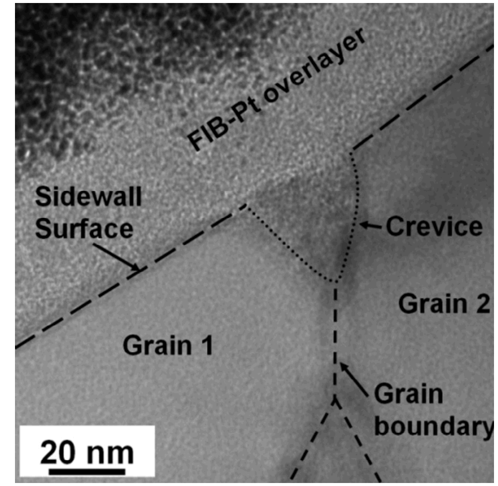
LDRD LABORATORY DIRECTED RESEARCH & DEVELOPMENT

Other Potential Sources of Variability in Tensile Strength: Stress inhomogeneity in a polycrystal

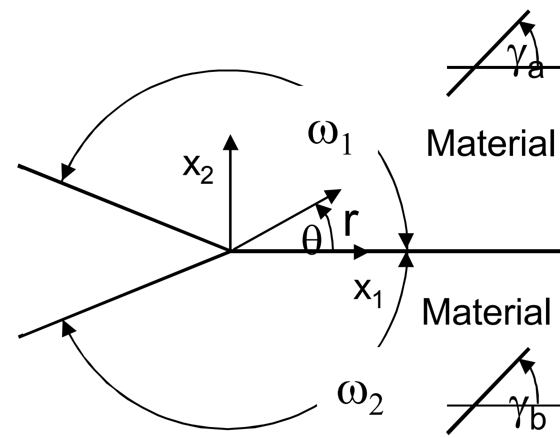


- Could edge notches with same geometry have notch-tip stresses that varied significantly with crystal orientation?
- Use Stroh's formalism for anisotropic elasticity to determine strength of singularities at the tip of an edge V-notch that is bounded by two crystals:

$$u(\theta) = \sum_{\omega=1}^{2} \left(q_\omega a_\omega z_\omega^{\lambda+1} + h_\omega \bar{a}_\omega \bar{z}_\omega^{\lambda+1} \right) / (\lambda + 1)$$

$$t(\theta) = \sum_{\omega=1}^{2} \frac{1}{r} \left(q_\omega b_\omega z_\omega^{\lambda+1} + h_\omega \bar{b}_\omega \bar{z}_\omega^{\lambda+1} \right)$$

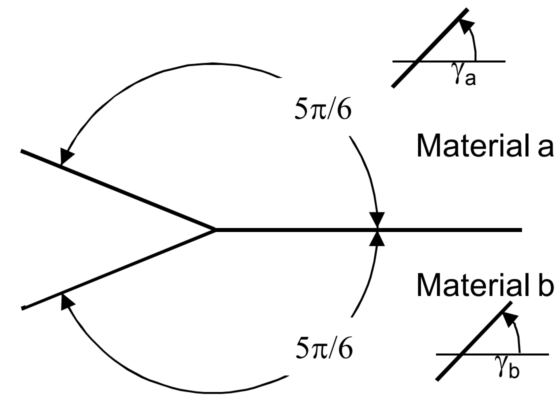
where q_w and h_w are arbitrary complex constants and $z_\omega = x_1 + p_\omega x_2 = r(\cos(\theta) + p_\omega \sin(\theta))$.
The scalars p_ω and the vectors $\boldsymbol{a}_\omega$ and $\boldsymbol{b}_\omega$ are the eigenvalues and the associated eigenvectors from a problem that depends on crystal stiffness



$$\sigma_{ij} = K_{a1} r^{\lambda_1} f_{ij1}(\theta) + K_{a2} r^{\lambda_2} f_{ij2}(\theta) \quad (i,j = r,\theta)$$

Solution for Strength of Singularities at the Tip of an Edge V-notch that is Bounded by Two Crystals: Use Stroh's formalism for anisotropic elasticity

- Tabular results for the strength of the more singular term (λ_1).
  - Results for a V-notch between two silicon crystals, with $\omega_1 = -\omega_2 = 5\pi/6$, (60^o wedge angle).
  - Normalized by the corresponding strength of the stress singularity at the tip of a V-notch in a homogeneous, isotropic material (λ_1=-0.488).



