SAND2018-0711PE

Making the Most of Our Failures

NM Tech Grad Seminar

Jan. 25, 2018

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

sjglass@sandia.gov

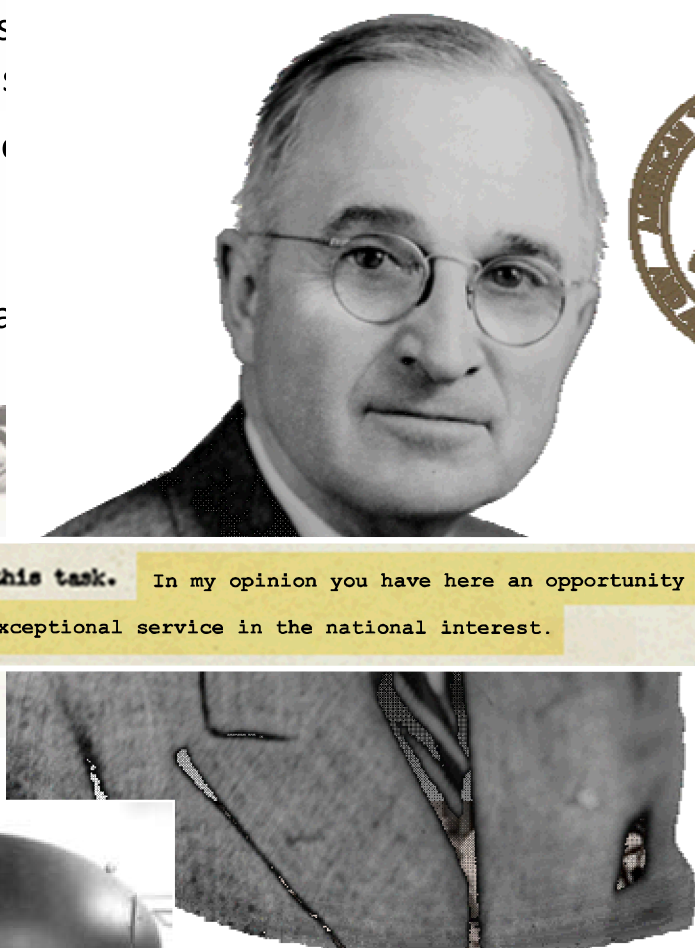
Sandia National Laboratories is a multimission laboratory managed and operated by National Technology and Engineering Solutions of Sandia, LLC., a wholly owned subsidiary of Honeywell International, Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA-0003525.

Outline

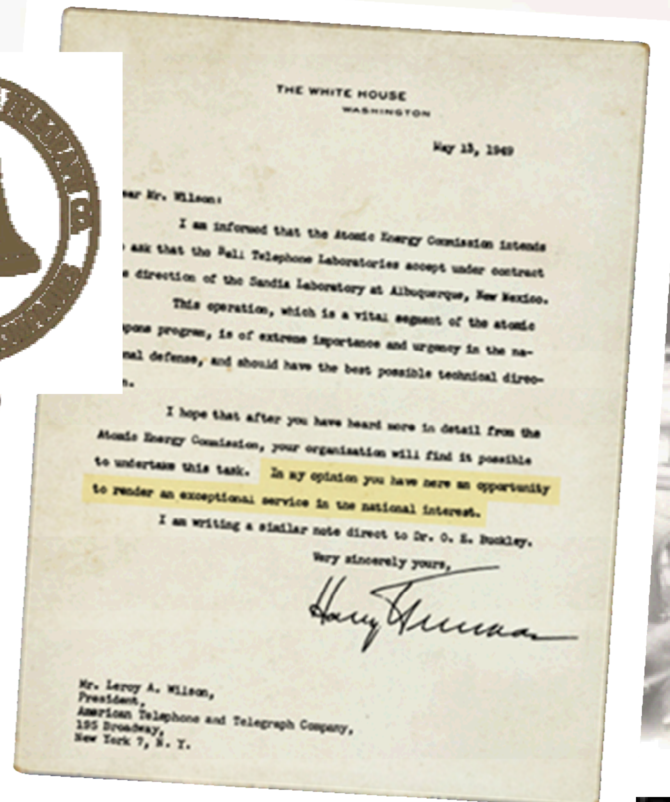
- Sandia National Labs
  - Overview
  - Education and Sandia Career
  - Materials and Process Sciences Center
  - Power Sources Technology Group
- Failure Ingredients
- Failure Examples
- Making the Most of Our Failures

Sandia's History:
Exceptional service in the national interest

- July 1945: Los
  creates Z Divi
- Nonnuclear c
  engineering
- November 1,
  Sandia Labora
  established



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



THE WHITE HOUSE
WASHINGTON

May 13, 1949

ear Mr. Wilson:

I am informed that the Atomic Energy Commission intends ask that the Bell Telephone Laboratories accept under contract e direction of the Sandia Laboratory at Albuquerque, New Mexico.

This operation, which is a vital segment of the atomic pons program, is of extreme importance and urgency in the nanal defense, and should have the best possible technical direc-n.

I hope that after you have heard more in detail from the Atomic Energy Commission, your organization will find it possible to undertake this task. In my opinion you have here an opportunity to render an exceptional service in the national interest.

I am writing a similar note direct to Dr. O. E. Buckley.

Very sincerely yours,

Harry Truman

Mr. Leroy A. Wilson,
President,
American Telephone and Telegraph Company,
195 Broadway,
New York 7, N. Y.

