

Making the Most of Our Failures

Richard E. Tressler Lecture in Materials
Penn State University
Apr. 21, 2016



S. Jill Glass
Power Sources Component Development Dept.
Sandia National Labs
Albuquerque, NM 87185

sjglass@sandia.gov





1984 – First Visit to Penn State

Penn State vs. U. of Penn.

Tressler Presentation at U. Penn in 1988

Environmental Degradation of Ceramics Materials:
Richard E. Tressler, Ceramic Science & Engineering
Program, Materials Science and Engineering
Department, Penn State; 4 p.m. Auditorium.

<http://www.upenn.edu/almanac/v29pdf/n13/113082.pdf>



Fractography of Glass, Edited by Richard C. Bradt and Richard E. Tressler, 1994

To introduce these articles, it is appropriate to briefly discuss **fractography** and to also address the issue of why glass is the focus of these articles and this monograph. Perhaps part of the reason is because fractography is concerned with the phenomenon of fracture, and glass, having one of the lowest fracture toughness values (resistance to fracture) of all materials, is particularly prone to fracture or break. **Of course, glass has been prone to breakage ever since the first glasses were produced many centuries ago.**

The word fractography does not appear in Webster's finest. It probably originates from a combination of the two terms: fracture and topography, deriving from the Latin frangere and topographye.

Fractography is the study of the relief features or surface structure of fractured surfaces, usually from an after-the-fracture or post mortem perspective. **Most modern fractography has been pursued from the perspective of examining the broken or fractured artifact, to attempt to reconstruct the manner and to identify the reason why the object fractured.**



Outline

- Sandia National Labs
 - Overview
 - Education and Sandia Career
 - Materials and Process Sciences Center
 - Power Sources Technology Group
- Failure Ingredients
- Failure Examples
- Making the Most of Our Failures



Sandia National Labs is a Government Owned, Contractor Operated (GOCO), R&D Facility

Sandia Corporation Operates Sandia National Laboratories for the US Department of Energy

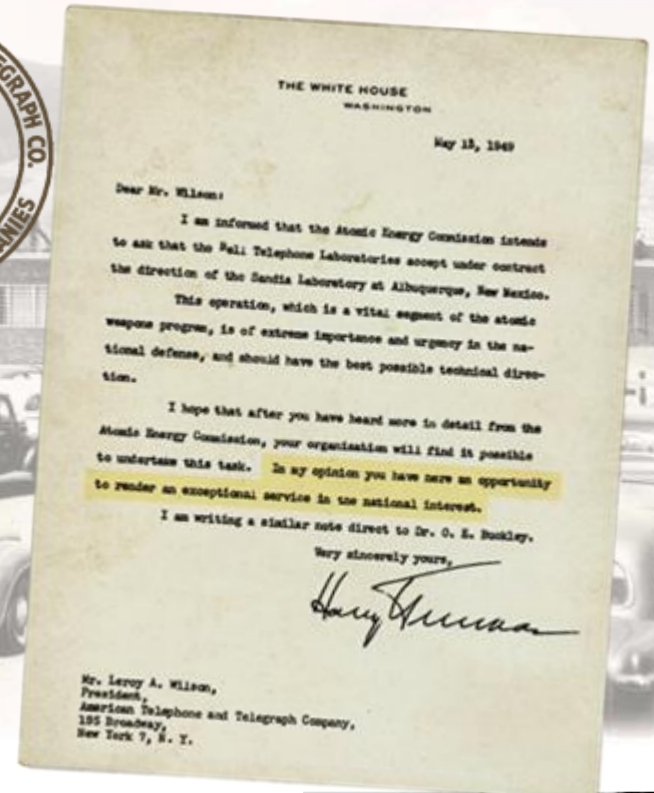
- AT&T: 1949–1993
- Martin Marietta: 1993–1995
- Lockheed Martin: 1995–present



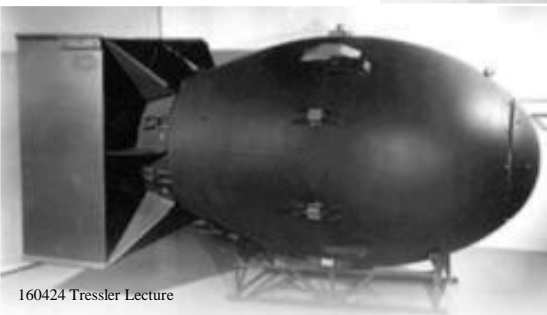
Sandia's History and Objective

Exceptional service in the national interest

- July 1945: Los Alamos creates Z Division
- Nonnuclear component engineering
- November 1, 1949: Sandia Laboratory established



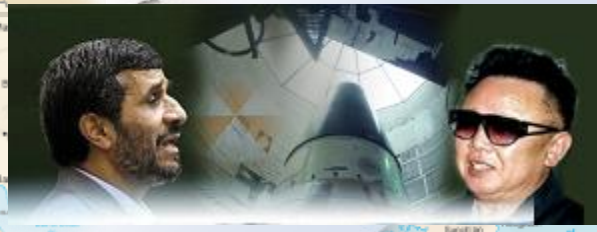
to undertake this task. In my opinion you have here an opportunity to render an exceptional service in the national interest.



Sandia's Mission Space Addresses our Evolving National Security Environment



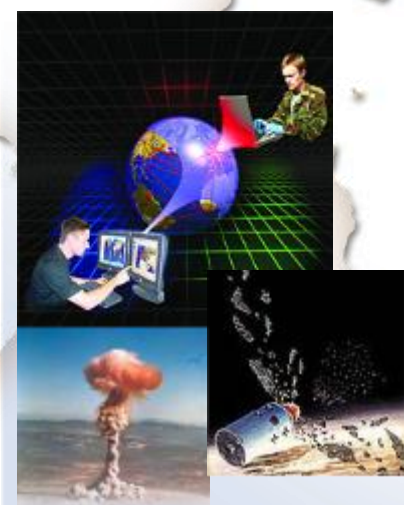
Traditional strategic nuclear threats



Threats from other nation states



Threats from non nation states



Threats of tech surprise

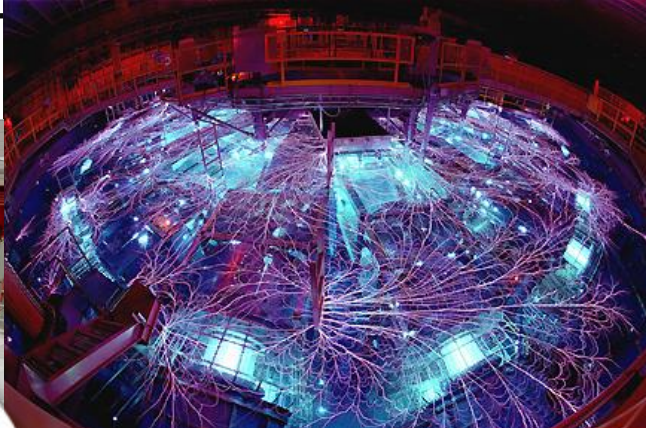


Other threats: natural disasters, climate change, energy supply

Sandia's Science and Technology Framework

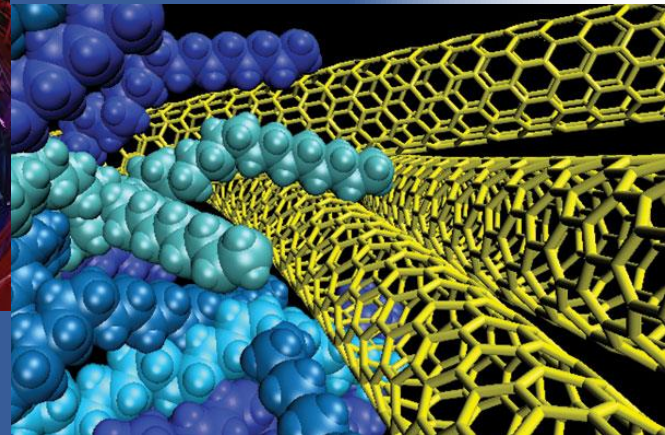
Strong research foundations play a differentiating role in our mission delivery

Computing & Information Sciences

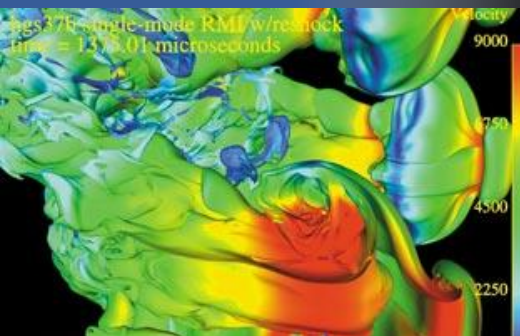


Radiation Effects & High Energy Density Science

Materials Sciences

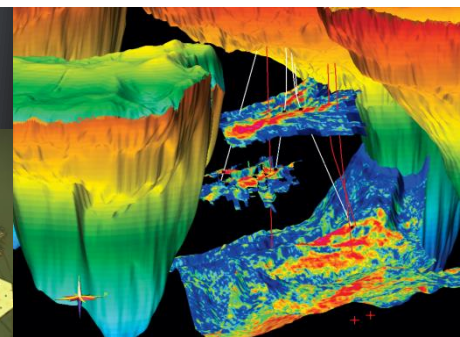


Engineering Sciences



Bioscience

Nanodevices & Microsystems



Geoscience

Sandia Sites

Albuquerque, New Mexico



Livermore, California



Kauai, Hawaii



*Waste Isolation Pilot Plant,
Carlsbad, New Mexico*

*Pantex Plant,
Amarillo, Texas*



*Tonopah,
Nevada*





Education and Sandia Career

Education

McMaster University – Ceramic Engineering (BS, 1984)

Penn State – Ceramic Science (MS, 1987 & PhD, 1990)

Career

Center 1800 – Materials & Process Sciences (1990-2015)

Group 1850 – Materials Aging and Reliability

Dept. 1852 – Materials Reliability

Center 2500 – Energetic Components (2015-present)

Group 2540 – Power Sources Technology Group

Dept. 2547 – Power Sources Component Development



Materials and Process Sciences R&D

Synthesis and Processing

Characterization

Performance and Reliability

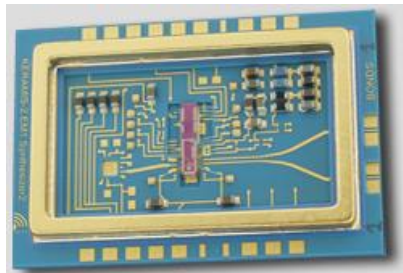
– Mechanical Performance

- Ductile Materials
- Brittle Materials

Brittle Material Containing Components

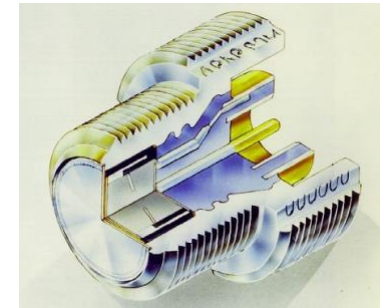
Materials

- 94% and 96% Al_2O_3
- Mo - Al_2O_3 Cermets
- Bulk Glasses and Glass Ceramics
- Glass Fibers
- Ceramic Ferrites
- PZT (lead zirconate titanate)
- PMN-PT (lead magnesium niobate - zirconate titanate)
- Barium Titanate
- ZnO Varistor Materials
- Polysilicon
- LTCC (low temp co-fired ceramic)



Components (Sandia and Externally Produced)

- Batteries and other Power Sources
- Slim Loop Ferroelectrics
- Rolamite Switches
- Neutron Sources
- Safe Enable Wheels
- Stronglinks
- Voltage Bars
- Current Stacks
- Discoidal Capacitors
- Electronic Substrates
- Clocks
- Photo Diodes
- Connectors





Sandia Products

Microelectronics

Explosives

Switch Tubes

Capacitors

Neutron Sources

Power Sources

- Thermal batteries
- Li primary and Li ion batteries
- Thermoelectrics

Power Sources Technology Group (PSTG)

- Scientific and engineering solutions to meet national needs for power sources in nuclear weapons, advanced conventional weapons, energy, and security applications.
- Primary customers are Department of Energy, Department of Defense, other government agencies, and industry.
- We are designated as the DOE back-up thermal battery production capability.



Production Dry-Room



Thermal, Li Primary, and Li Ion Batteries



Cell Prototyping



Battery Testing Including Battery Abuse Testing



Strength is Flaw Size Dependent for a Brittle Material

$$\text{When } \sigma_a \text{ or } \sigma_r > \sigma_f = \frac{K_{IC}}{Y \cdot \sqrt{c}}$$

Where:

σ_f = fracture strength

K_{IC} = fracture toughness or critical stress intensity

c = flaw size

Y = geometrical factor

σ_a = applied stress

σ_r = residual stress

Ingredients for Brittle Material Failure

When $\sigma_{\text{applied}} >$
or residual

$$\sigma_f = \frac{K_{IC}}{Y\sqrt{c}}$$

Poor Design - Excessive stress

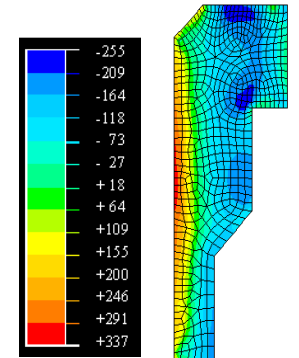
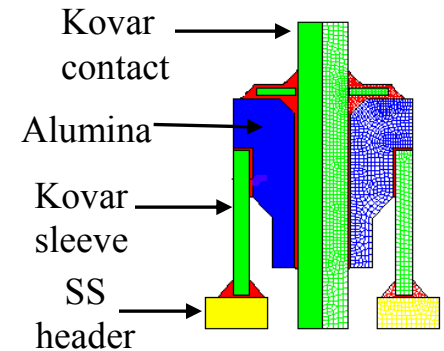
- Excessive applied/local stresses
- Materials combinations and/or processing that lead to excessive residual stress

Presence or introduction of defects

- Intrinsic microstructural features
- Processing related
- Machining, in-service damage

Material degradation

- Environmental effects, decrease in K_{IC}
- Increase in flaw size - subcritical crack growth

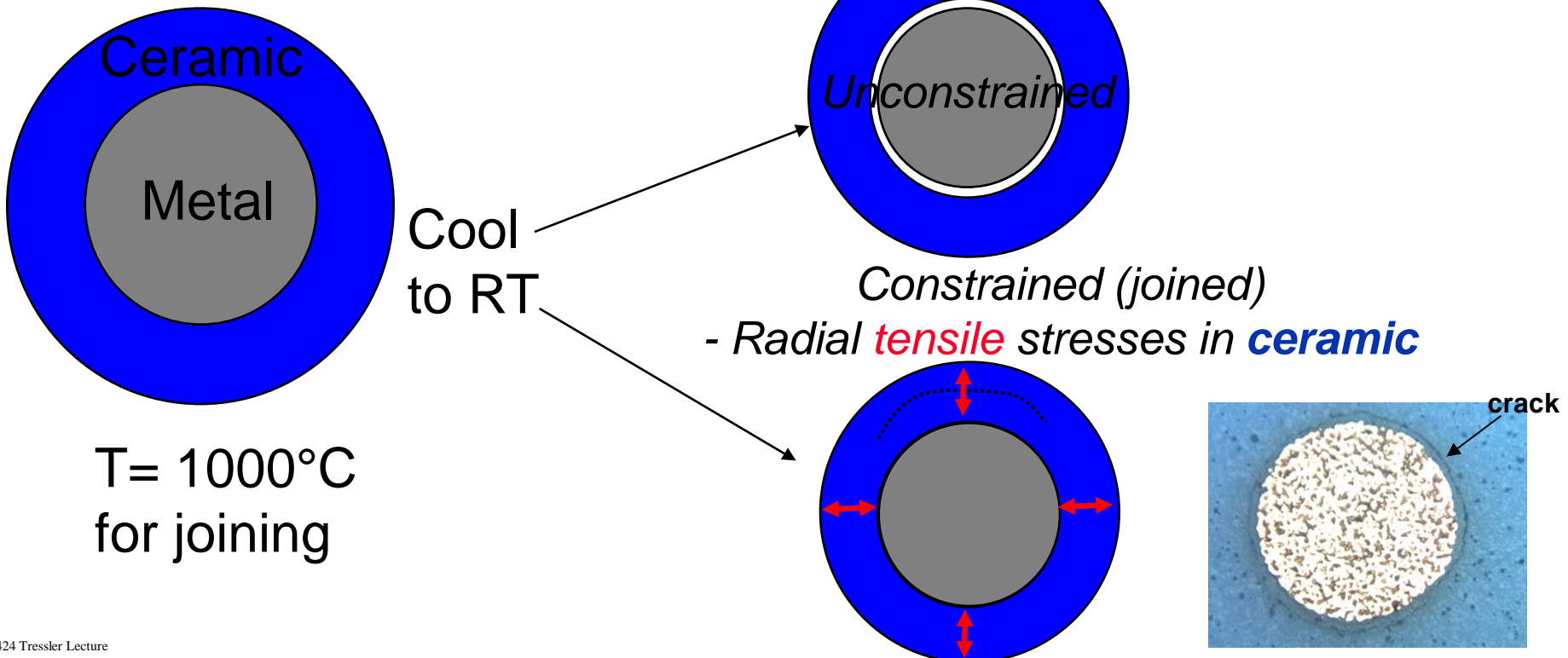


max. prin. stress
(MPa) in alumina

All of the above!