	γ_b					
γ_a	0	15	30	45	60	75
0	1.0005	1.0020	1.0018	1.0002	0.9985	0.9987
15	0.9987	1.0003	1.0001	0.9983	0.9964	0.9966
30	0.9985	1.0002	1.0000	0.9981	**0.9962**	0.9964
45	1.0002	1.0017	1.0016	0.9999	0.9981	0.9983
60	1.0018	1.0032	1.0031	1.0016	1.0000	1.0001
75	1.0020	**1.0033**	1.0032	1.0017	1.0002	1.0003

Min and max values of λ_1 shown in bold

Other Potential Sources of Variability in Tensile Strength: Stress inhomogeneity in a polycrystal

- Investigated notch-tip stress fields in a tensile bar where explicitly model notch-tip grains.
  - 60^o sharp V-notch
  - a=50 nm, h=200 nm
  - bar subjected to 1% nominal strain (1.6 GPa)

– when $\gamma_a=45^o, \gamma_b=0^o$

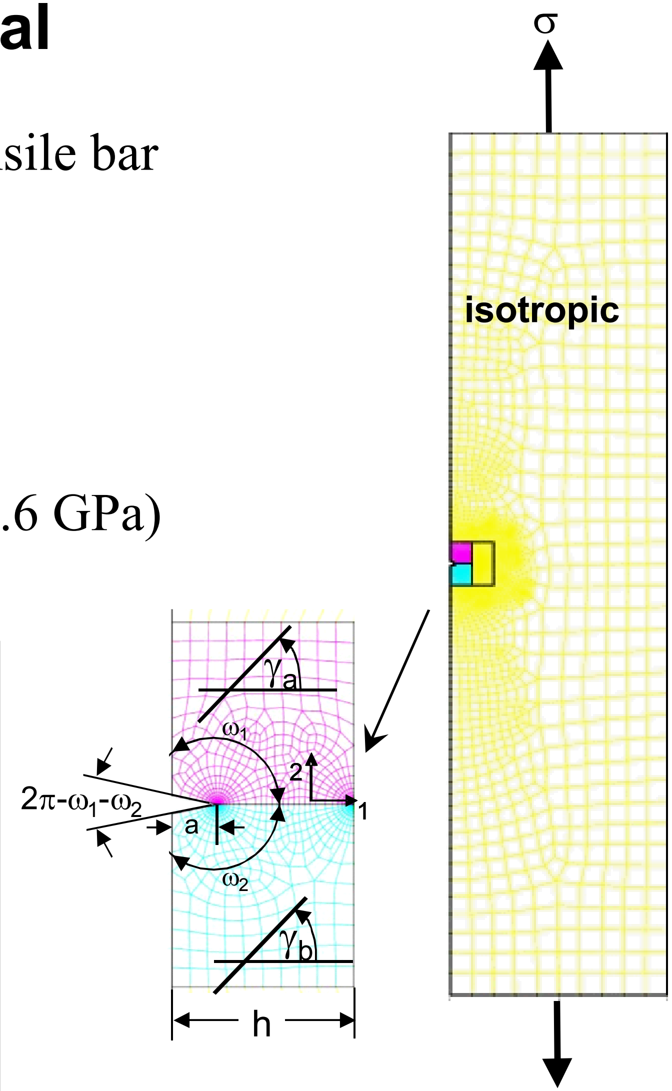
$$\sigma_{22}=9359r^{-0.486}$$

–when $\gamma_a=45^o, \gamma_b=60^o$

$$\sigma_{22}=9763r^{-0.485}$$

– edge crack in an isotropic material has

$$\sigma_{22}=9206r^{-0.500}$$



=> Stress fields similar to sharp crack in an isotropic material!