Governance of Sandia Laboratories

Sandia Corporation

- AT&T: 1949–1993
- Martin Marietta: 1993–1995
- Lockheed Martin: 1995–2017
- National Technology and Engineering Solutions of Sandia, LLC, a wholly owned subsidiary of Honeywell International Inc.: 2017-present
- Government owned, contractor operated

Federally funded research and development center

Sandia Sites

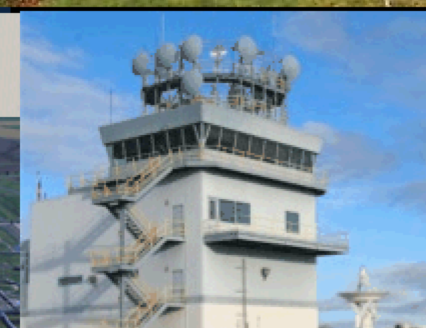
Albuquerque, New Mexico

Livermore, California

Kauai, Hawaii

Waste Isolation Pilot Plant,
Carlsbad, New Mexico

Pantex Plant,
Amarillo, Texas

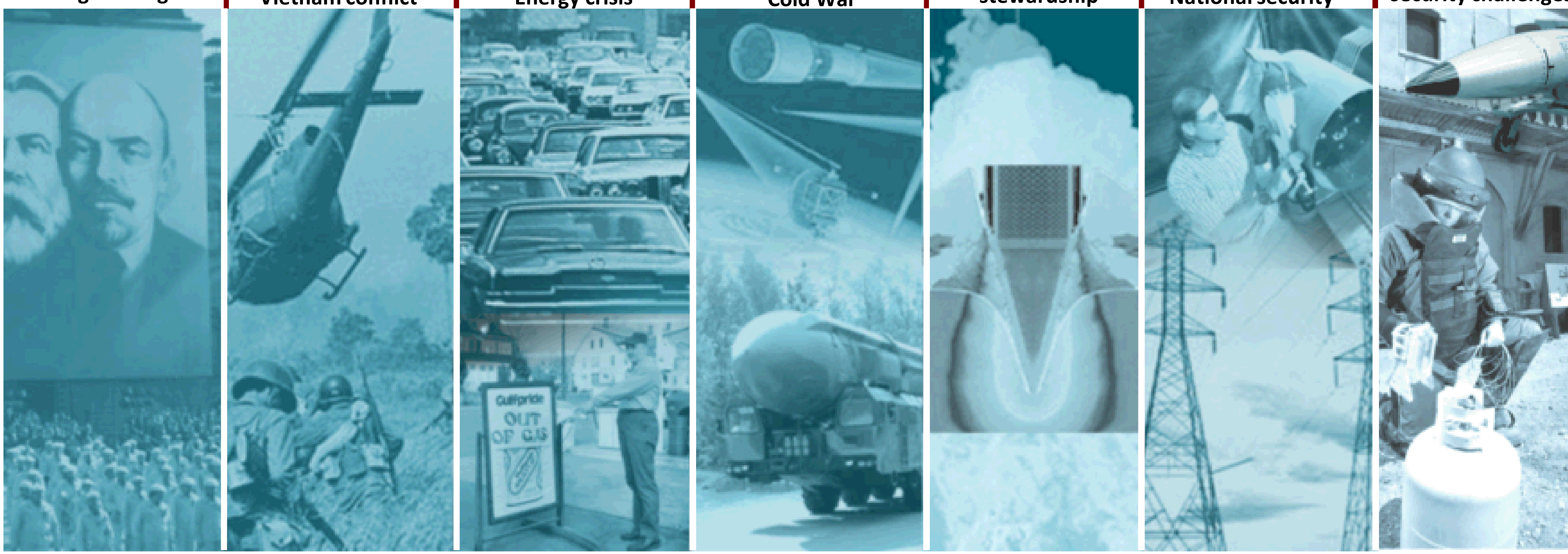


Tonopah,
Nevada

Sandia develops advanced technologies to ensure global peace

Sandia Addresses National Security Challenges

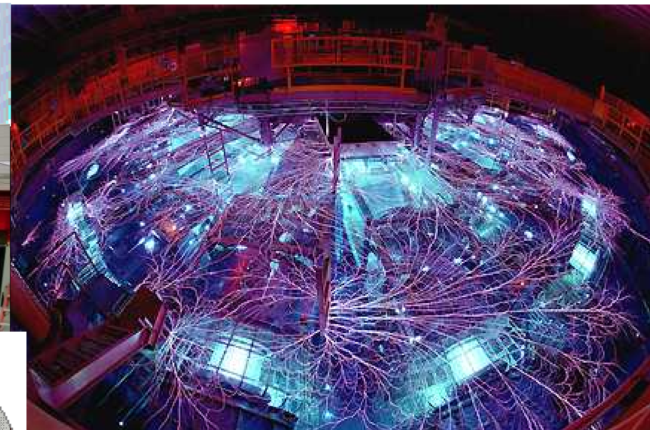
1950s	1960s	1970s	1980s	1990s	2000s	2010s
Nuclear weapons	Development engineering	Multiprogram laboratory	Missile defense work	Post–Cold War transition	START Post 9/11	LEPs Cyber, biosecurity proliferation
Production and manufacturing engineering	Vietnam conflict	Energy crisis	Cold War	Stockpile stewardship	National security	Evolving national security challenges



Sandia's Science and Technology Framework

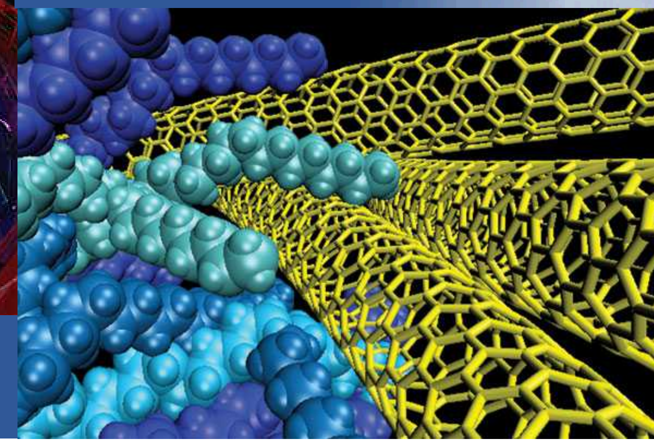
Strong research foundations play a differentiating role in our mission delivery