Thermal and Residual Stresses Can Produce Cracking of a Glass or Ceramic

- Uneven cooling produces thermal stresses
- Joining dissimilar materials produces residual stresses because of thermal expansion (CTE) mismatch



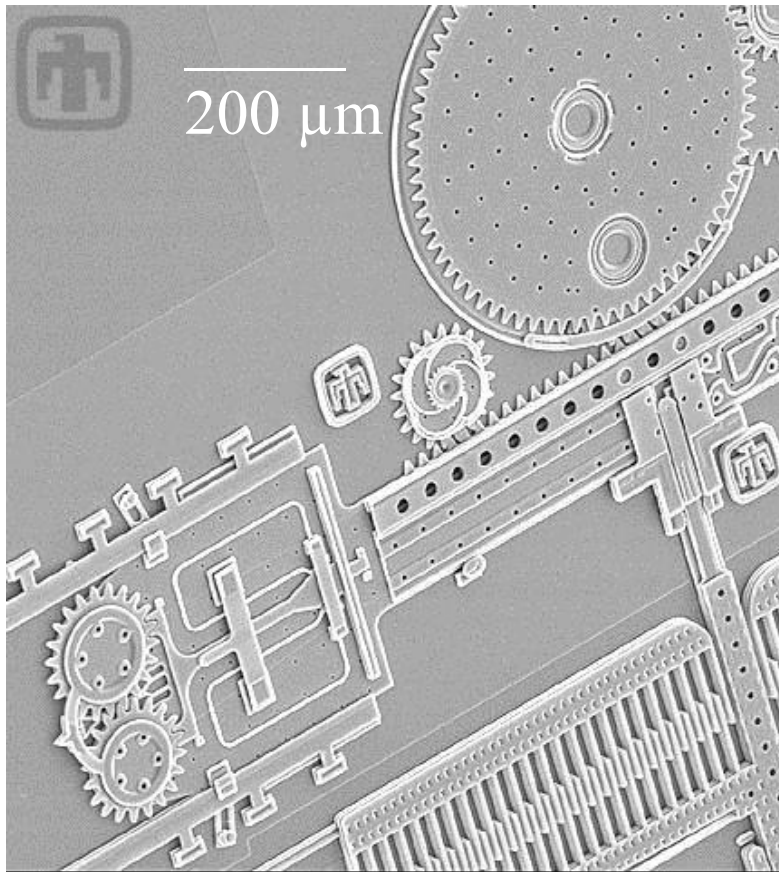


Brittle Materials Failure Examples

- MEMS polysilicon (Buchheit and Boyce)
- LTCC's with vias (Tandon, Monroe, Newton, Ewsuk)
- LTCC strength samples (Tandon, Monroe, Newton, Roth)
- ZnO varistors (Watson, Lockwood, and Diantonio)
- Pressurized sulfuric acid tank from industry (many)
- Ni lead failure (many)

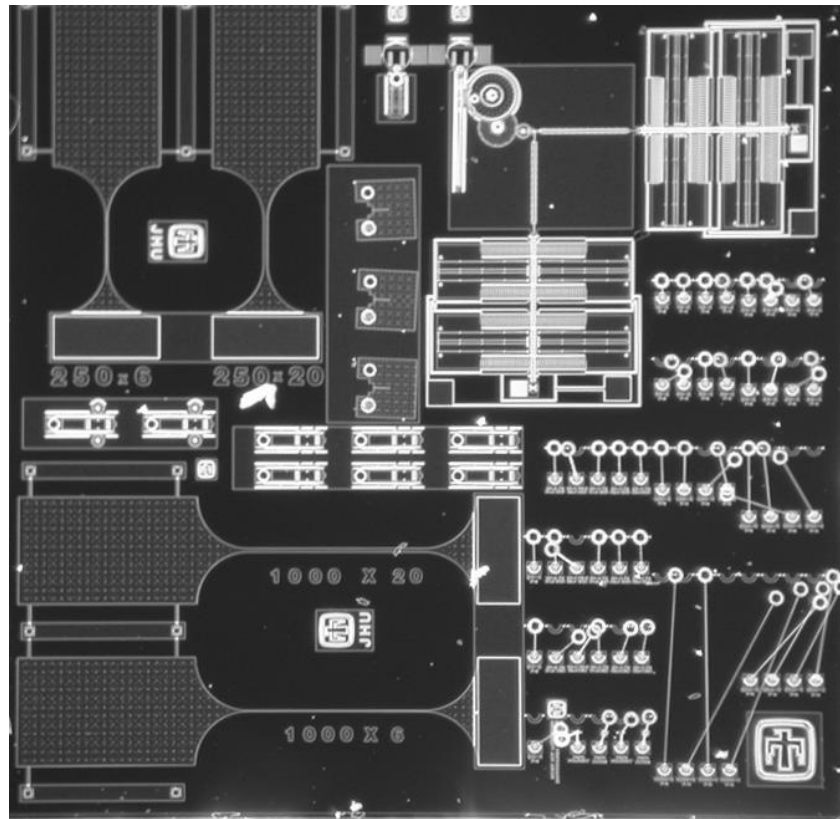
Fracture Strength Testing and Failure Origins for Polysilicon MEMS Materials

24-bit lock with 24-bit path, drive gears, anti-reversing gears, meshing gears, etc.

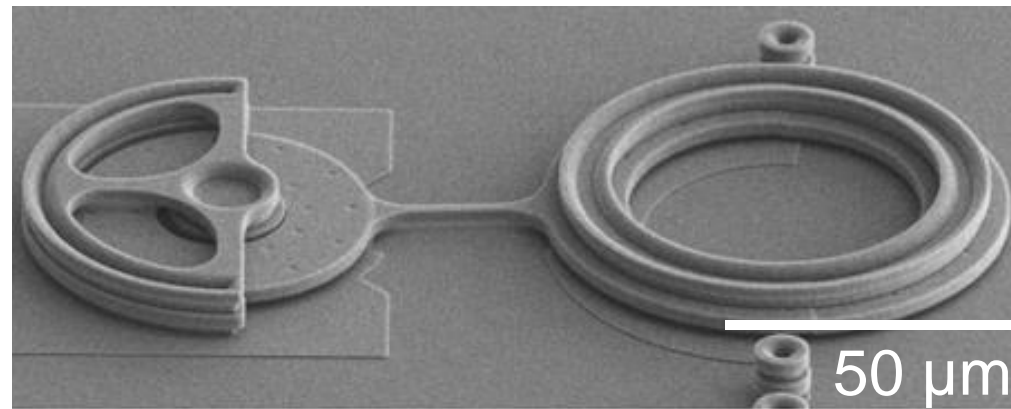


- Modest structural properties ($K_{IC}=1.0-1.4 \text{ MPa}\sqrt{\text{m}}$)
- High degree of reactivity with oxygen, water and other reagents

Polysilicon Mechanical Testing Samples Fabricated Directly on the Die

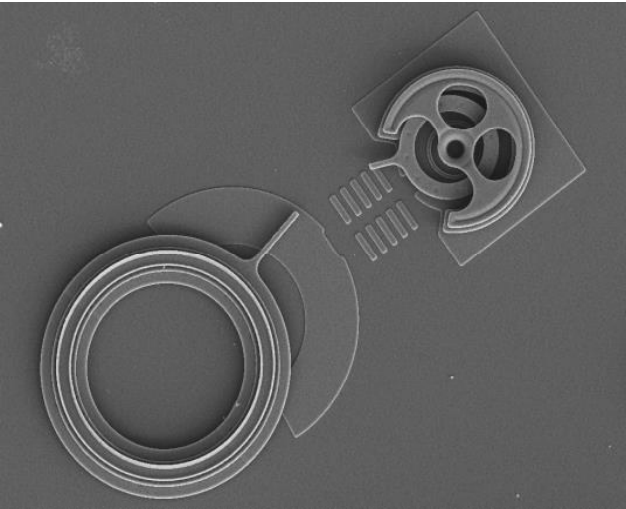


Pull-tab tensile strength sample.

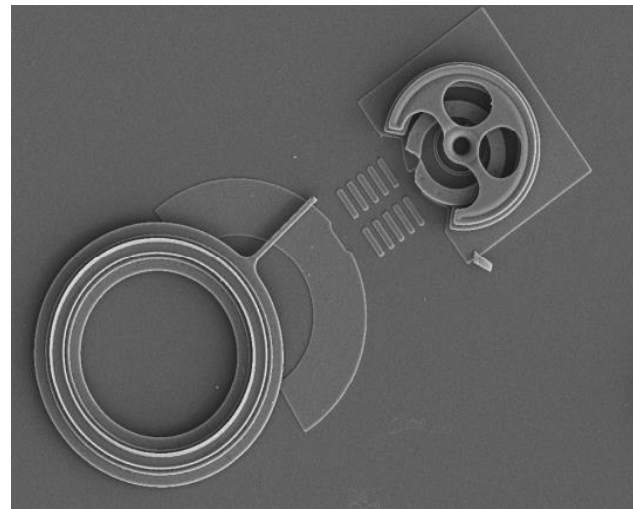


2 of 3 Failure Modes for 48 First Generation Polysilicon Samples Due to Pull-Tab Design

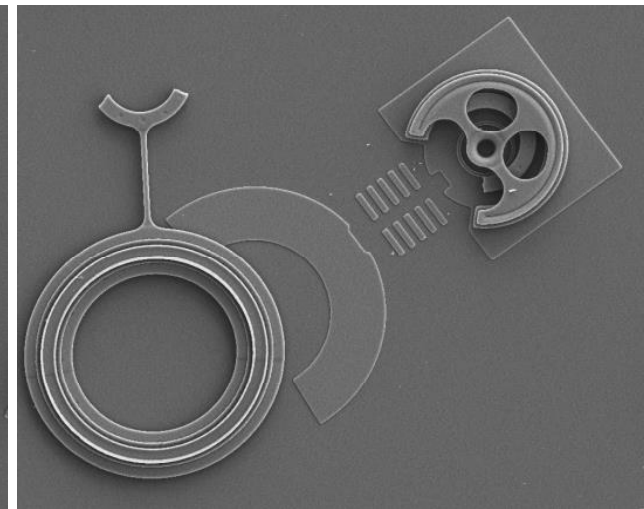
Average $\sigma_f = 2.24 \text{ GPa} \pm 0.35$



7 Strongest

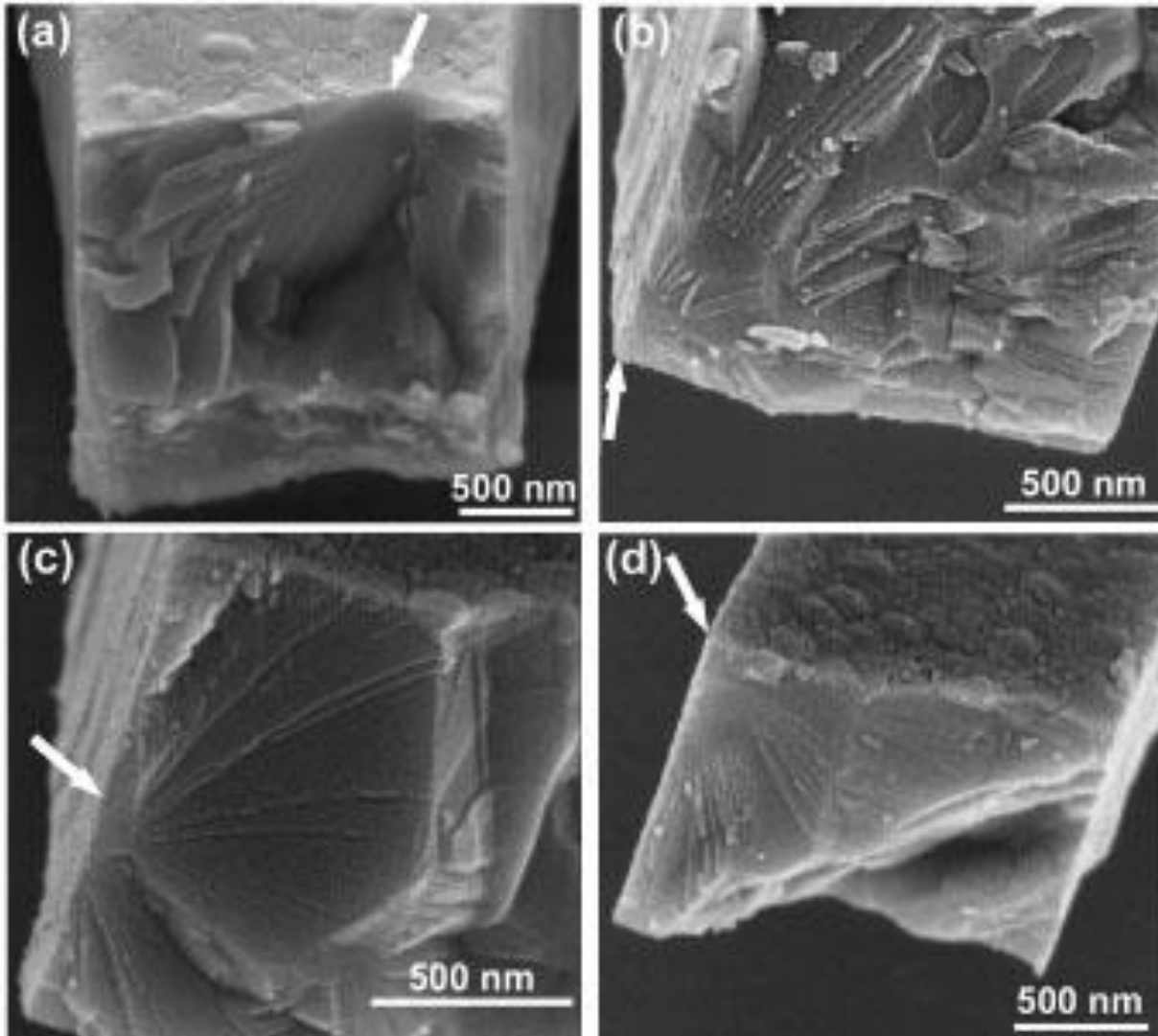


Intermediate Strength



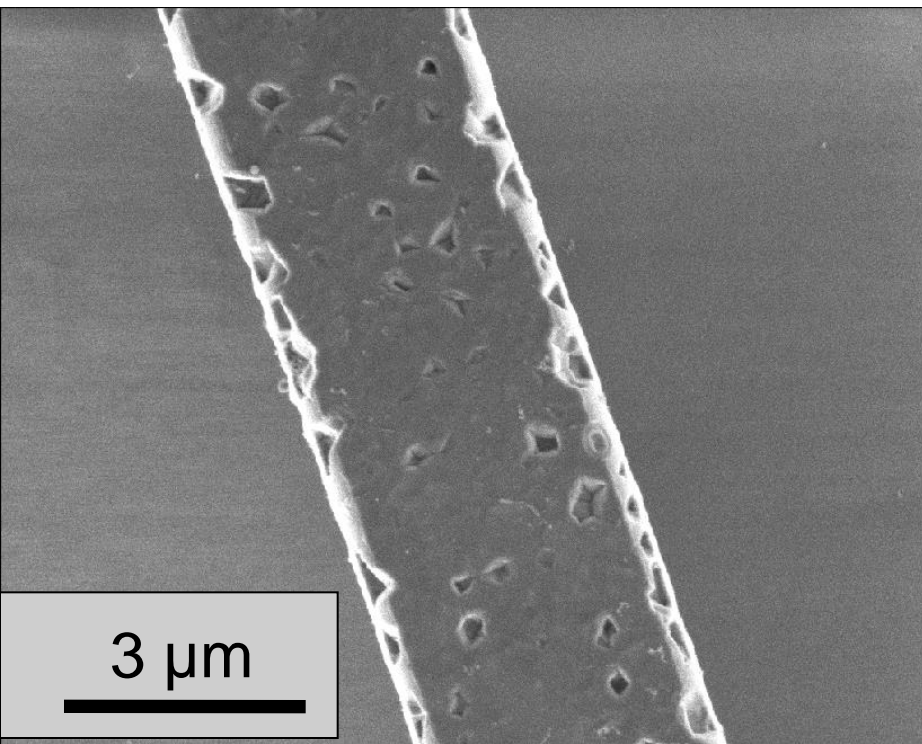
Weakest

Failures Due to Both Etching Flaws (a & c) and Corner Stress Concentrations (b & d)

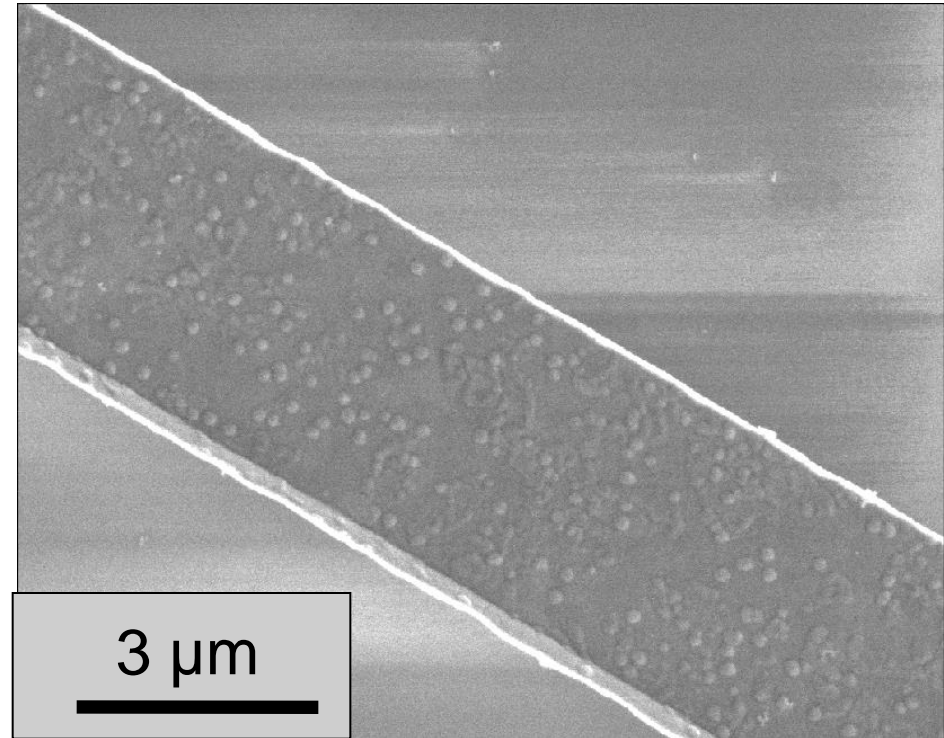


B. Boyce,
Sandia, 2005

Strengths Are Sensitive to Surface Condition (Etching Flaws) for Different Layers