Other Potential Sources of Variability in Tensile Strength: Apparent Edge Flaw Fracture Toughness

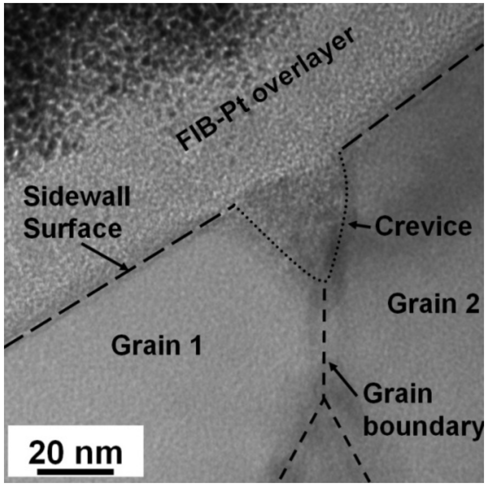
- Single crystal silicon toughness varies with cleavage plane.
  - Cleavage anisotropy is modest.
  - K_{IC} =0.82-0.95± 0.1 MPa-m$^{1/2}$

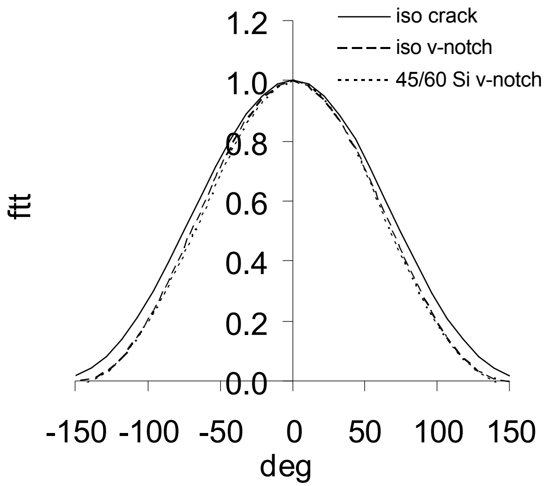
Plane	K_{IC} (MPa-m$^{1/2}$)	source
(111)	0.82±0.07	Chen
(110)	0.90±0.11 0.91±0.09	Chen Yasutake
(100)	0.95±0.05 0.95±0.10	Chen Yasutake

Chen, et.al., ACS Bull 59: 469-472.; Yasutake, et.al., "JMS 21: 2185-2192.

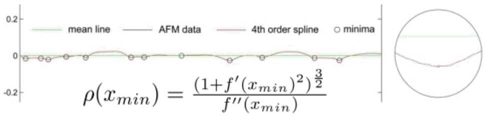
- Variability introduced when cleavage plane is not aligned plane of peak cleavage stress.

- For a 60^o V-notch
  - Cleavage stress ↓ 6% over 45^o notch-tip sector
  - Cleavage stress ↓ 11% over 60^o notch-tip sector

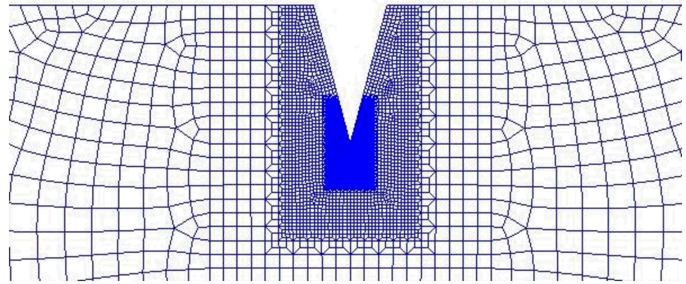




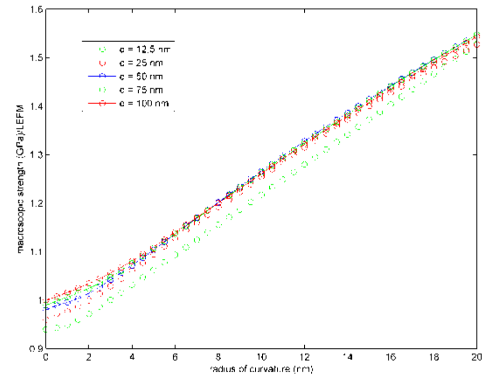
CZ FEA results applied to AFM images to predict strength