Computing & Information Sciences

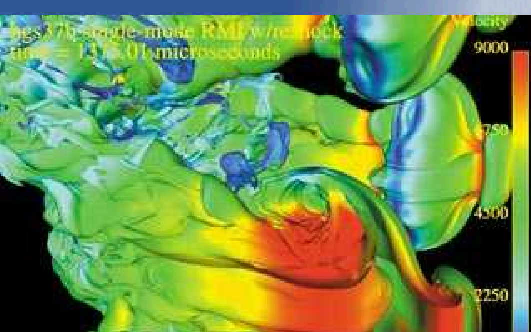


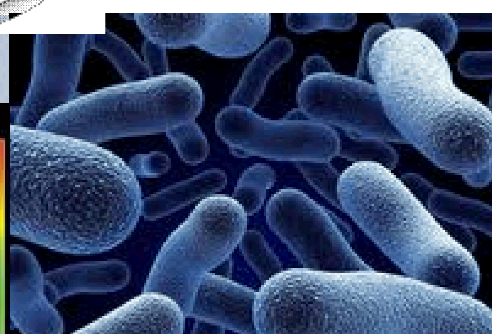
Radiation Effects & High Energy Density Science

Materials Sciences



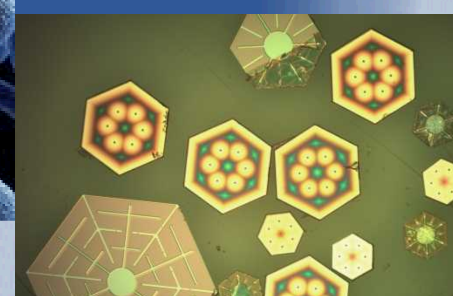
Engineering Sciences

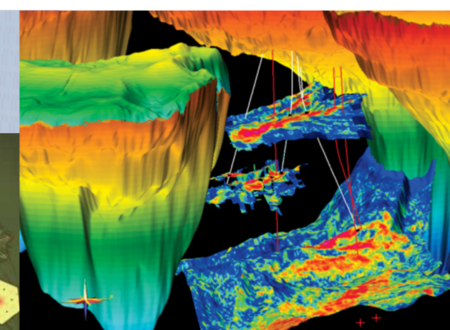




Bioscience

Nanodevices & Microsystems





Geoscience

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 – Component Sci., Engr. & Production (2015-2018)

Group 2540 – Power Sources Technology Group

Dept. 2547 – Power Sources Component Development

Materials and Process Sciences R&D

Synthesis and Processing

Characterization

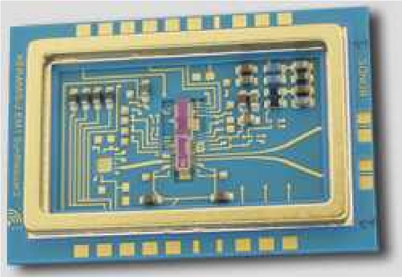
Performance and Reliability

- Mechanical Performance
  - Ductile Materials
  - Brittle Materials

Brittle Material Containing Components

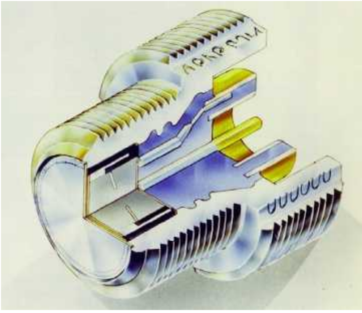
Materials

- 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

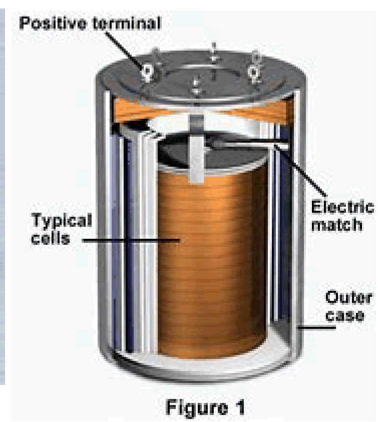
Power Sources

- 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

Failure in Brittle Materials

Strength is Flaw Size Dependent for a Brittle Material

When σ_a or σ_r **>** $\sigma_f = \dfrac{K_{IC}}{Y\sqrt{c}}$

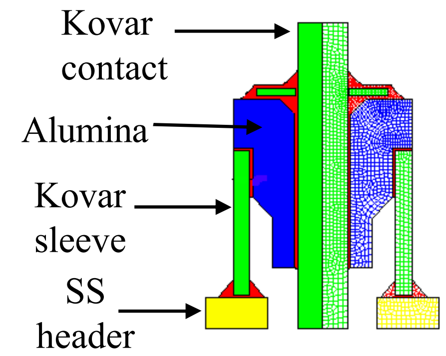
Where:

σ_f = fracture strength
K_{IC} = fracture toughness or critical stress intensity
c = flaw size
Y = geometrical factor

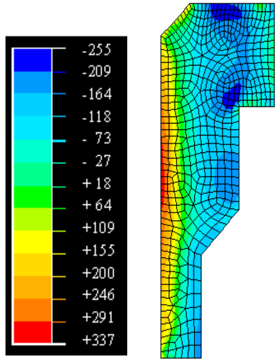
σ_a = applied stress
σ_r = residual stress