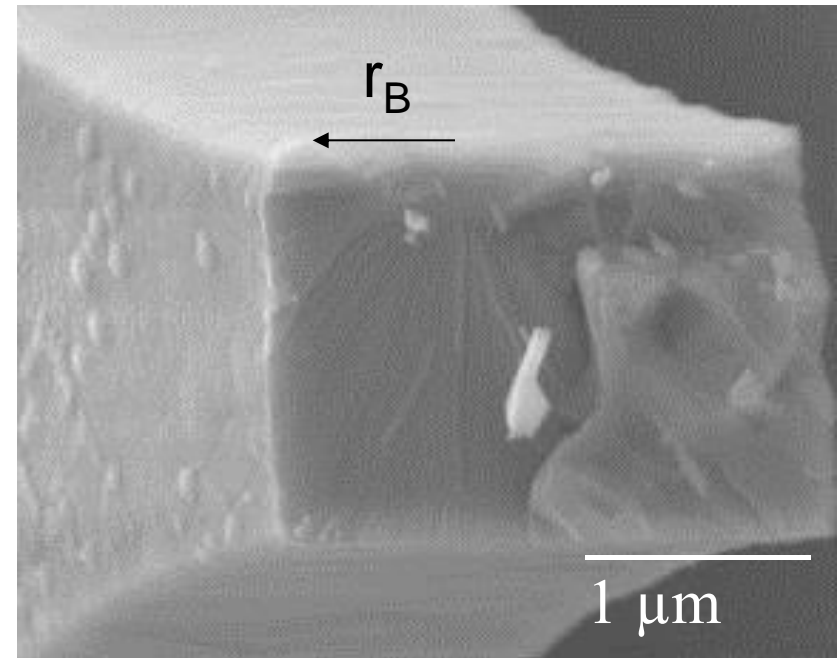
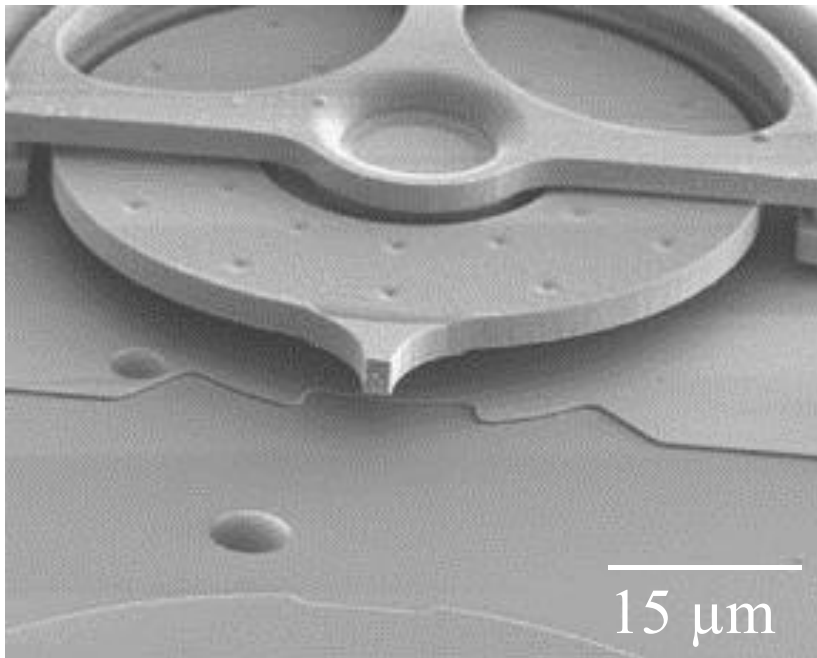


Weak



Strong

Strength Can be Estimated from Fracture Branching Boundaries on Fracture Surface



$$\sigma_f Y r_B^{1/2} = \text{constant} = K_B$$

$$K_B = 2.0 \text{ MPa}\sqrt{\text{m}}$$

(Tsai & Mecholsky, 1992)

$$\text{Therefore, } \sigma_f = 2.1 \text{ GPa}$$



Ingredients for Polysilicon Microelectro-Mechanical Systems (MEMS) Failures

When σ_{applied} > $\sigma_f = \frac{K_{IC}}{Y\sqrt{c}}$
or residual

Poor Design - Excessive stress

- Excessive applied or **local stress concentrations**
- Materials combinations and/or processing that lead to excessive residual stress

Presence or introduction of defects

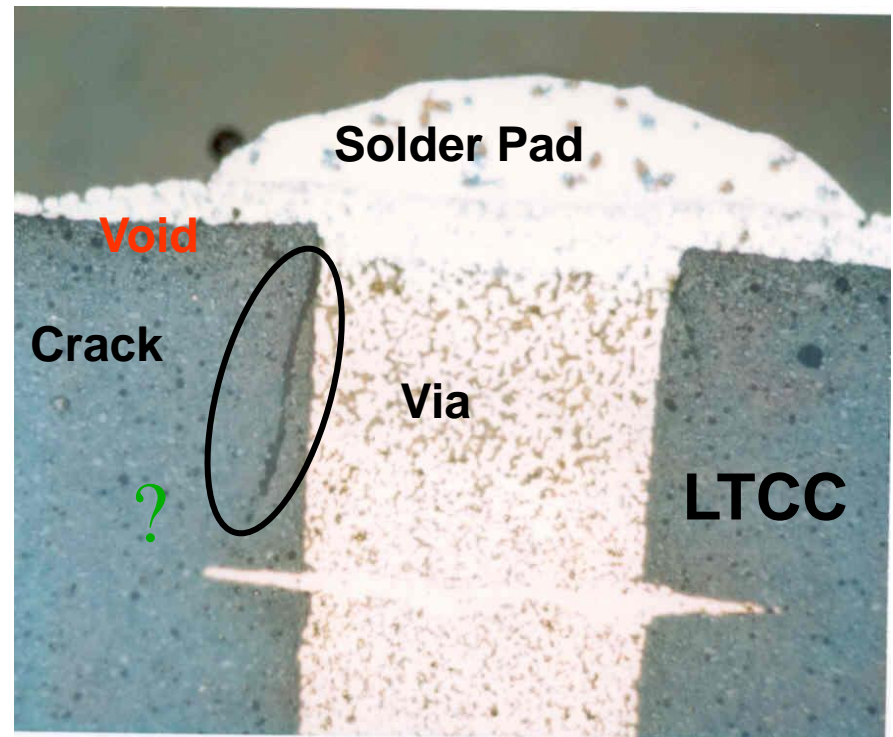
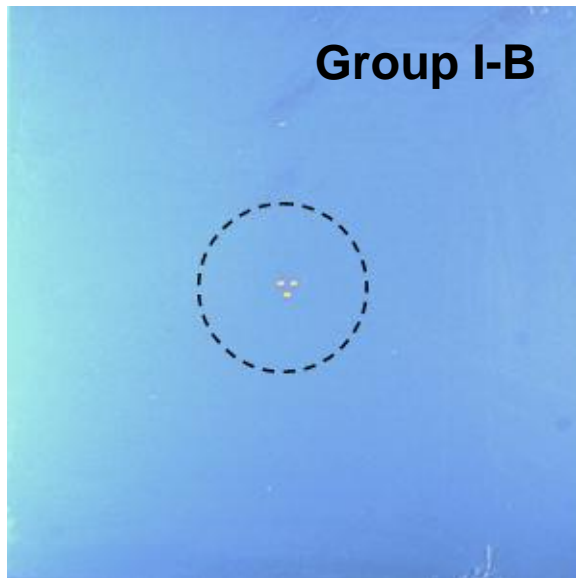
- Intrinsic microstructural features
- ***Processing related***
- Handling or in-service damage

Material degradation (loss of strength)

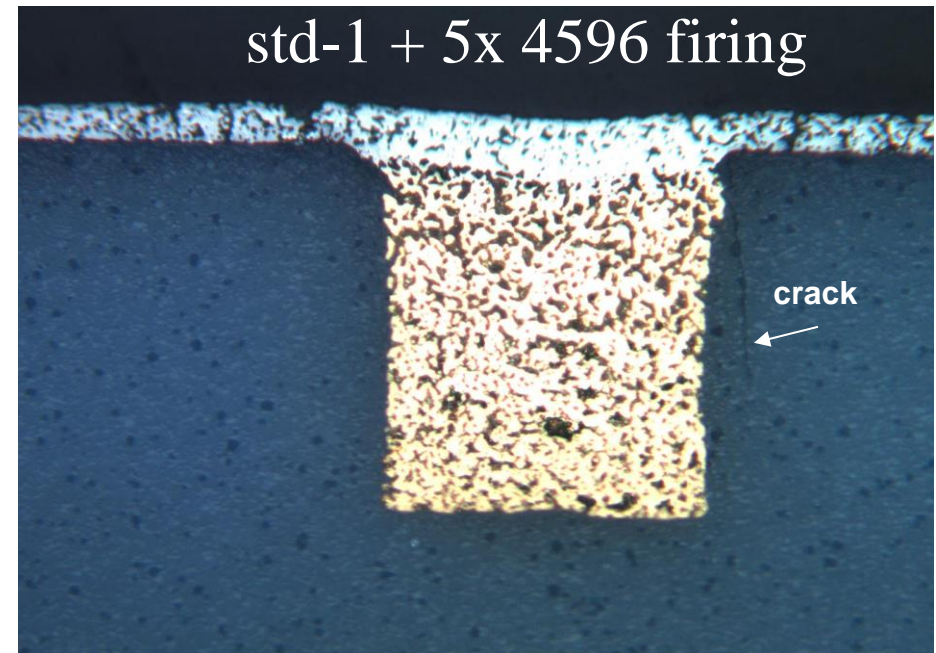
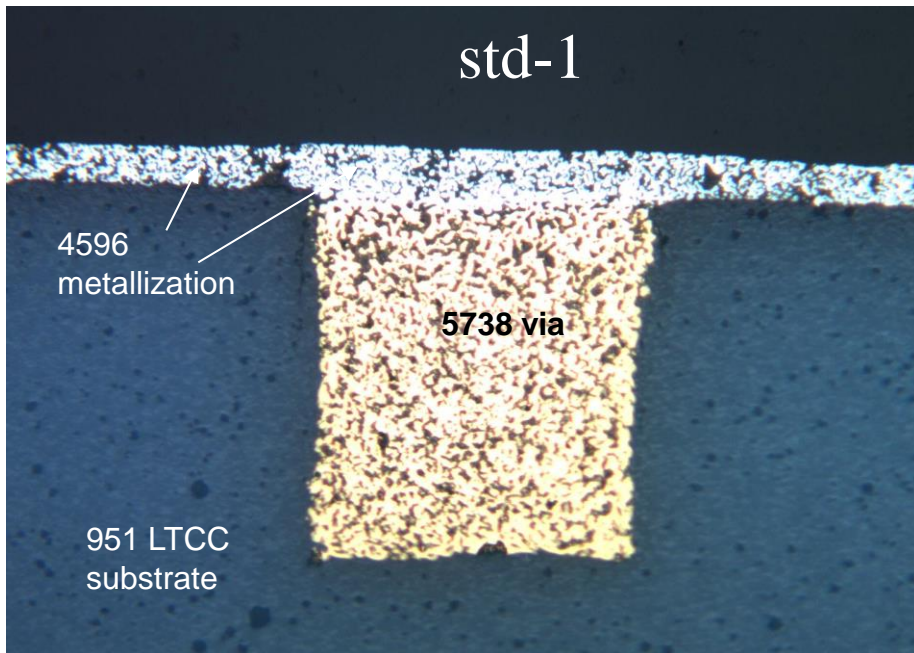
- Environmental effects, decrease in K_{IC}
- Increase in flaw size - subcritical crack growth

Why Does Cracking Occur in the LTCC Near Vias after Metallization and Firing Cycles?

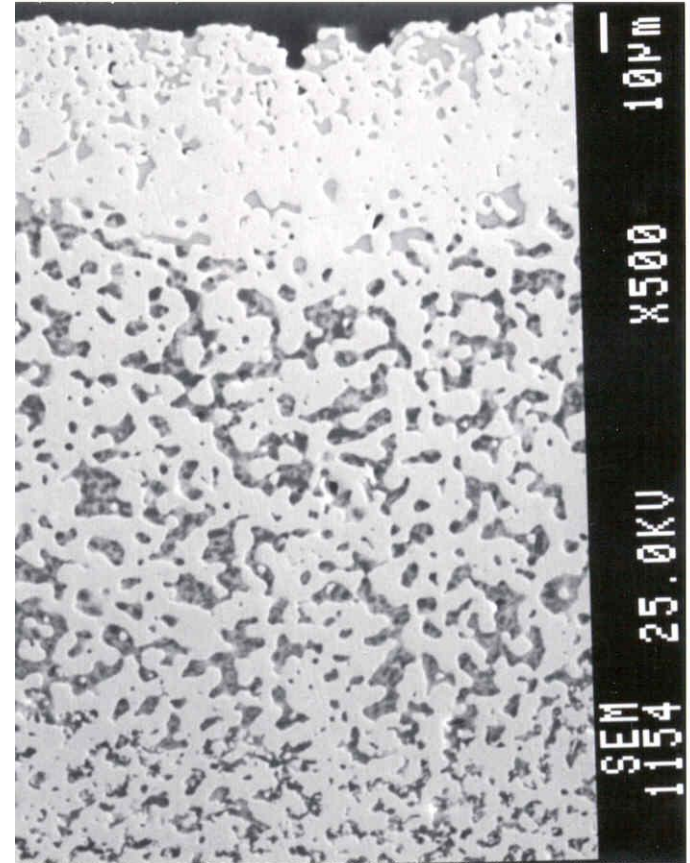
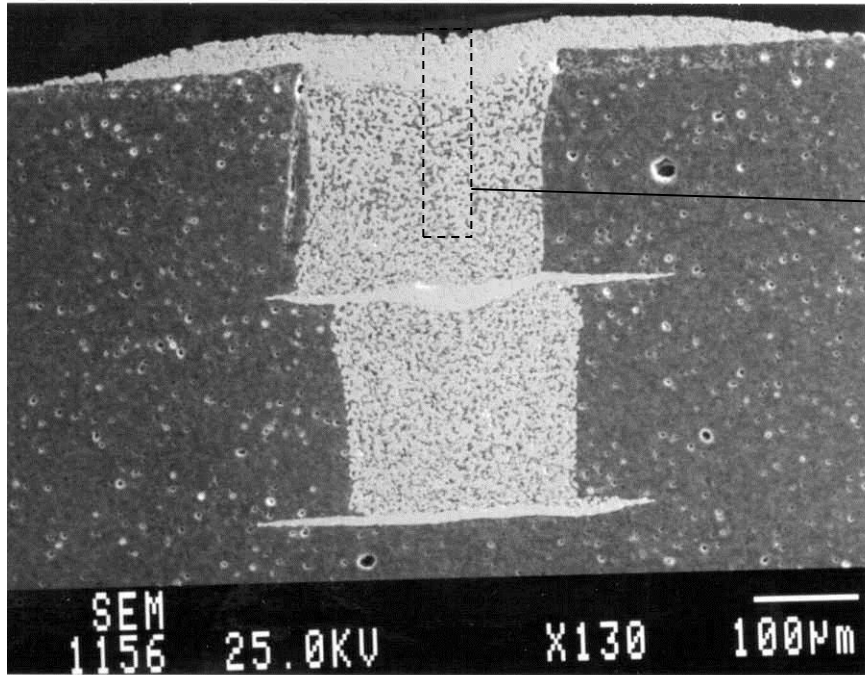
Low temperature co-fired ceramic (LTCC) parts were made with multiple layers of DuPont 951 green tape™ with stacked electrical vias