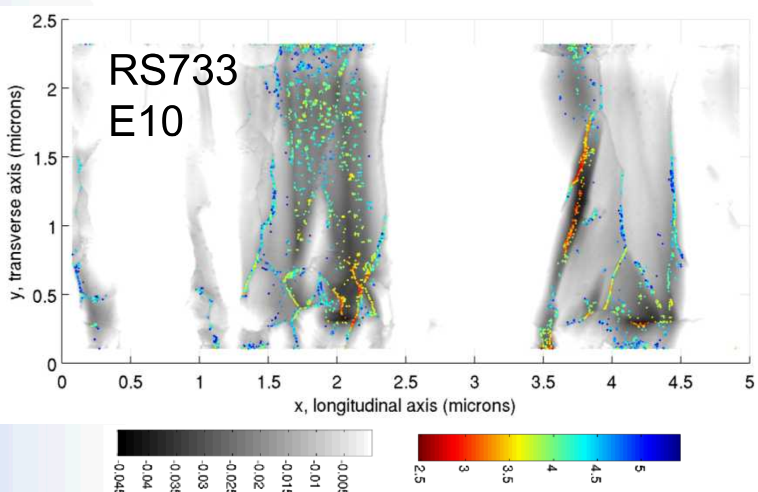
fit AFM image to determine depth a root radius



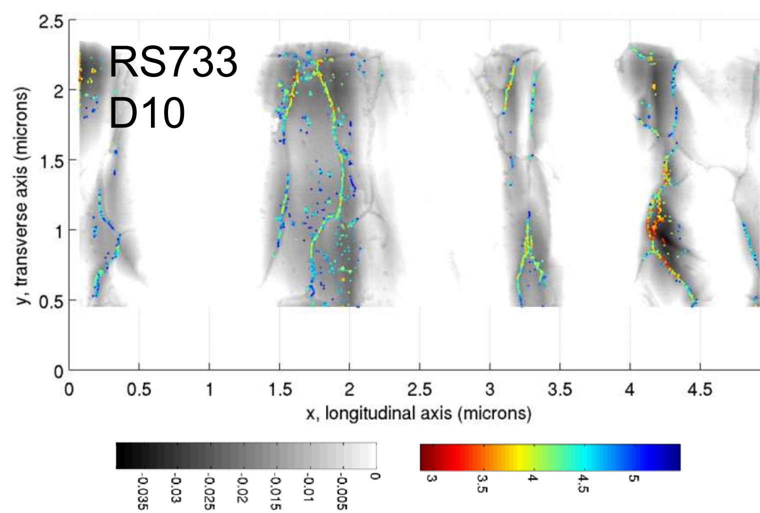
analyzed blunted notches



determined deviation from LEFM



depth (μm) strength (GPa)



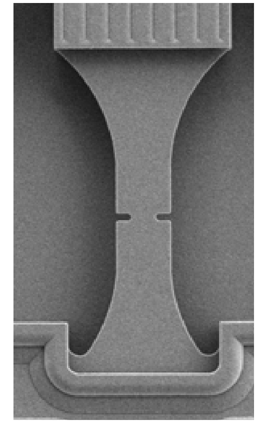
depth (μm) strength (GPa)

depth > 15 nm, curvature > 0.075 1/nm

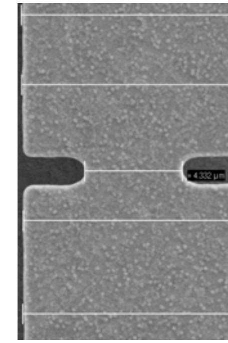
RS733 Poly3 Double Edge-Notched (DEN) Specimen