Ingredients for Brittle Material Failure

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



- 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

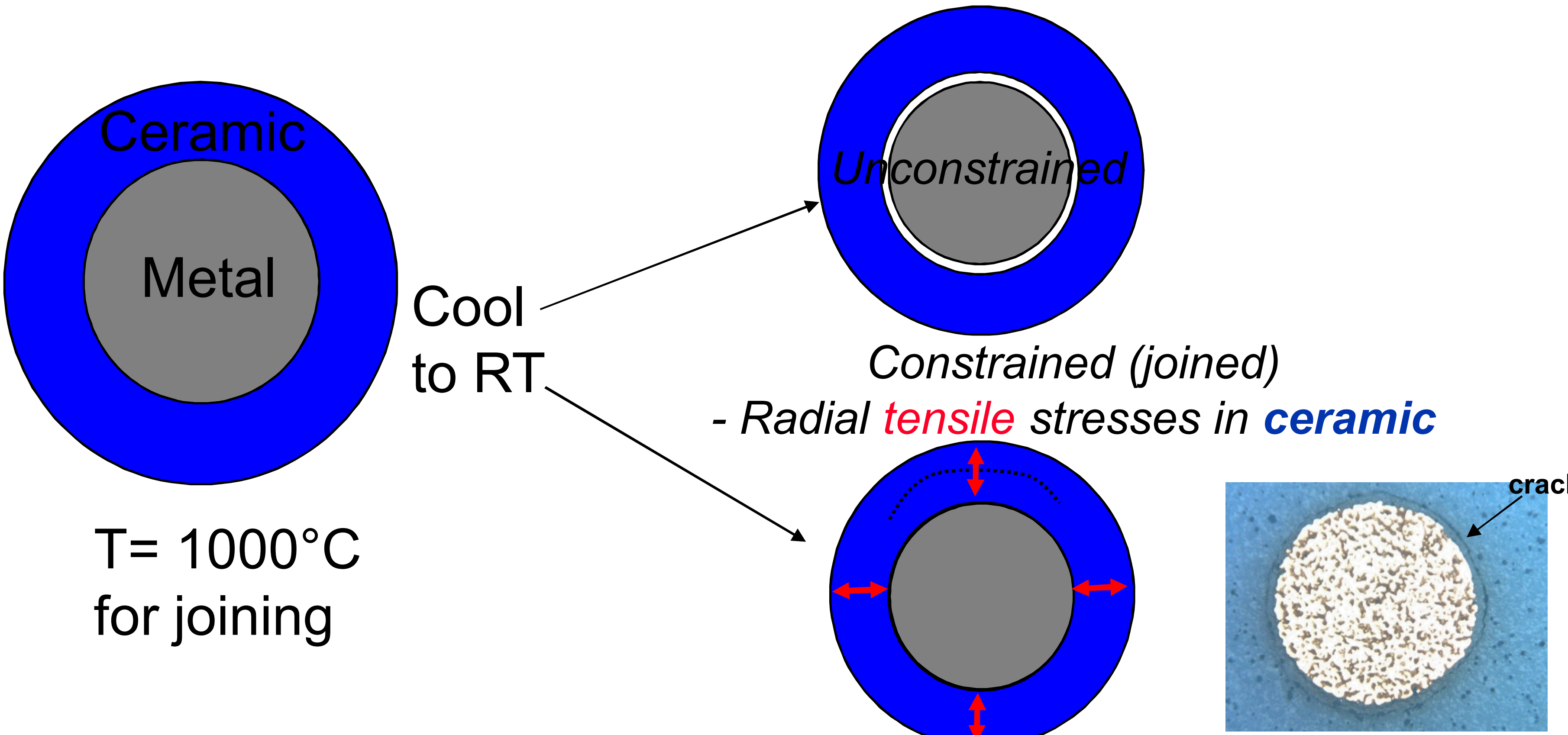


max. prin. stress (MPa) in alumina

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

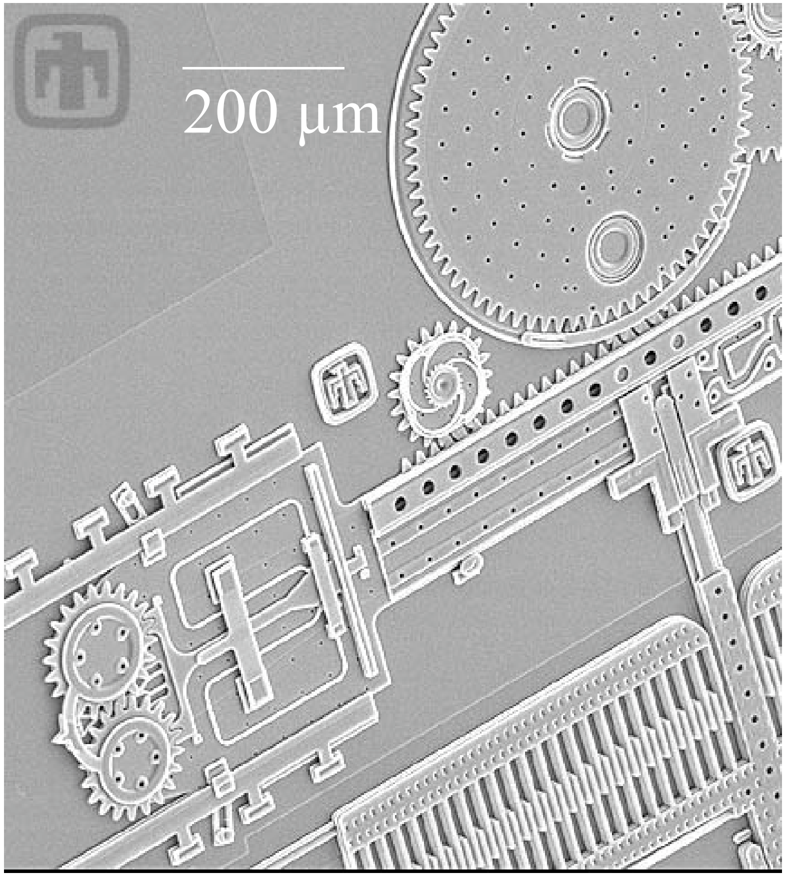


Brittle Materials Failure Examples

- 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)