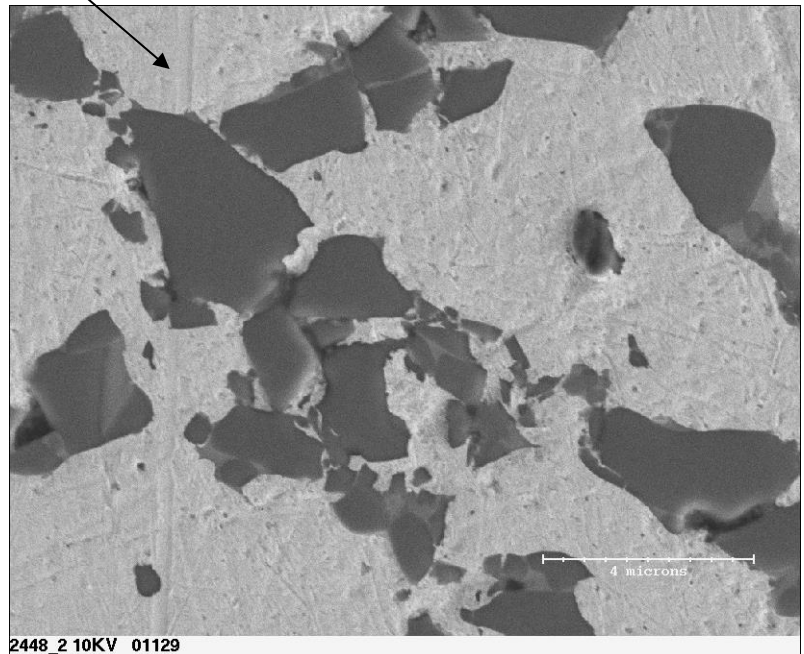
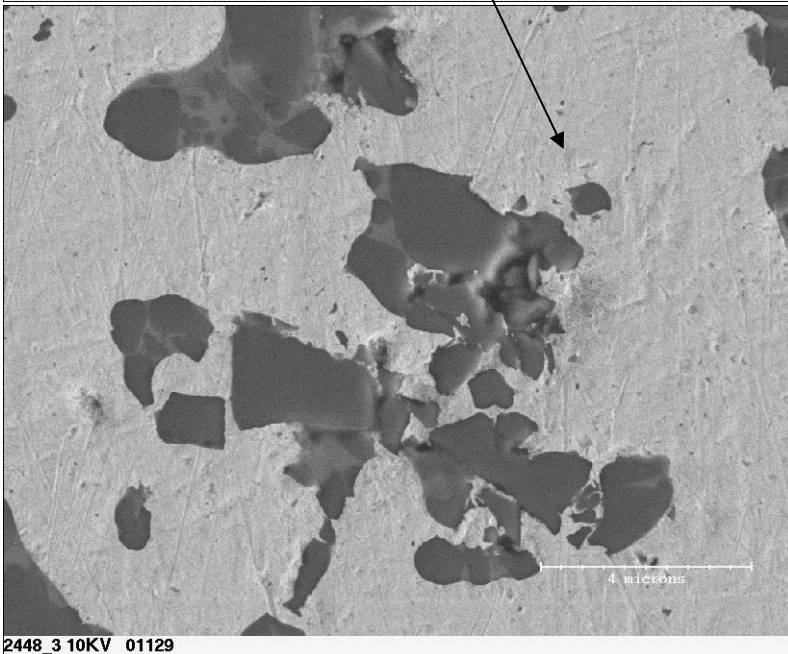
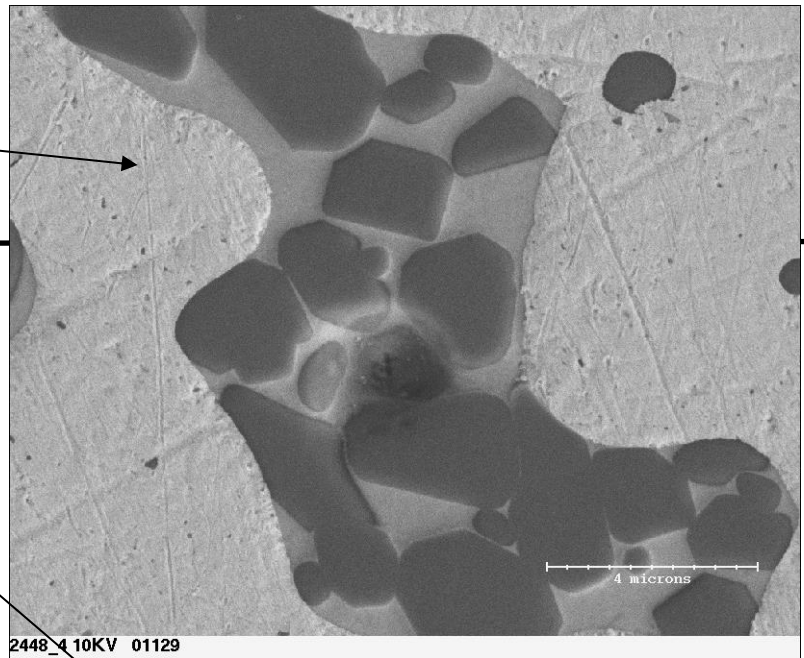
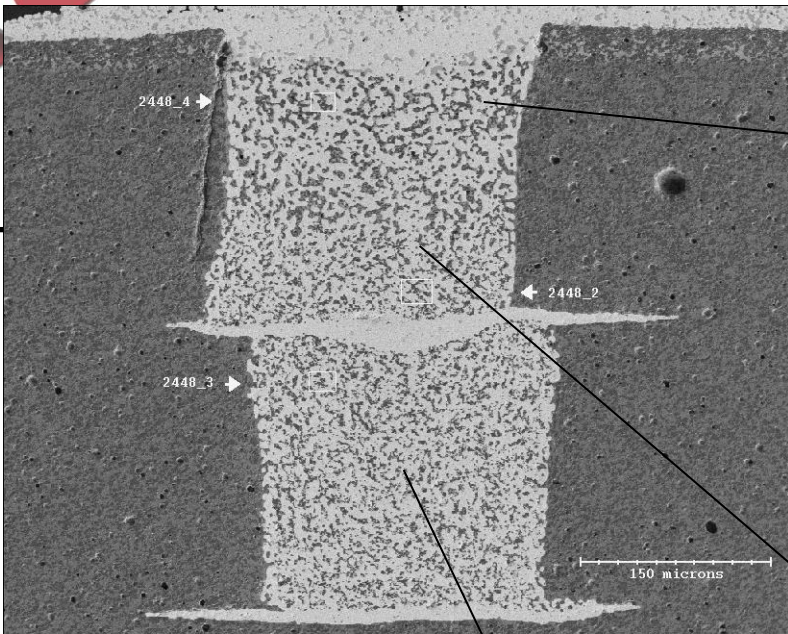
Longitudinal Sections Show LTCC, 5837 Via, and 4596 Metallization



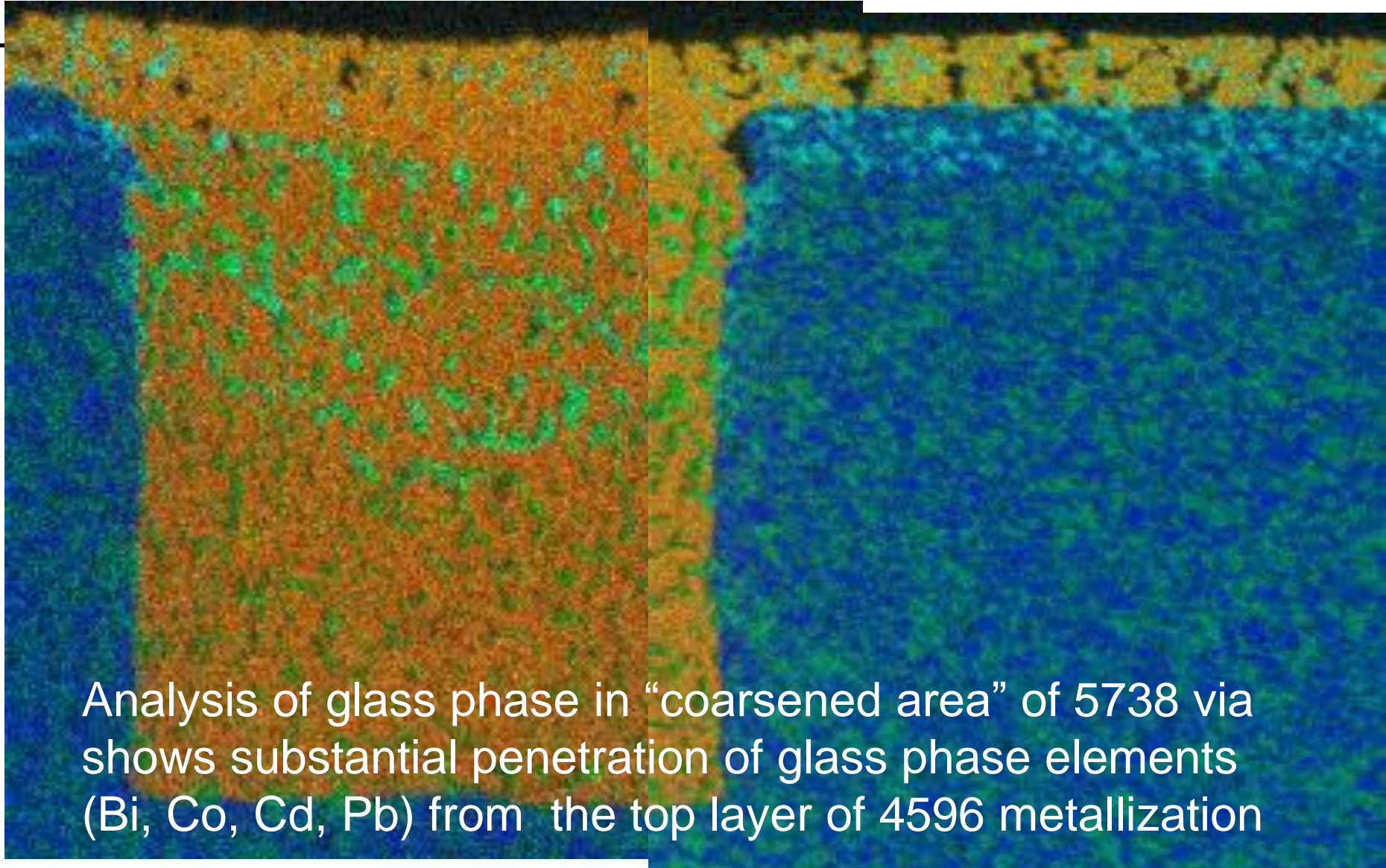
Electron Microprobe Shows Interactions Between Surface Metallization and Via Material



Microprobe analysis provided by Paul Hlava



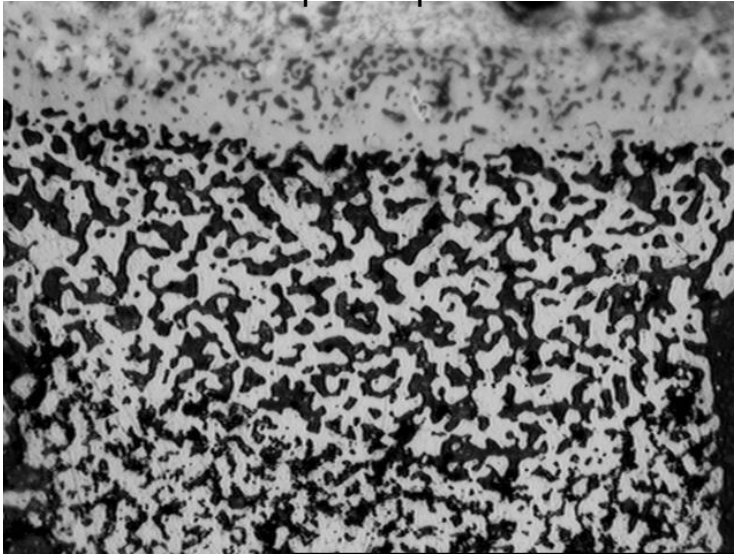
SEM Spectral Image Analysis of Std1_5xFires Parts



Analysis of glass phase in “coarsened area” of 5738 via shows substantial penetration of glass phase elements (Bi, Co, Cd, Pb) from the top layer of 4596 metallization

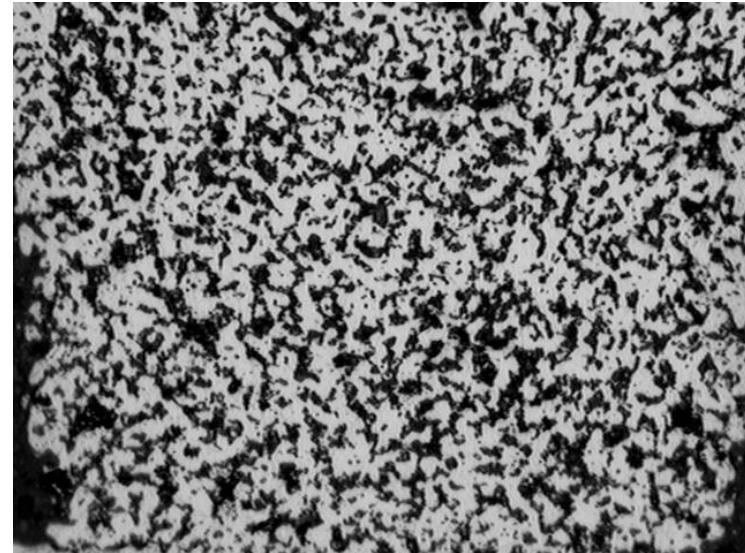
Estimated CTE for Via used in FEA Stress Predictions Based on Original Volume Fractions

Top of top via



area fraction metal= 53%
area fraction other= 47%
MORE Glass than expected

Bottom of top via and internal vias



area fraction metal= 60%
area fraction other= 40%



Ingredients for Failure of a Low Temp Co-Fired Ceramics (LTCCs) with Vias

When σ_{applied} > $\sigma_f = \frac{K_{IC}}{Y\sqrt{c}}$
or residual

Excessive stress

- Excessive applied stress or macroscopic stress concentrations
- ***Materials combination and/or processing that lead to excessive residual stress***

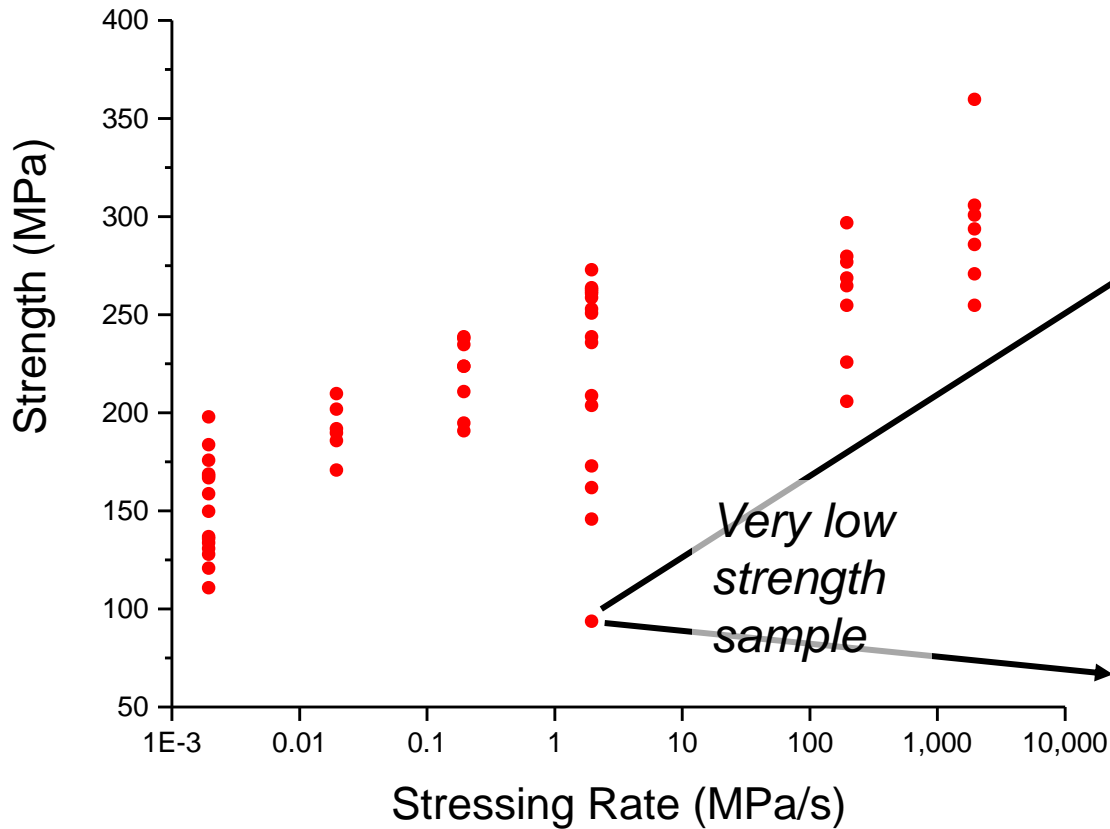
Presence or introduction of defects

- Intrinsic microstructural features
- Processing related
- Machining, in-service damage

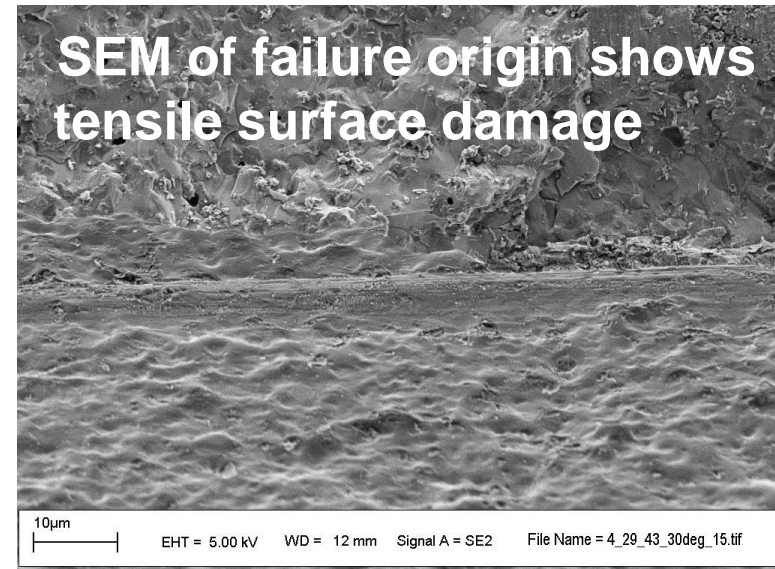
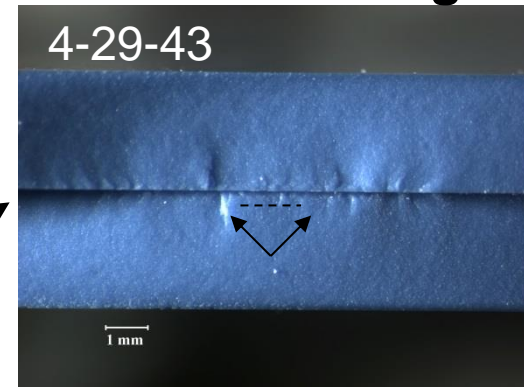
Material degradation (loss of strength)