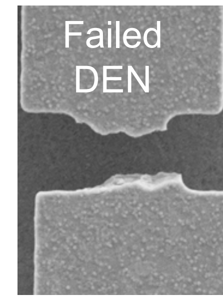
- Tested DEN specimens to investigate effect of micron-scale stress concentrations.
- Slack-chain sequential test method used to test 331 Poly3 DEN specimens.
- Performed linear elastic finite element analysis of the Poly3 DEN specimen.
  - notch tip stress concentration factor is 2.75 (applied to net-section stress).
  - large stress gradients over distances of ~0.1 μm.
- SEM images of 40 DEN specimens showed:

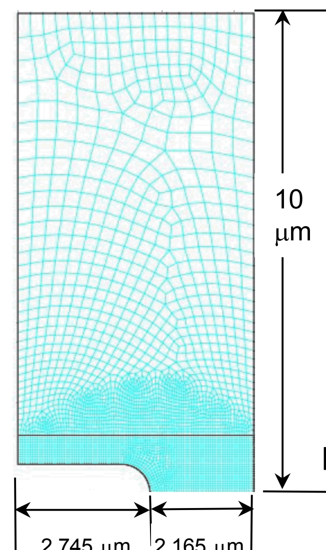
	avg (μm)	max (μm)	min (μm)
Ligament width	4.33	4.28	4.37
Radius	0.58	0.54	0.64



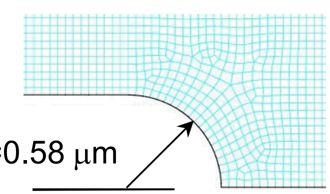
Poly3 layer
2.33 mm thick





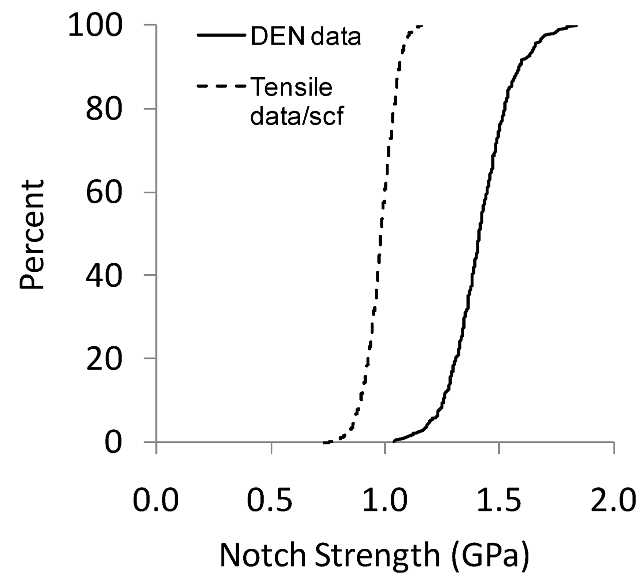


- Quarter FEA model with symmetry conditions imposed.
- Top edge uniformly displaced upward.



RS733 Poly3 Double Edge-Notched (DEN) Specimen

- If assume failure occurs when local stress at the notch tip reaches a critical tensile value, can estimate DEN strength distribution directly from tensile strength distribution.
  - DEN ligament stress at failure = tensile strength/SCF.
  - recall SCF for this DEN is 2.75.
  - Based on 95% CI for RS733-RS784 tensile strength data, predicted threshold strength for DEN specimen fails in the range of 0.59-0.71 GPa
  - ~3 flaws subjected to a high stress in a DEN specimen while ~100 flaws in tensile specimens subjected to a high stress.



Note: notch strength is defined as the average ligament stress at failure.