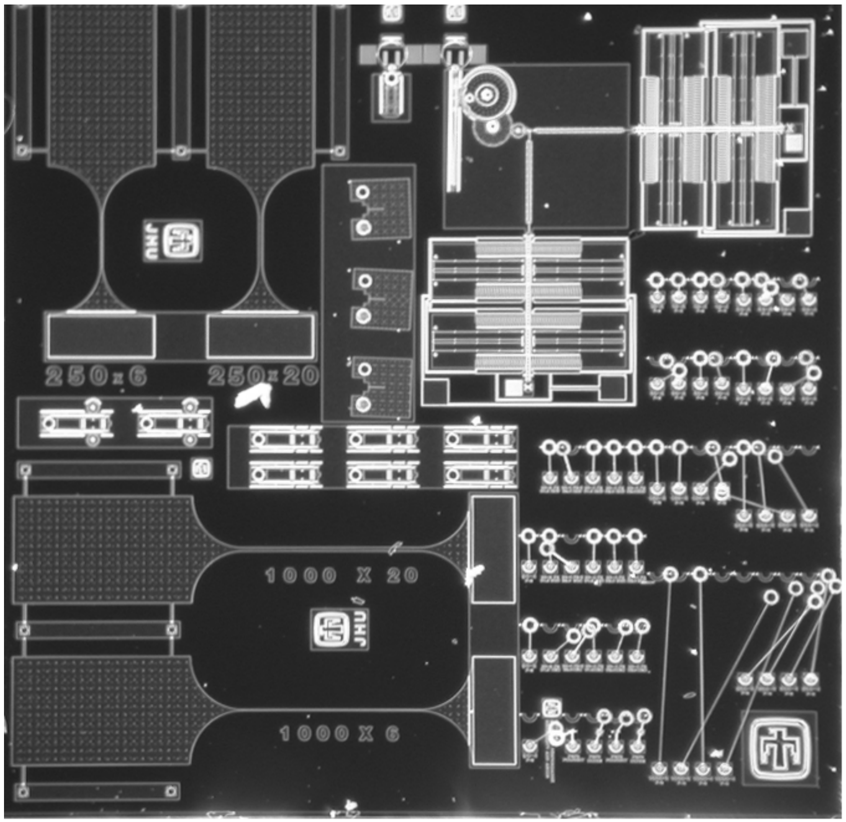
Fracture Strength Testing and Failure Origins for Polysilicon MEMS Materials

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

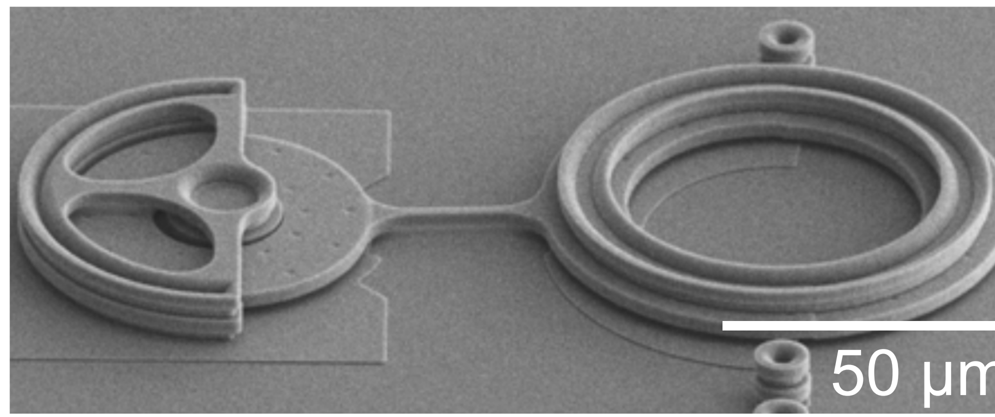


- Modest structural properties (K_{IC}=1.0-1.4 MPa√m)
- High degree of reactivity with oxygen, water and other reagents

Polysilicon Mechanical Testing Samples Fabricated Directly on the Die

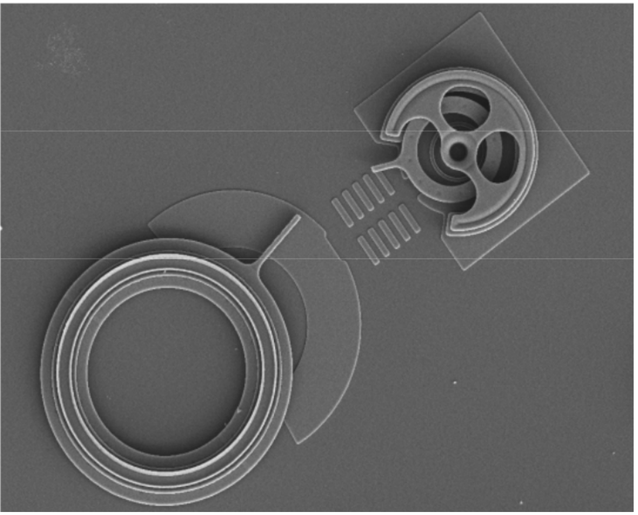


Pull-tab tensile strength sample.