- Increase in flaw size due to subcritical crack growth
- Decrease in K_{IC}

LTCC Subcritical Crack Growth Data Collected for Lifetime Predictions (crack velocity= AK^n)



Optical exam used to locate failure origin





Ingredients for Failure of Low Temp Co-Fired Ceramic (LTCC) Strength Samples

When σ_{applied} > $\sigma_f = \frac{K_{IC}}{Y\sqrt{c}}$
or residual

Excessive stress

- Excessive applied stress or macroscopic stress concentrations
- Materials combinations and/or processing that lead to excessive residual stress

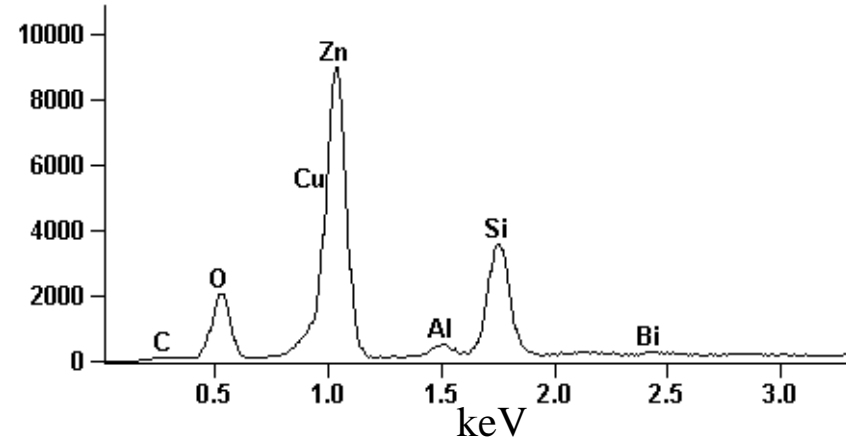
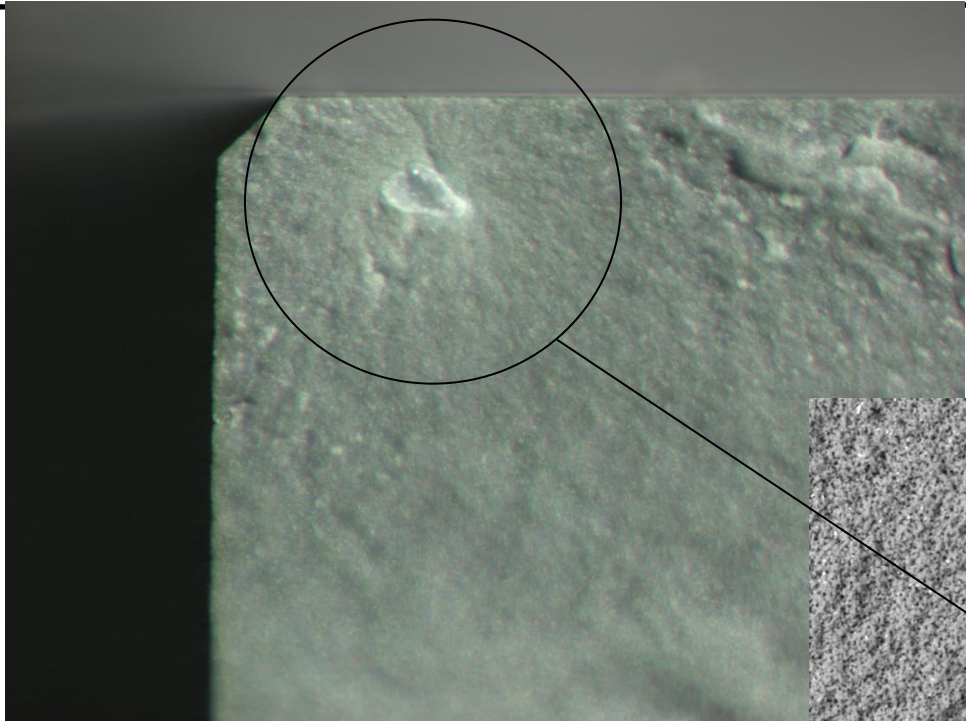
Presence or introduction of defects

- Intrinsic microstructural features
- Processing related
- ***Machining, in-service damage***

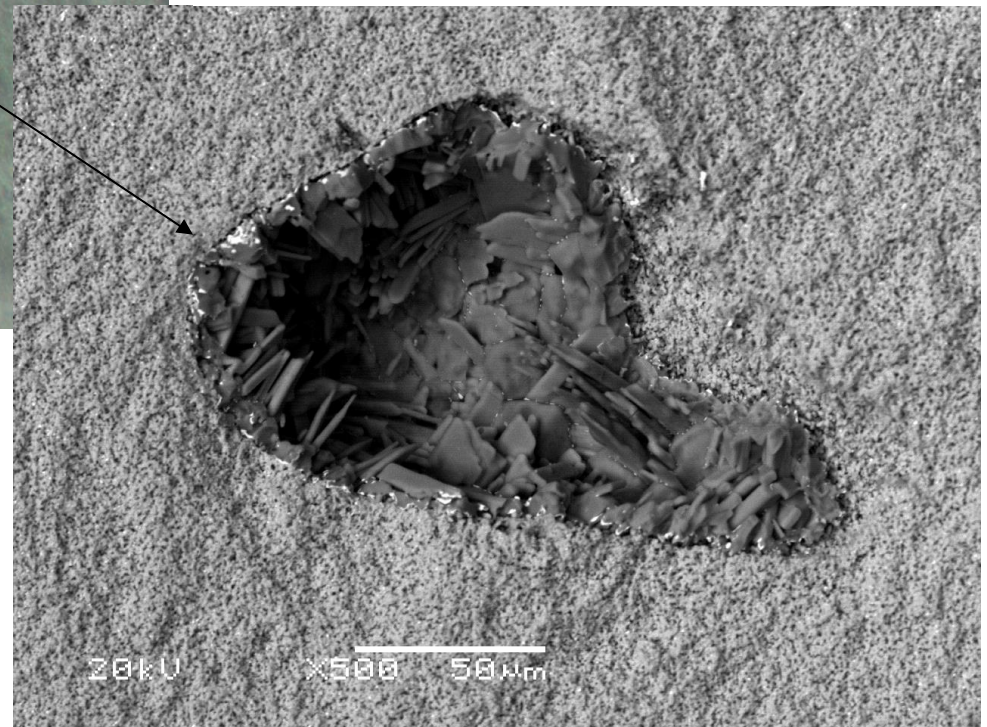
Material degradation (loss of strength)

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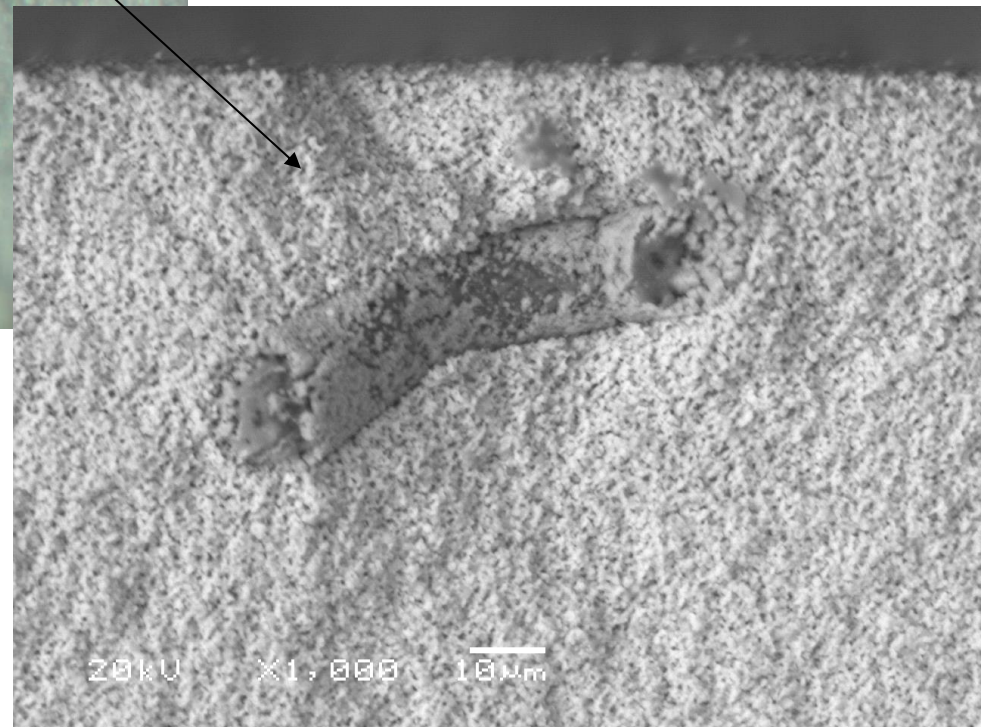
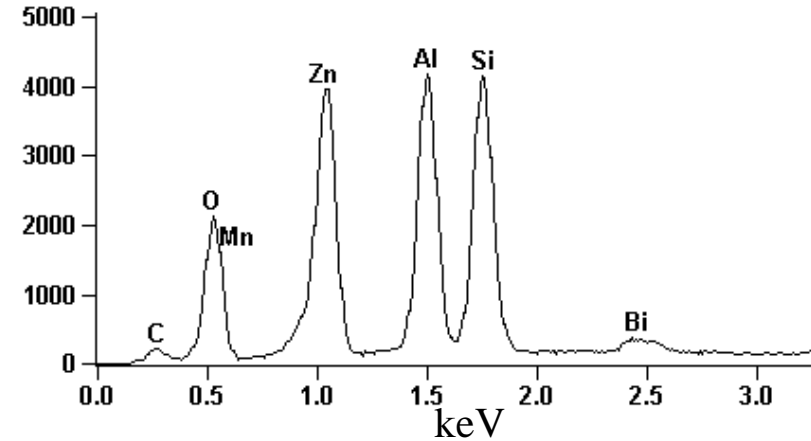
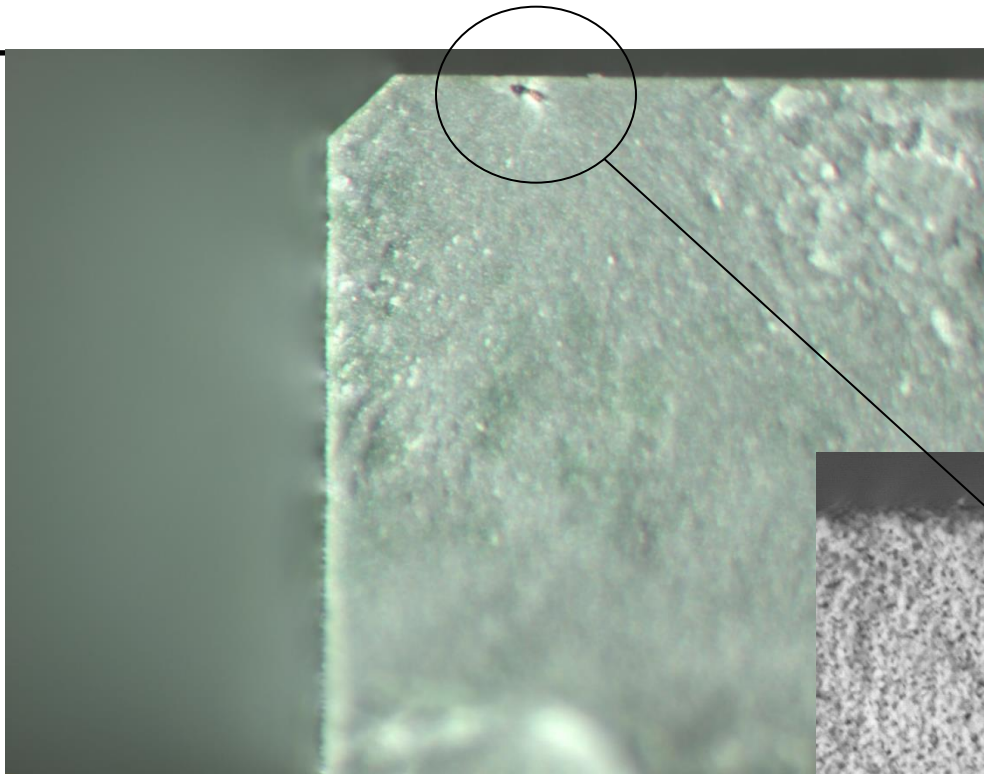
Fractography of ZnO Varistor Bend Bars Reveals that Contaminants Serve as Failure Origins



Si-rich inclusion



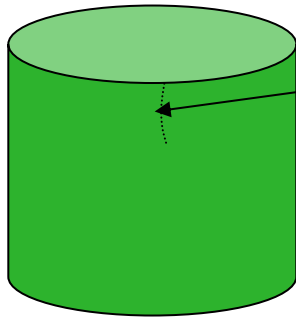
Fractography of ZnO Varistor Bend Bars Reveals that Contaminants Serve as Failure Origins



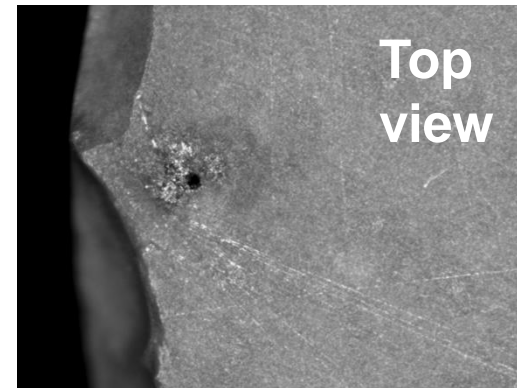
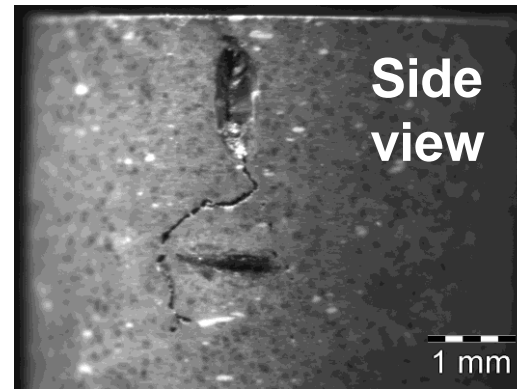
Mn, Al, Si rich inclusion

Surface Damage Studied to Determine How it Contributes to ZnO Electrical Breakdown

Prior to electrical
pulse testing



Crack



- Cracks parallel to current flow produce early breakdowns

Ingredients for ZnO Varistor Failure (Mechanical and Electrical Breakdown Strength)

When σ_{applied} > $\sigma_f = \frac{K_{IC}}{Y\sqrt{c}}$
or residual

Excessive stress

- Excessive applied stress or macroscopic stress concentrations
- Materials combinations and/or processing that lead to excessive residual stress

Presence or introduction of defects

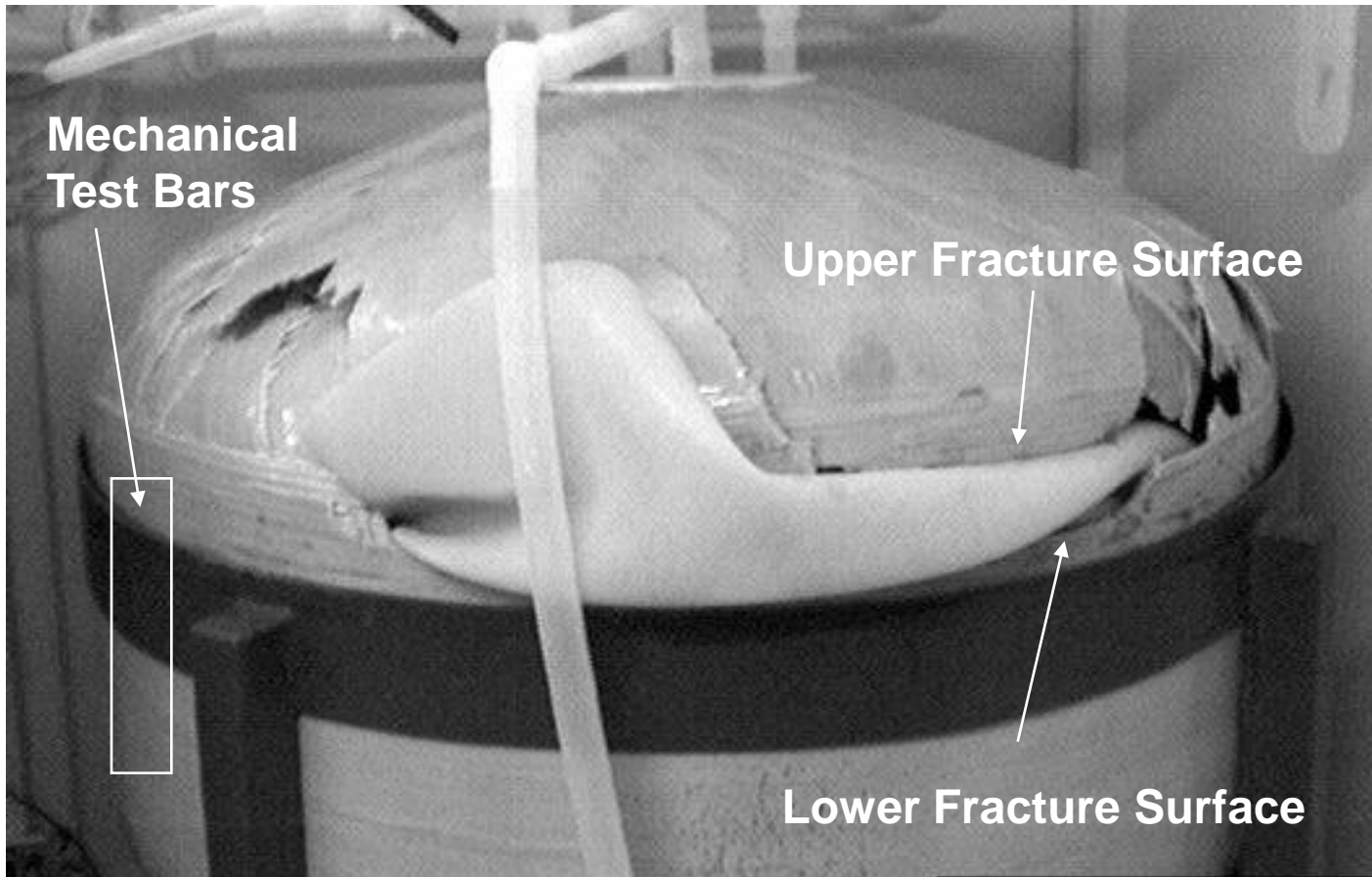
- Intrinsic microstructural features
- **Processing related**
- **Machining, in-service damage**

Material degradation (loss of strength)

- Increase in flaw size due to subcritical crack growth
- Decrease in K_{IC}

Failure and Fractography of a Fiberglass Reinforced Composite (FRC) Acid Tank

FRC tank containing sulfuric acid failed catastrophically during a pressurization cycle.