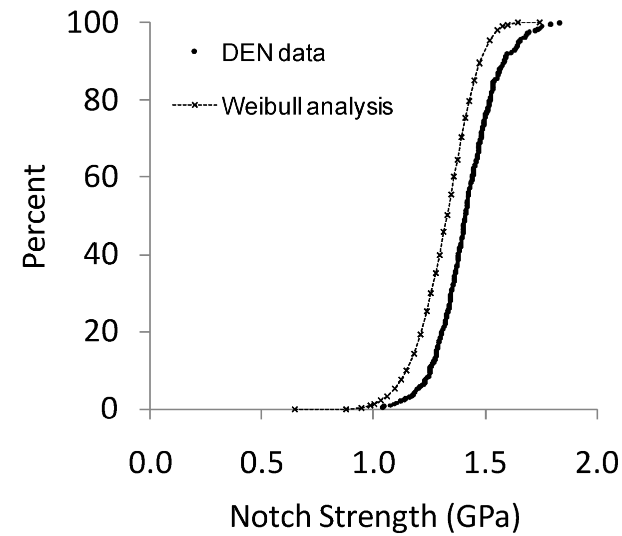
	n	min (GPa)	max (GPa)
Tensile	1287	2.03	3.18
DEN	331	1.04	1.83

Weibull Failure Analysis of RS733 Poly3 DEN Specimen

- For a 3-parameter Weibull distribution that is generalized to a non-uniform stress field, calculate failure probability is:

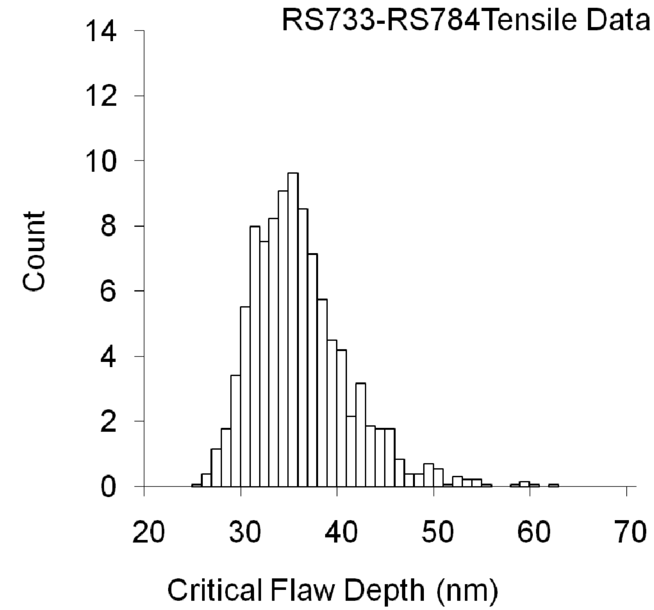
$$P = 1 - \exp\left[-\int_A \left(\frac{\sigma - \sigma_u}{\sigma_o} \right)^m dA \right]$$

- Integrate maximum principal stress over all tensile regions of the DEN specimen's sidewall surface.
  - Only significant contributions to integral occurs over an ~ 60^o segment at notch tip.
- Weibull analysis underestimates strength.
  - Predicted threshold strength is only 0.65 GPa.
- Weibull analysis assumes that a representative population of flaws lie within region << dimensions, stress gradients. Not true.
- Measured tensile Weibull distribution is biased towards bigger critical flaws.



- Used Weibull parameters determined from tensile strength data (RS733-RS784 composite) in conjunction with DEN stress state determined by FEA.
- In highly stressed region, characteristic element length scale is 0.025 μm (essentially same result when 0.050 μm).