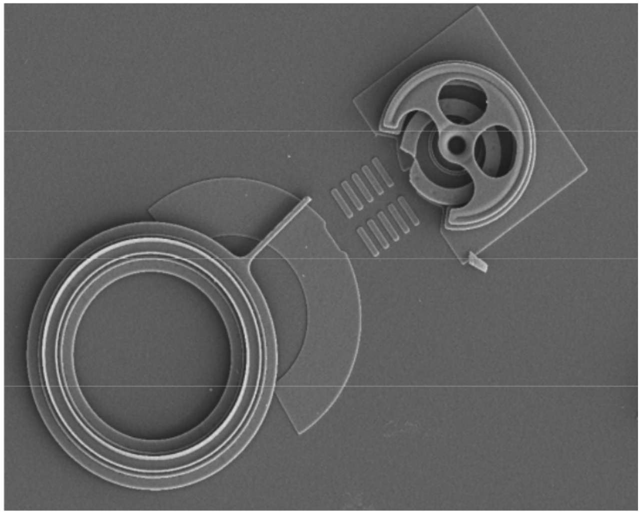


2 of 3 Failure Modes for 48 First Generation Polysilicon Samples Due to Pull-Tab Design

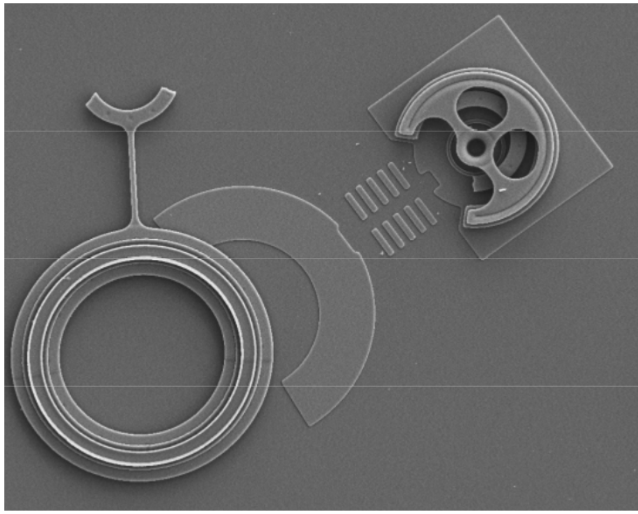
Average σ_f = 2.24 GPa ±0.35



7 Strongest

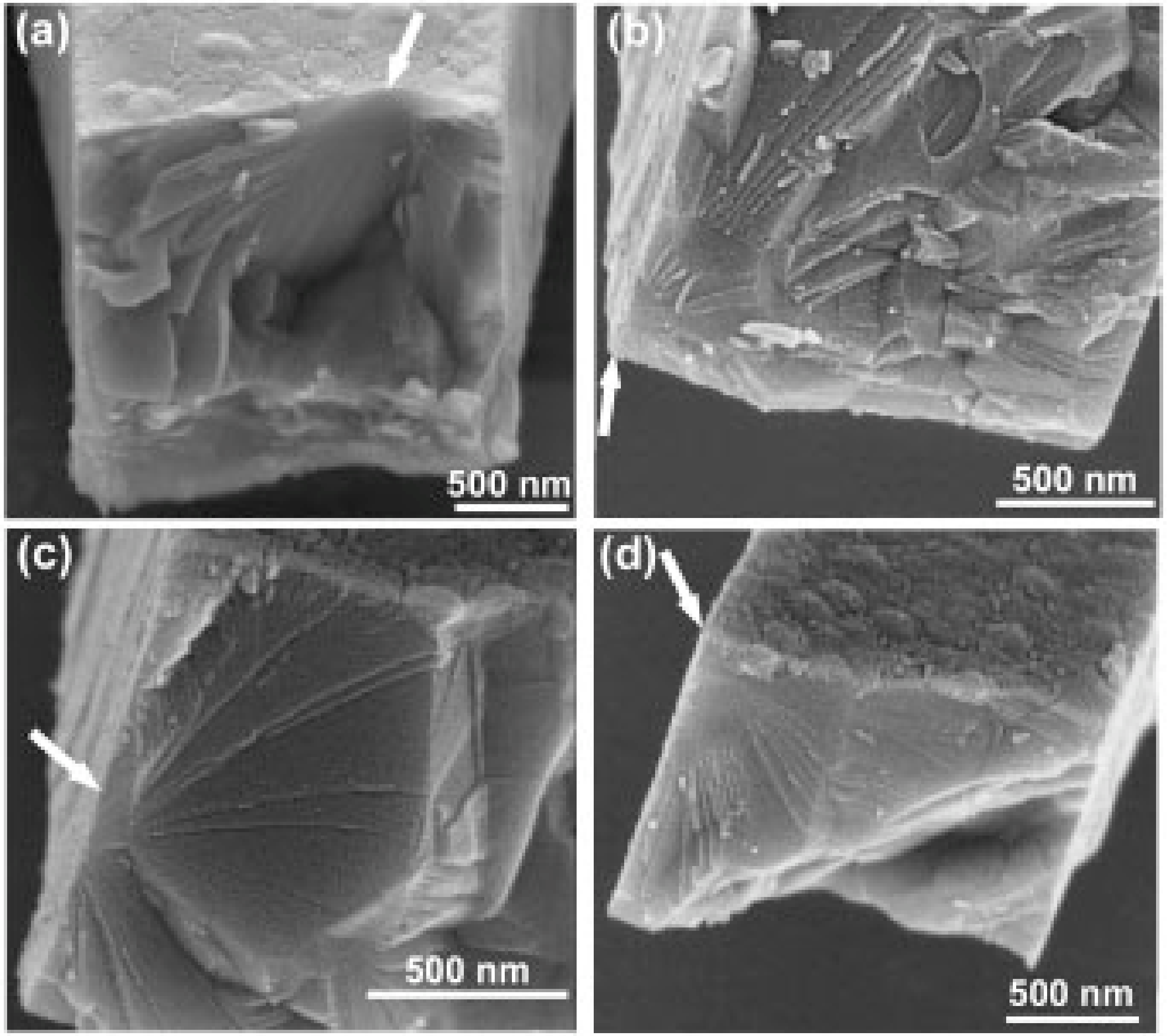


Intermediate Strength



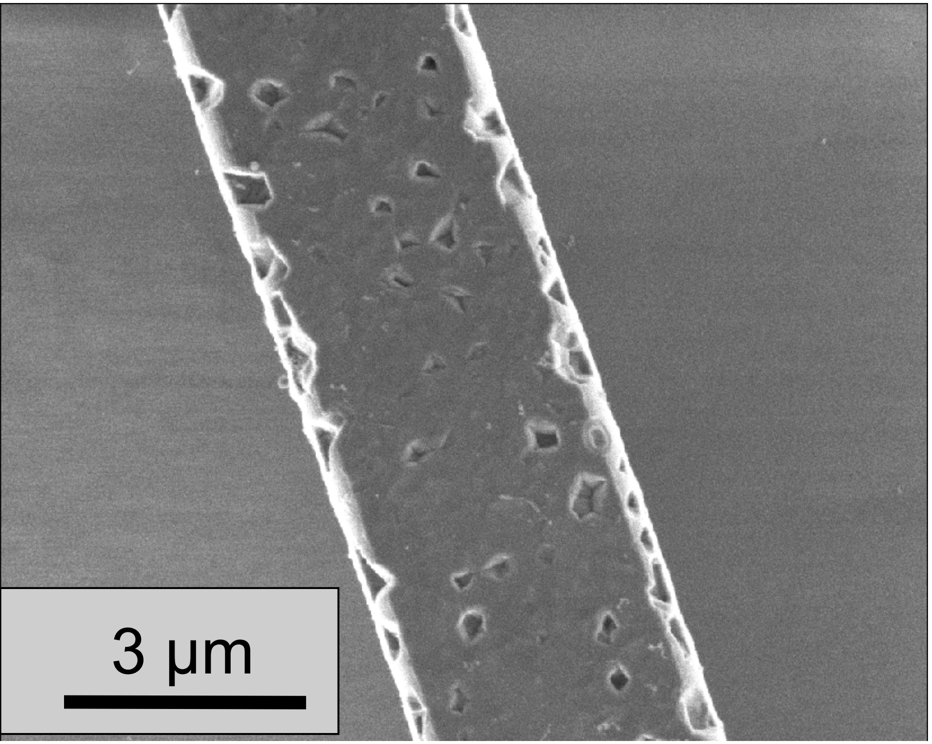
Weakest

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

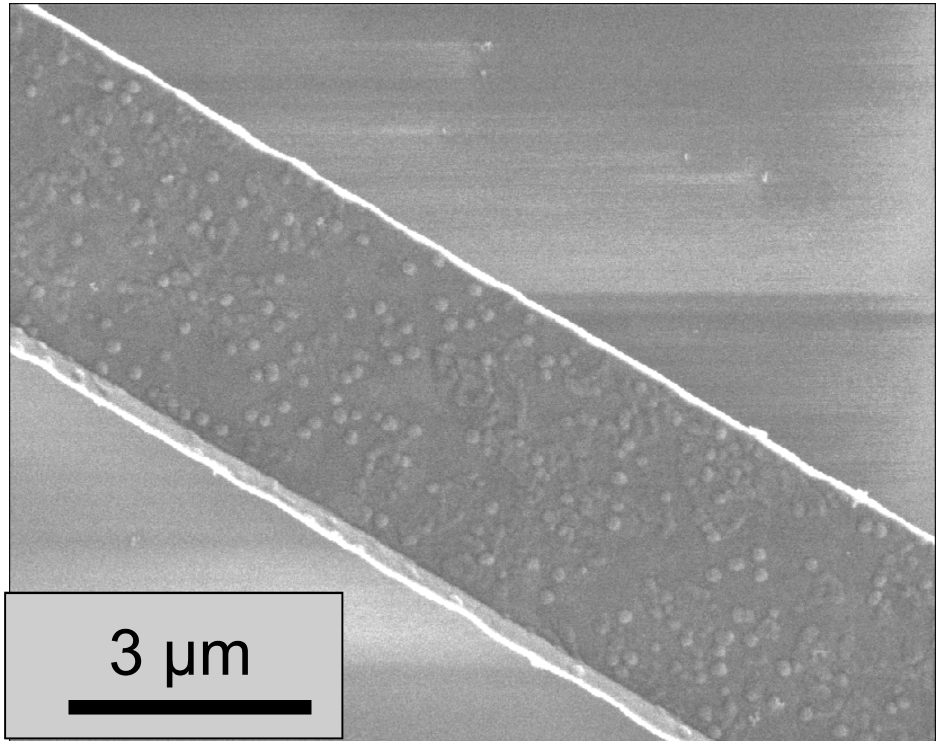