Tank Failure was Due to Several Factors:

- High tensile stress in region of circular wrap just above the constraining cradle
- Cracking of epoxy in circ wraps exposed cross ply fibers
- Sulfuric acid was introduced into the cracks
- Fibers in the cross plies were severely degraded and weakened by acid
- Sequential failure of fibers in cross plies occurred during pressurization cycles until crack of critical dimensions produced
- Catastrophic crack propagation occurred



Ingredients for Failure of a Fiberglass Reinforced Composite (FRC) Tank

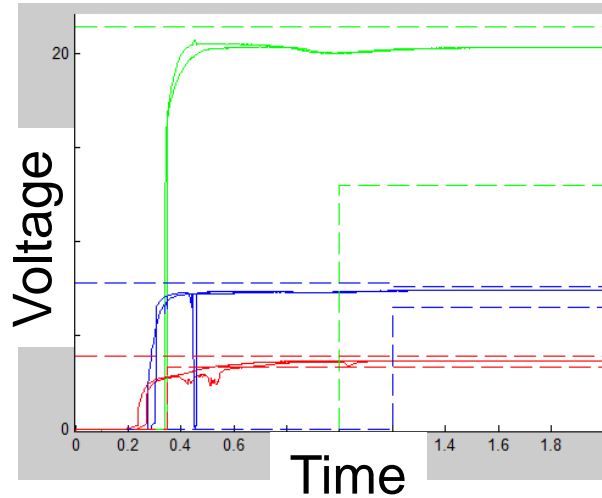
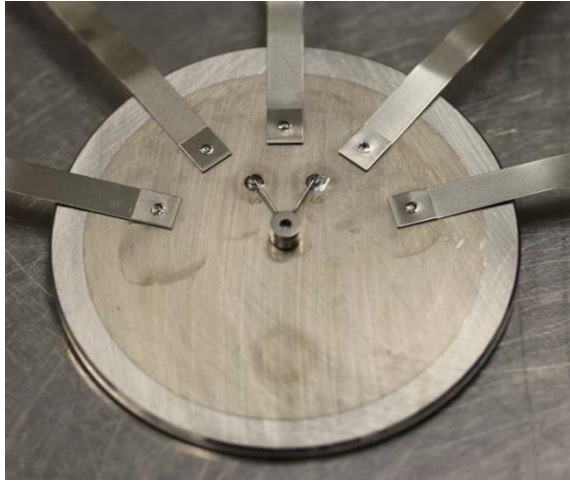
When σ_{applied} >
or residual

$$\sigma_f = \frac{K_{IC}}{Y \cdot \sqrt{c}}$$

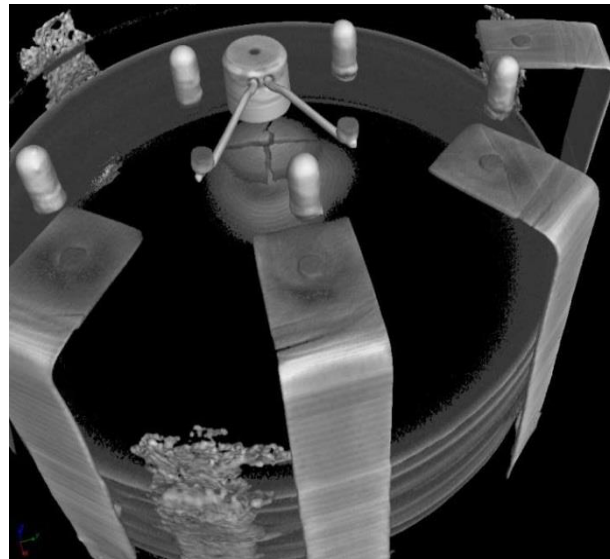
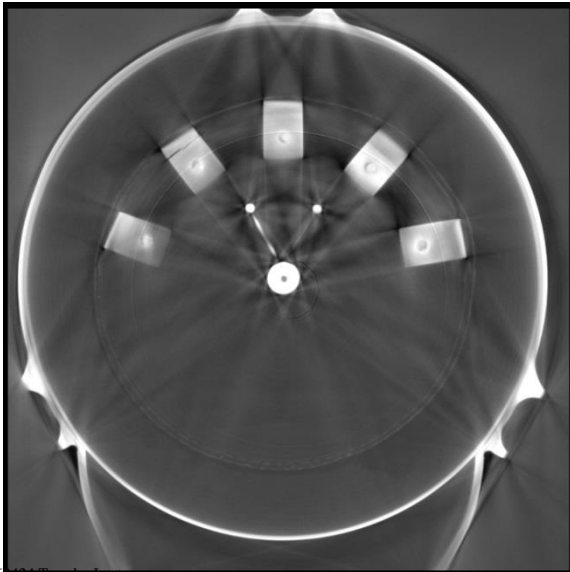
- Excessive stress
 - ***Excessive applied stress or macroscopic stress concentrations***
 - Materials combinations and/or processing that lead to excessive residual stress
- Presence or introduction of defects
 - Intrinsic microstructural features
 - Processing related
 - ***Machining, in-service damage***
- Material degradation (loss of strength)
 - ***Increase in flaw size due to subcritical crack growth***
 - ***Decrease in K_{IC}***

All of the above!

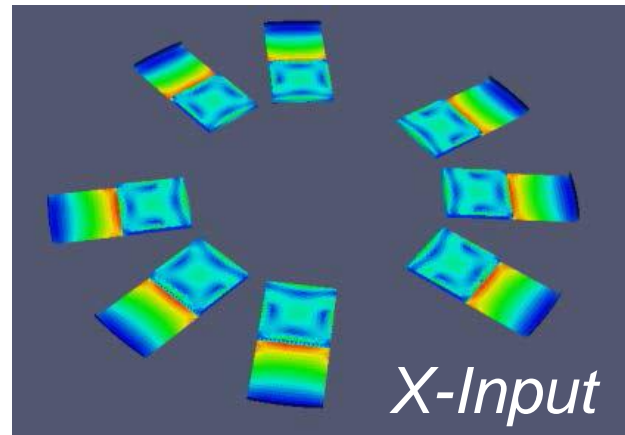
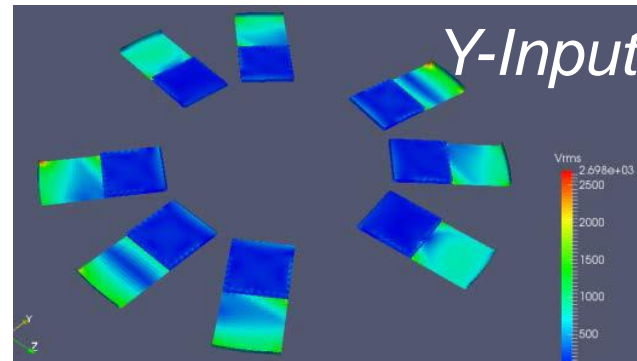
Ni Lead Failures in Thermal Batteries During Testing in Various Vibration Environments



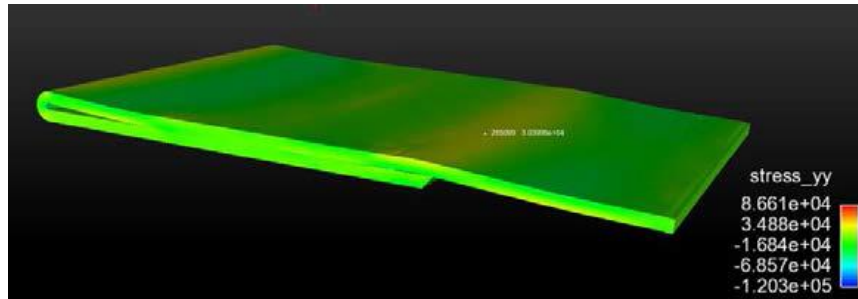
Electrical testing of a new thermal battery design in a vibration environment showed unacceptable drops in voltage vs. time.



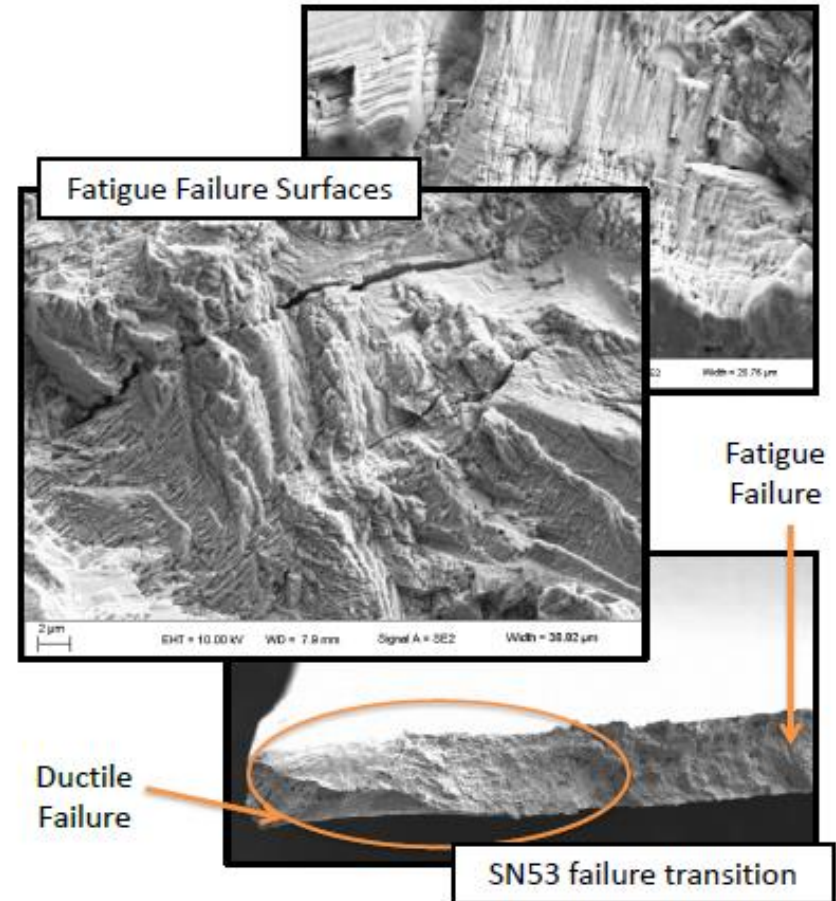
Stress Analyses of Ni Leads For Vibration Tests Showed Stresses were Highly Axis Dependent



Fracture Surface Analysis Confirmed High Cycle Fatigue Failure

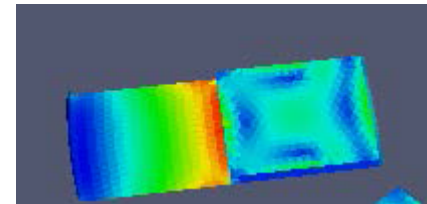


- All three failed Ni leads show surface that appears brittle, with tightly spaced striations throughout
- Based on these features, failures are consistent with low amplitude, high cycle fatigue (transportation vibration environment)
- Remainder of #53 was 'failed' in lab environment – portion exhibited ductile failure as expected for Ni 201



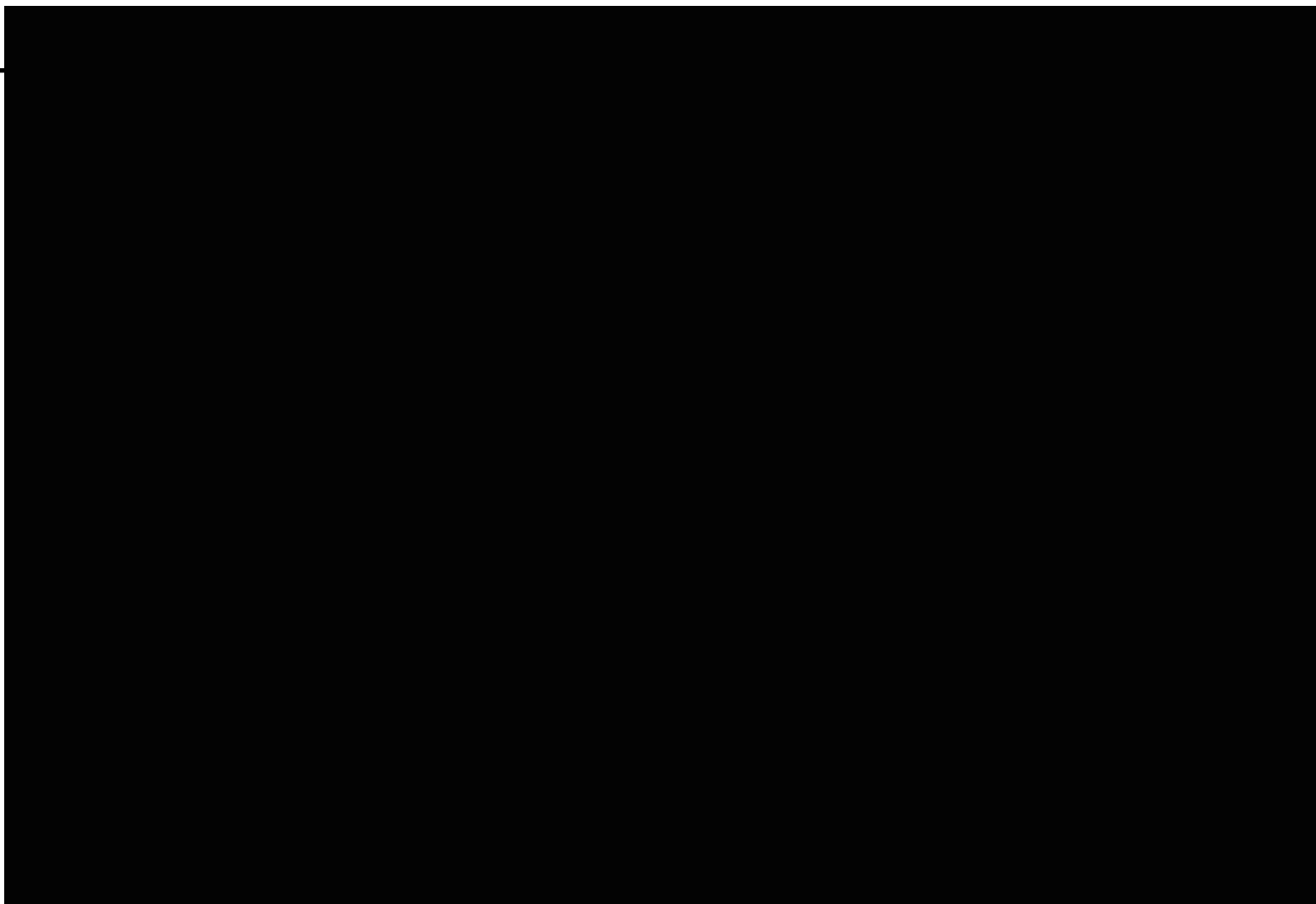
Ingredients for the Failure of a Battery Lead

- Design - Excessive stress
 - **Excessive applied stress/macroscopic stress concentrations**
 - Materials combinations and/or processing that lead to excessive residual stress
- Presence or introduction of defects
 - Intrinsic microstructural features
 - **Processing related defects**
 - Machining, in-service damage
- Material degradation (loss of strength)
 - **Increase in flaw size due to fatigue crack growth**
 - Decrease in K_{IC}





Glass Failure





Making the Most of Our Failures

- Failure/survival depends on properties, flaws, design - loading (applied and residual), and environment
- Failure often due to a combination of factors
- We have valuable opportunities to improve our materials, processing, designs, and analyses by “*observing, measuring, and interpreting our failures*” (D. Hull)

Materials Failure Experiences as a Metaphor for Human Failure

FAMOUS FAILURES

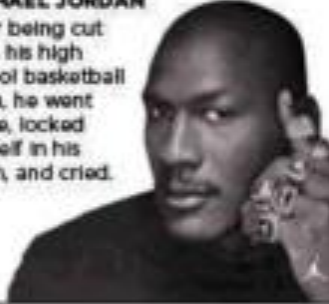
ALBERT EINSTEIN

He wasn't able to speak until he was almost 4-years-old and his teachers said he would "never amount to much"



MICHAEL JORDAN

After being cut from his high school basketball team, he went home, locked himself in his room, and cried.



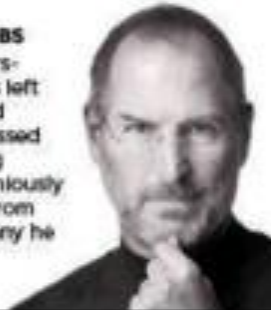
WALT DISNEY

Fired from a newspaper for "lacking imagination" and "having no original ideas."



STEVE JOBS

At 30-years-old he was left devastated and depressed after being unceremoniously removed from the company he started.