Lower Bound Estimate Using LEFM Flaw-Tolerance Approach



- Assume the largest critical flaw that could ever exist is located at the most highly stressed region and normal to the first principal stress.
- Estimate critical flaw depth from tensile strength data using LEFM.
  - assume crack-like edge flaws, K_{IC}=1 MPa-m$^{1/2}$, and use K_I calibration for a small edge crack

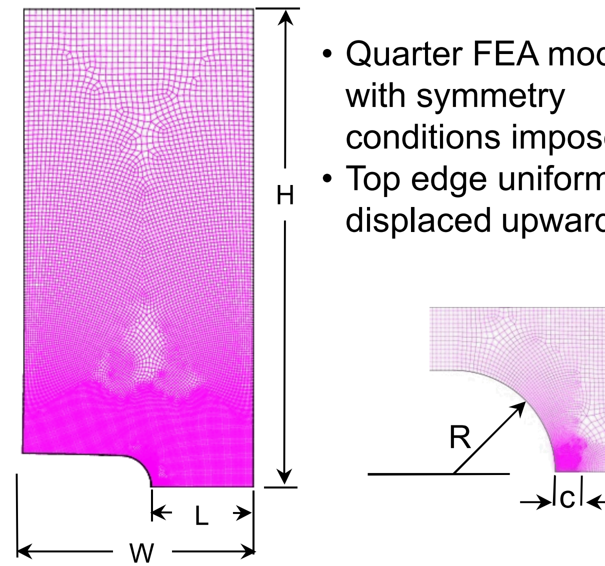
$$c_{cr} = \frac{1}{\pi}\left(\frac{K_{IC}}{1.122\sigma_f}\right)^2$$

where c_{cr} is the critical flaw depth and σ_f is tensile strength.

  - calculated c_{cr} is an "effective" crack depth: depends on ratio of K_{IC}/σ_f, also reflects variations in flaw geometry, etc.

A threshold tensile strength implies the existence of a max critical flaw depth.
- σ_u =[1.62-1.94 GPa]; 95% confidence interval for RS733-RS784
- based on σ_u, the max c_{cr} falls within the range of 67-96 nm.

Lower Bound Estimate for Strength of Structure with a Stress Concentration (DEN)



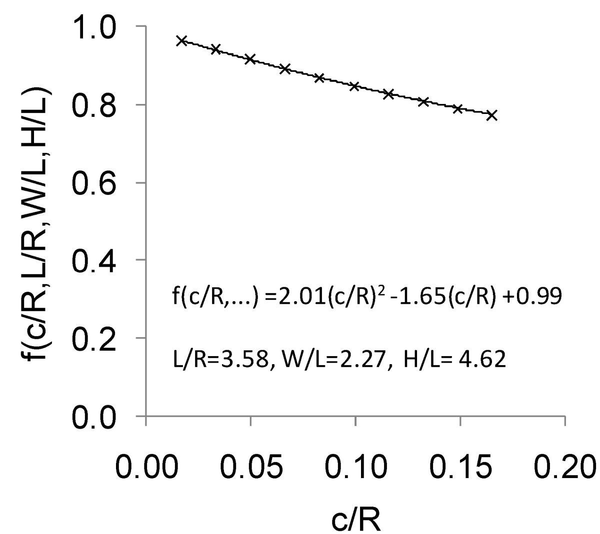
- Quarter FEA model with symmetry conditions imposed.
- Top edge uniformly displaced upward.

- Use K_I calibration for RS733 Poly3 DEN geometry with K_{IC}=1 MPa-m$^{1/2}$.

$$K_I = 1.122 k \sigma_{lig} \sqrt{\pi c}\, f(c/R, L/R, W/L, H/L)$$

where k is the notch's SCF and σ_{lig} is the average ligament stress.

- For c_{cr}=67-96 nm, the predicted net section notch strength σ_n= 0.76-0.85 GPa.
- Minimum σ_n measured in 331 DEN tests is 1.04 GPa.



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Summary

- Developed novel test methods that enabled measurement of tensile strength tails.
- Data indicates that there is a tensile strength threshold that implies the existence of a maximum flaw size.
- Quantified sources of strength variations. Variation in depth of edge flaw appear to dominate.
- CZ FEA in combination with AFM images of sidewalls predicts tensile strength that falls within range of those observed.
- Used a fracture mechanics "flaw tolerance" design approach based on tensile strength data to predict a lower bound estimate for DEN specimens.

(Accepted for publication in Journal of Microelectromechanical Systems 4/13/11)