B. Boyce, Sandia, 2005

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

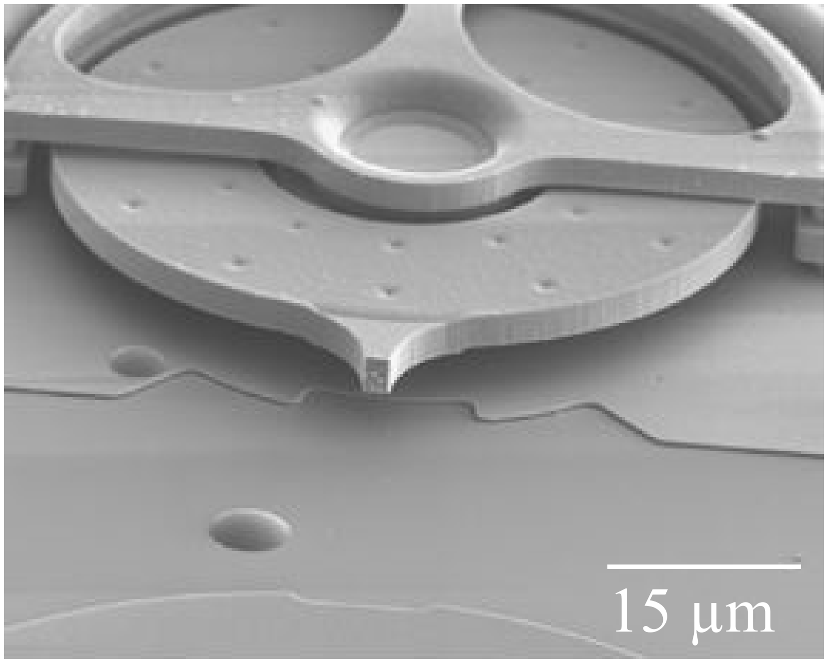


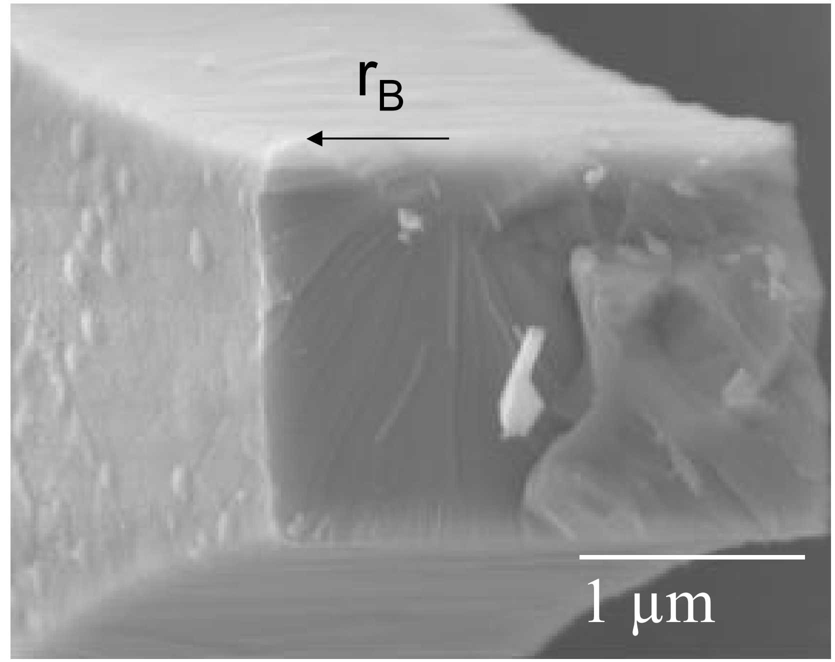
Weak



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 MPa√m
(Tsai & Mecholsky, 1992)

Therefore, σ_f = 2.1 GPa

Ingredients for Polysilicon Microelectro-Mechanical Systems (MEMS) Failures

When $\sigma_{applied}$ **>** $\sigma_f = \dfrac{K_{IC}}{Y\sqrt{c}}$