OPRAH WINFREY

Was demoted from her job as a news anchor because she "wasn't fit for television."



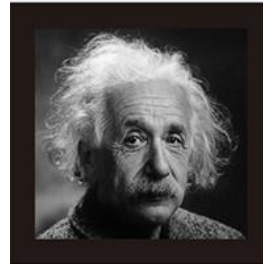
THE BEATLES

Rejected by Decca Recording Studios, who said "We don't like their sound—they have no future in show business."



104 TIMES

Albert Einstein didn't speak until he was four and didn't read till seven. He went on to win a Nobel Prize and became the face of modern physics.



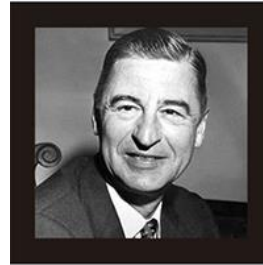
12 TIMES

J.K. Rowling was famously rejected by a mighty 12 publishers before Harry Potter and The Philosopher's Stone was accepted.



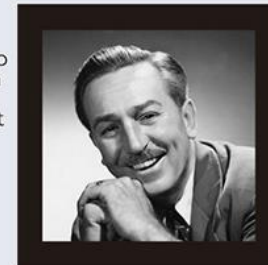
27 TIMES

Dr. Seuss's first book To Think That I Saw It on Mulberry Street was rejected by 27 different publishers.



302 TIMES

Legend has it Walt Disney was turned down 302 times before he got financing for creating Disneyland.





Acknowledgments

- Penn State – Dr. David Green, Dr. Gary Messing, Dr. Richard Tressler, Dr. John Hellman, Dr. Jack Mecholsky (U. of Florida), and many others
- Sandia Colleagues - Ed Beauchamp, Brad Boyce, Tom Buchheit, Bonnie McKenzie, Sandy Monroe, Clay Newton, Christine Roth, Rajan Tandon, Chad Watson, Eric Tumilowicz, Richard Heller, Henry Padilla, Louie Cano, David Enos, Don Susan, Joe Romero, Environmental Testing Lab colleagues, and many others

Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Security Administration under Contract DE-ACO4-94AL85000.



Thank you!



Failure Quotes

“Materials are a lot like people, imperfections control their behavior” Rusty Gray

“All major weaknesses have been exposed, analyzed, and replaced with new weaknesses.” Bruce Leverett

**Failure is not an option. It comes bundled with your software.
Unattributed**



Back-Up Slides



“Safety Movie”



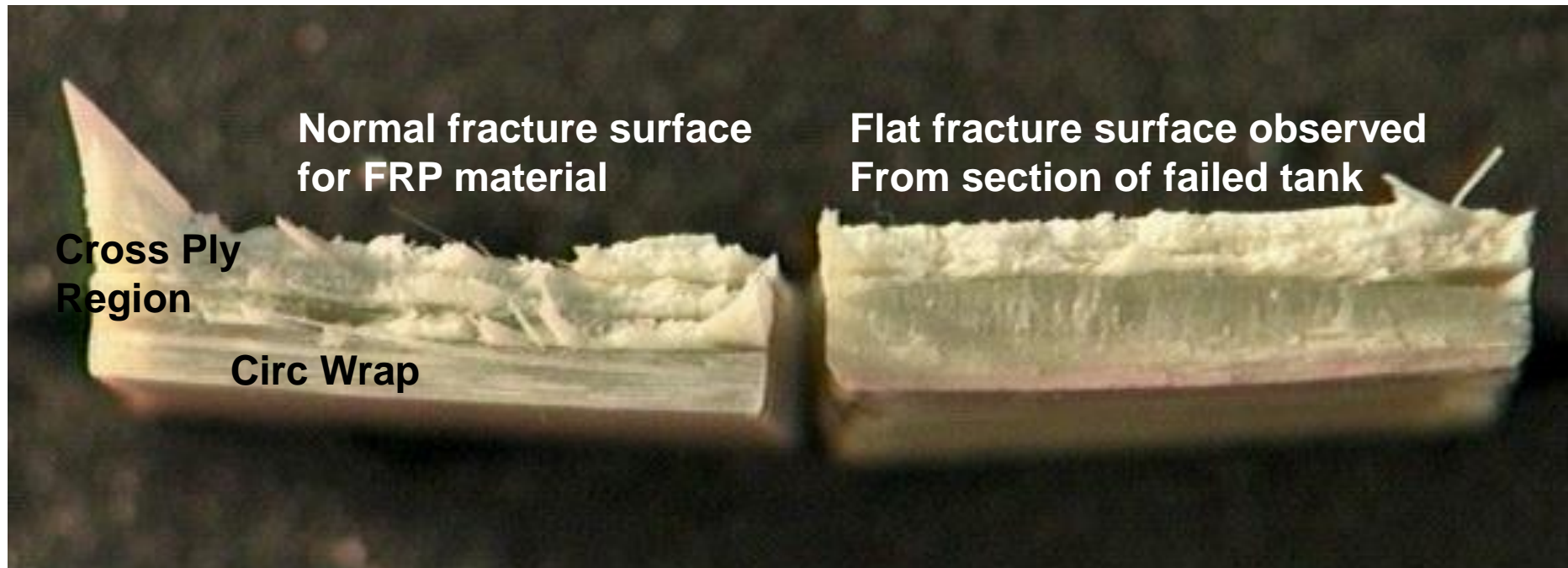


Environments/Requirements under which Sandia Components Must Remain Reliable

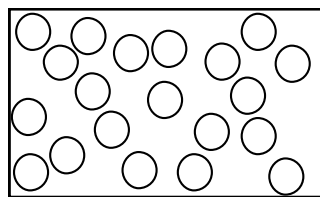
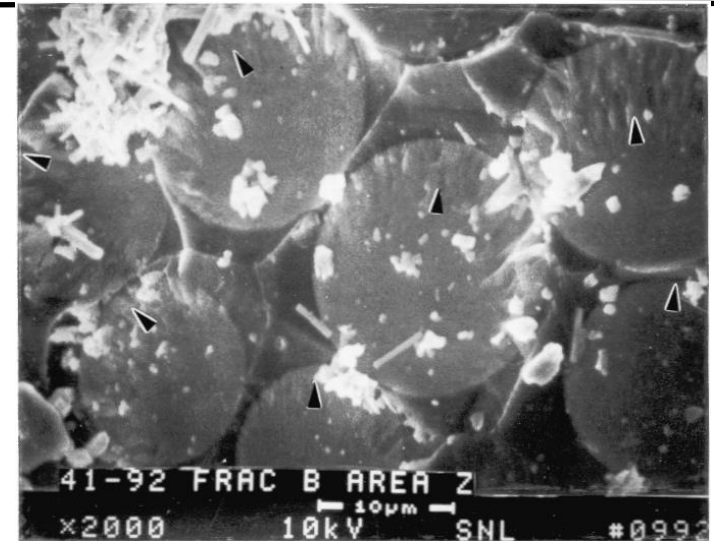
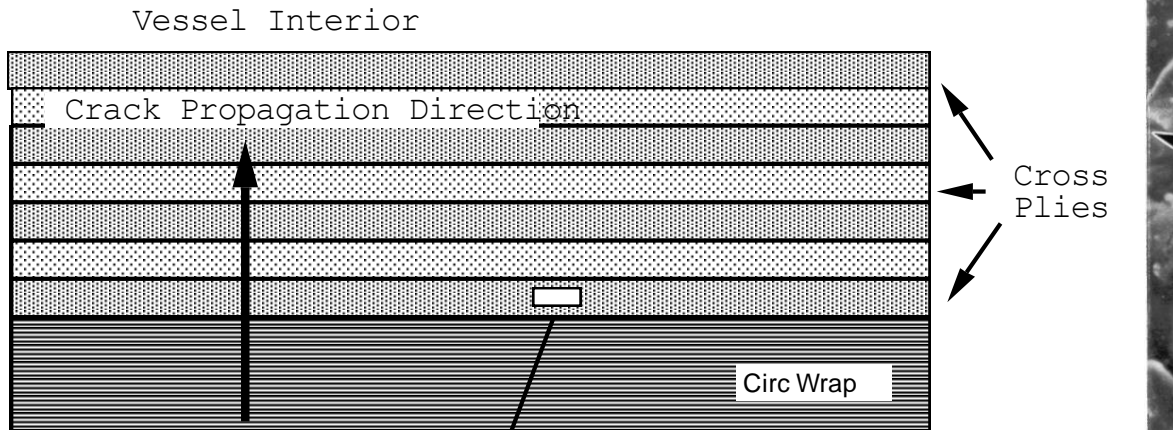
- Temperature Cycling from -55 to 85C
- Humidity for Storage - < 25% RH
- Shock and Vibration
- Lifetimes - 20-30 years

Optical Examination Revealed Unusually Flat Fracture Surfaces

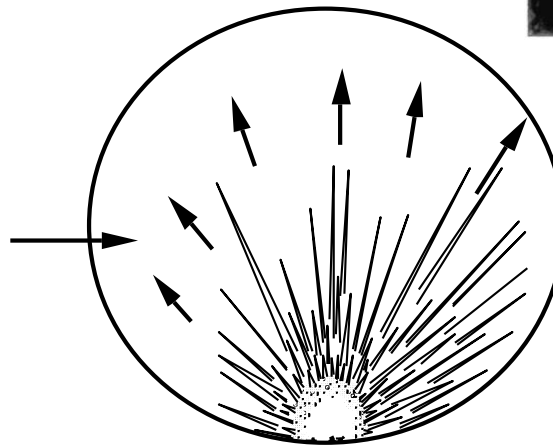
- Few fibers protruded from fracture surfaces of failed tank



SEM Exam of Fracture Features on Fibers



Blow-up of cross ply region.



Blow-up of individual glass fiber. Hackle marks indicate crack propagation direction



Glass Fiber Strength Estimates using Mirror Radii Showed Lower than Expected Strengths

- Estimated failure strengths 20-40% of expected strength for E-glass fibers

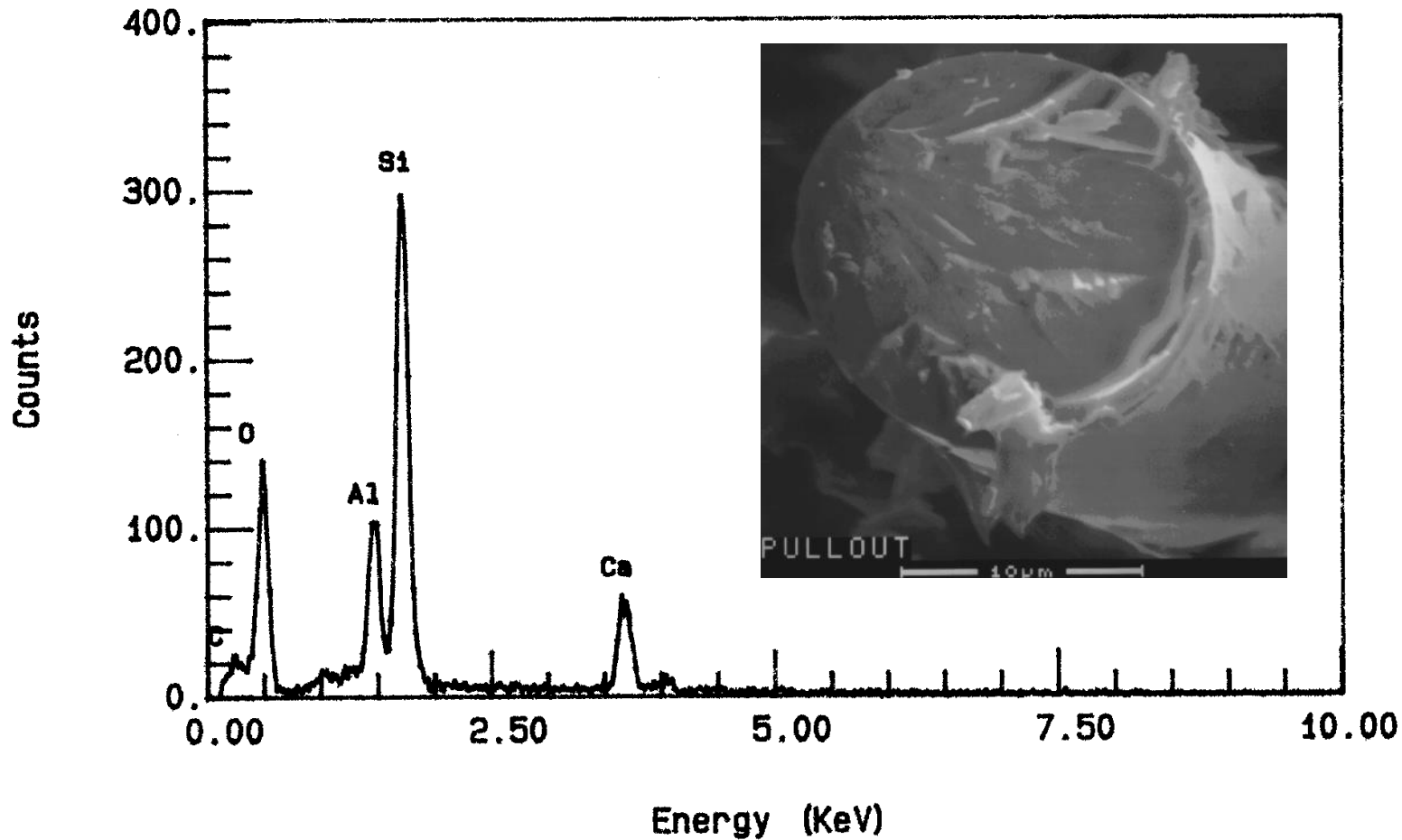
$$\sigma_f \sqrt{r_{\text{mirror}}} = A_{\text{mirror}}$$

$$A_m \sim 1.5 - 2$$

(620-1207 MPa vs. 3100 MPa or 90-175 ksi vs. 450 ksi)

- Why was glass weaker than expected?
 - Wrong glass
 - Mechanical damage
 - Environmental degradation (acid was present)

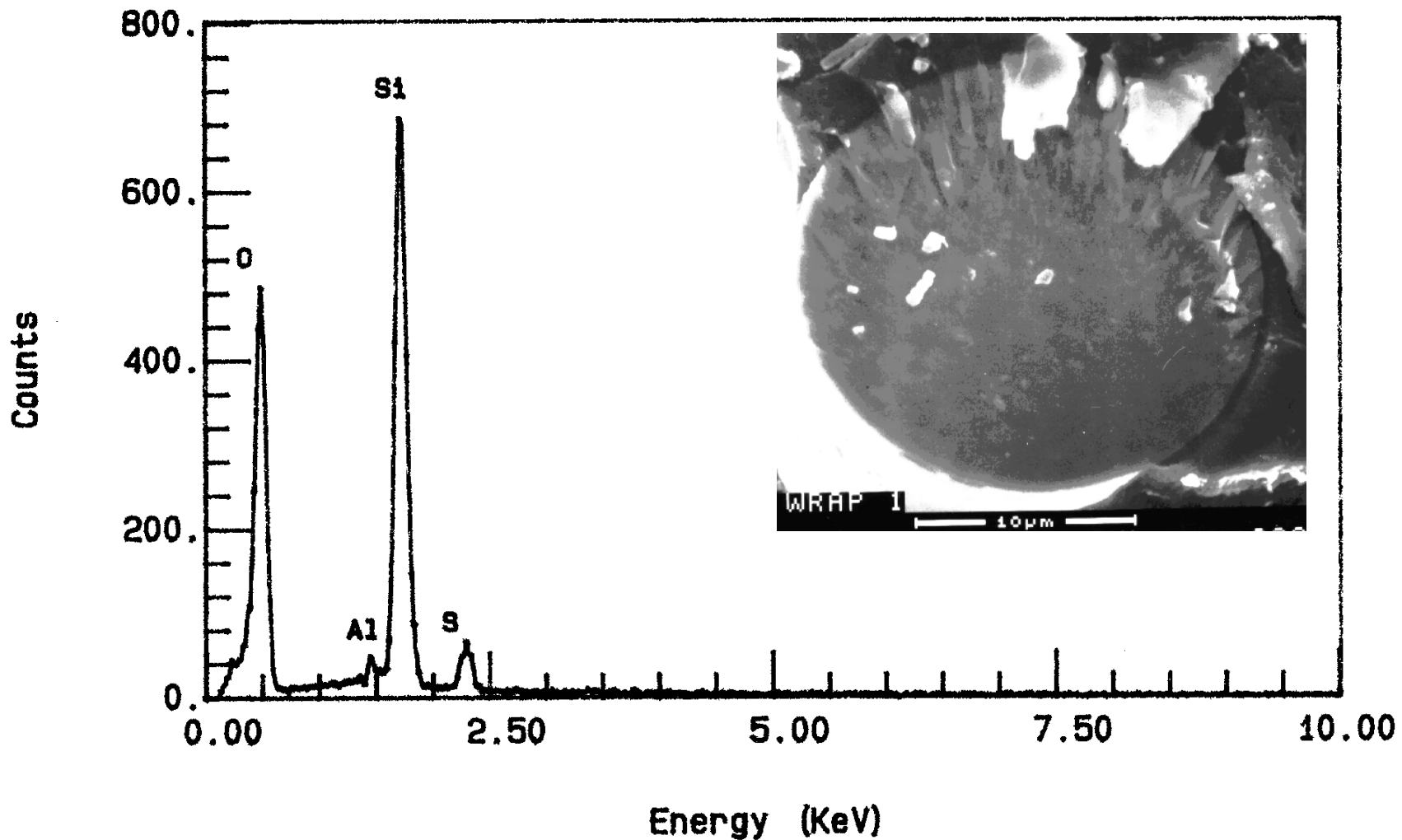
Chemical Analyses Performed on Strong Glass Fibers Showed Expected Elements



E-glass composition by wt%:

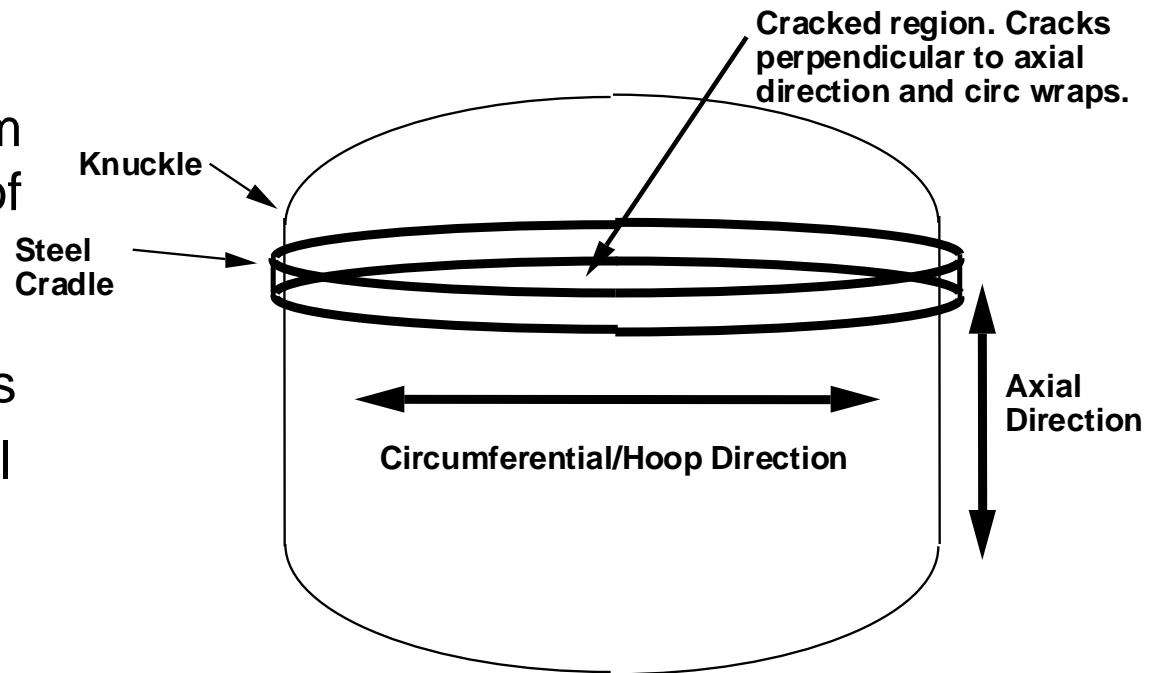
16% CaO, 14.5 Al₂O₃, 9.5% B₂O₃, 5% MgO, ~1% Na₂O and K₂O, and 55% SiO₂

Weak Glass Fibers Showed Al and Ca Depletion and the Addition of a S Peak



Finite Element Analysis Showed Stresses Approached Material Strength in Failed Regions

- FEA predicted axial stresses of ~ 49 MPa in the circ wraps just above the cradle due to constraint
- Flexure tests of material from this region in the good part of the failed tank indicated stresses of 55 ± 9 MPa to crack epoxy in the circ wraps
- Tank inspection using vicinal illumination revealed cracking only in the region predicted by the model



Dr. Richard E. Tressler



PennState
College of Earth
and Mineral Sciences

Department of Materials
Science and Engineering



Dr. Richard E. Tressler was an international leader in the development of high temperature materials. He pioneered high temperature fiber testing and use, advanced instrumentation and test methodologies for thermostructural materials, and design and performance verification of ceramics and composites in high temperature aerospace, industrial and energy applications. He was founding director of the Center for Advanced Materials (CAM) which supported many faculty and students from the College of Earth and Mineral Science, the Eberly College of Science, the College of Engineering, the Materials Research Laboratory and the Applied Research Laboratories at Penn State on high temperature materials. His vision for interdisciplinary research played a key role in the creation of the Materials Research Institute, and the establishment of Penn State as an acknowledged leader among major universities in materials education and research.

He earned his B.S. degree in Ceramic Technology from Penn State in 1963, his M.S. at MIT in 1964, and his Ph.D. in Ceramic Science at Penn State in 1967. He was a decorated veteran, serving as a Captain in the United States Air Force, then joined the Ceramics faculty at Penn State in 1972. He served as Head of the Department of Materials Science and Engineering from 1991 to 2001. During Professor Tressler's 10 year tenure he sought to enhance Penn State's reputation for excellence by hiring over 15 new faculty members.

Perhaps his most lasting accomplishment was the reorganization of the department from an option-focused organization into a global materials organization currently being emulated by the leading materials departments in the U.S. He also served on the technical advisory boards of Kyocera, Air Products, and Alcoa, as well as the Board of Directors of AVX Corporation. In recognition of his accomplishments he was named Academician of the World Academy of Ceramics, Honorary Member of the Societe Francaise de Metallurgie et de Materiaux, the International Prize of the Japan Fine Ceramics Association, and was named Distinguished Life Member of the American Ceramic Society, and received the Hosler Alumni Scholar Award of the College of Earth and Mineral Sciences.

He retired as Professor Emeritus of Materials Science and Engineering in 2001 and in recognition of his contributions received the Hosler Alumni Scholar Award of the College of Earth and Mineral Sciences.



Calvin Coolidge on Persistence

Nothing in the world can take the place of persistence. Talent will not; nothing is more common than unsuccessful men with talent.

Genius will not; unrewarded genius is almost a proverb. Education alone will not; the world is full of educated derelicts.

Persistence and determination alone are omnipotent."



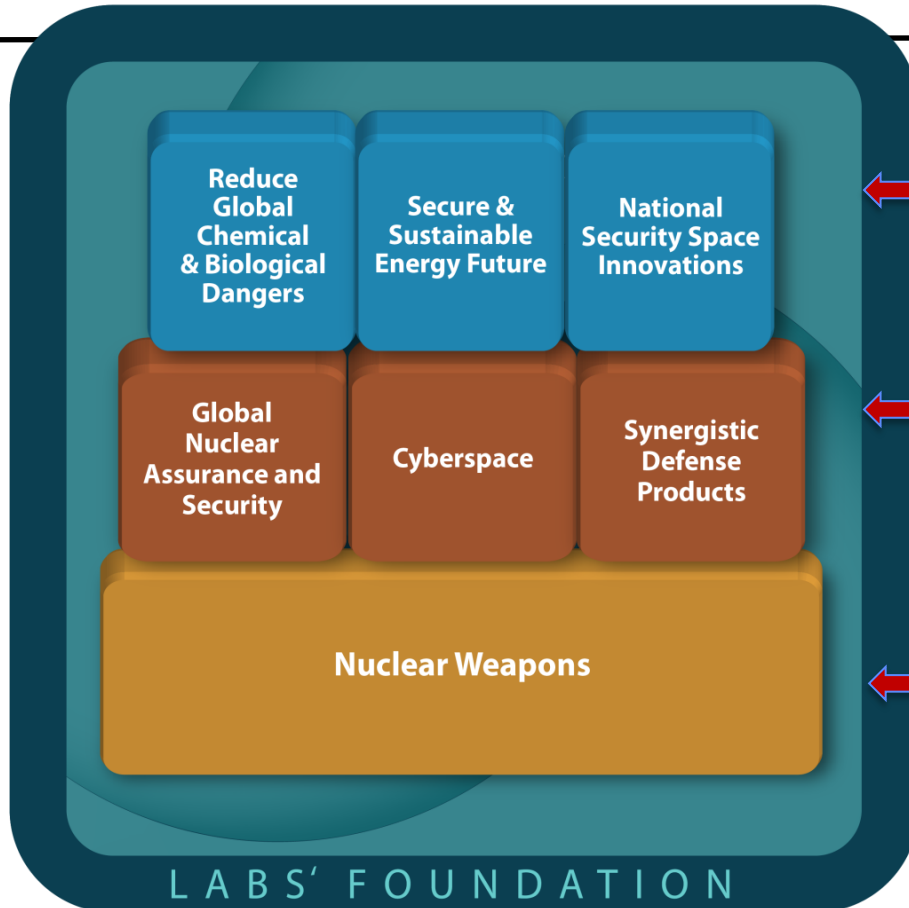
Fractography of Glass, Edited by Richard C. Bradt and Richard E. Tressler, 1994

We are grateful to the authors for their excellent contributions and their **persistence** in completing their manuscripts. We are appreciative of their patience and understanding of the difficulties in bringing this volume to its final form.

Richard C. Bradt, Tuscaloosa, AL

Richard E. Tressler, University Park, PA

Sandia's National Security Mission Areas



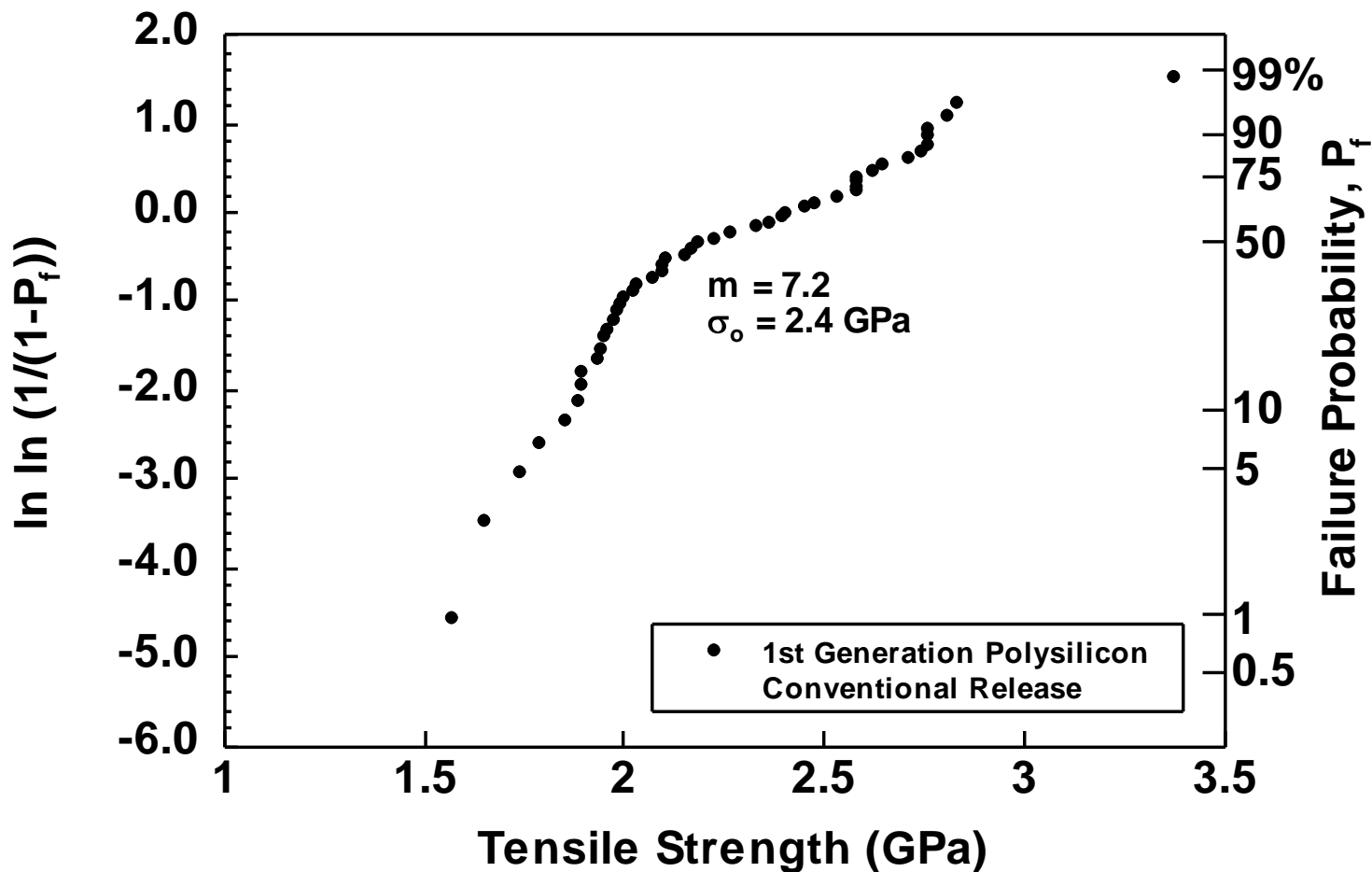
▪ Critical to our national security, these three mission areas leverage, enhance, and advance our capabilities.

▪ Strongly interdependent with NW, these three mission areas are essential to sustaining Sandia's ability to fulfill its NW core mission.

▪ Our core mission, nuclear weapons (NW), is enabled by a strong scientific and engineering foundation.

Underlying Sandia's national security mission space and much of the hardware that we design and build is the need for very high long-term reliability.

Weibull Plot for 1st Generation Polysilicon Pull-Tab Tensile Strength Samples





Flaw Size Estimates Indicate Flaws too Small to Measure Using SEM

$$K_{IC} = \sigma_f Y(\pi c)^{1/2}$$

σ_f = Fracture strength

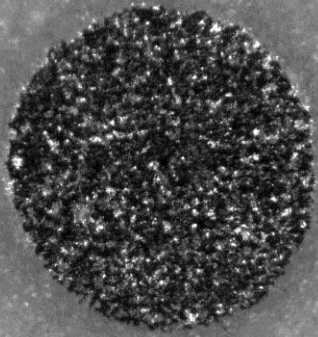
K_{IC} = Fracture toughness ~ 1 MPa \sqrt{m}

Y = Loading factor = 1.12

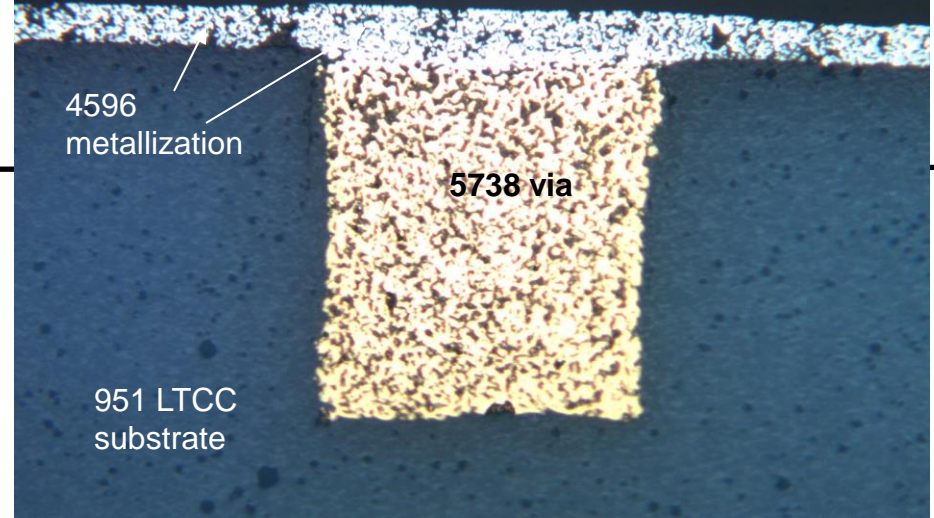
c = Flaw size

Material	Strength(GPa)	Flaw Size Estimate (nm)
1st gen.	2.2	44
2nd gen., CR	4.3	14
2nd gen., TC	3.0	28

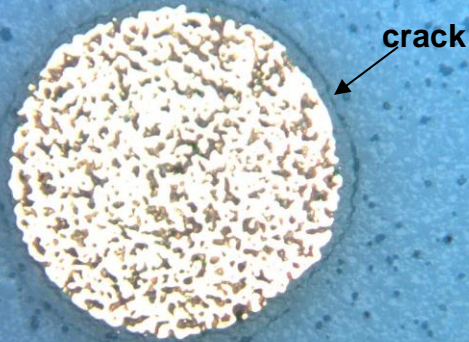
std-0 8 mil diameter via



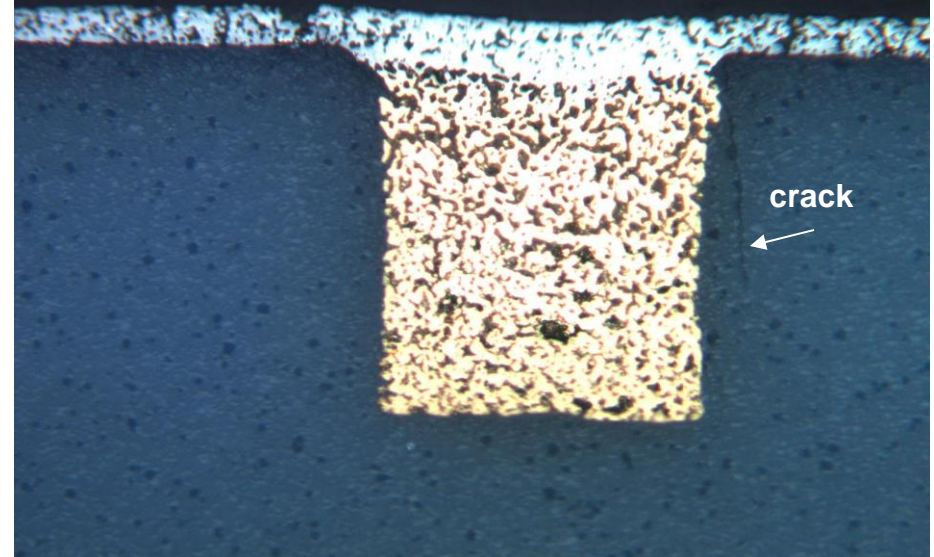
std-1



Std-6, 8 mil diameter via



std-1 + 5x 4596 firing



Planar sections with
4596 metal removed

Longitudinal sections expose LTCC,
5837 via and 4596 metallization