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

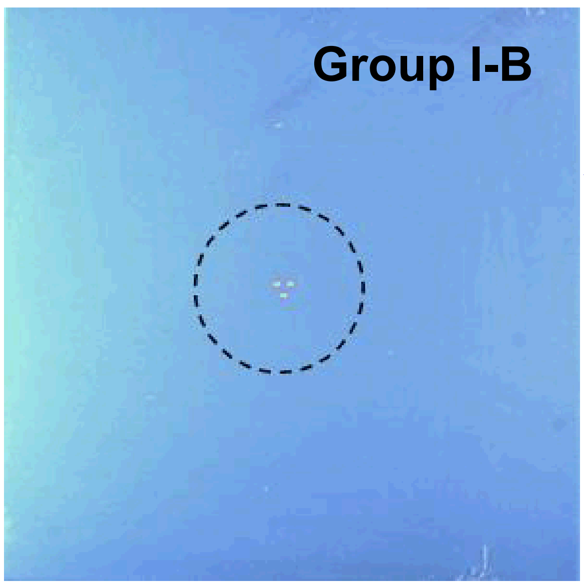
- Intrinsic microstructural features
- ***Processing related***
- Handling or in-service damage

Material degradation (loss of strength)

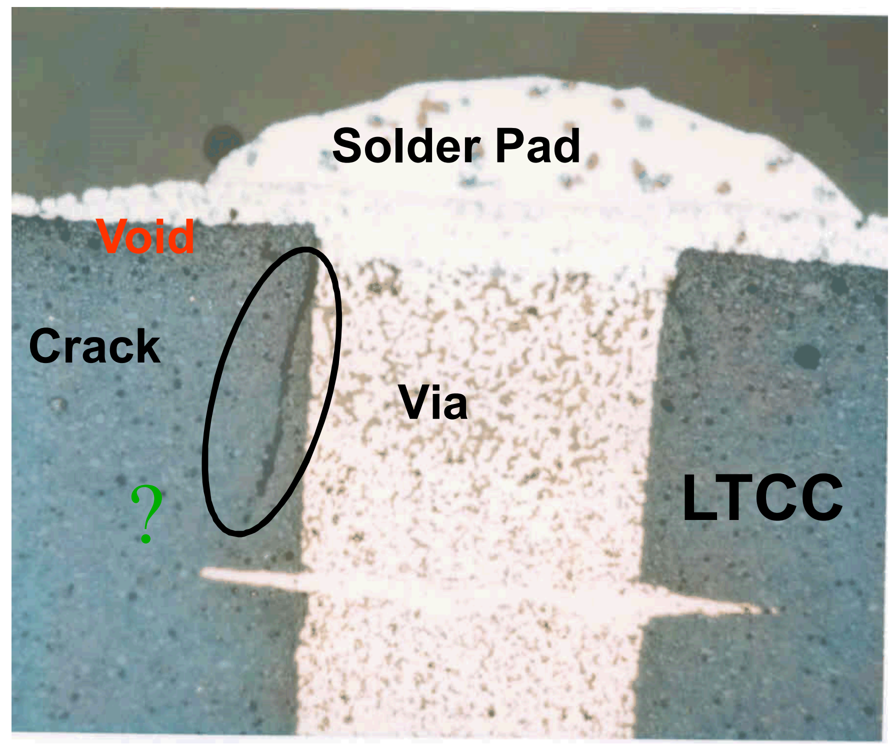
- Environmental effects, decrease in K_{IC}
- Increase in flaw size - subcritical crack growth

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

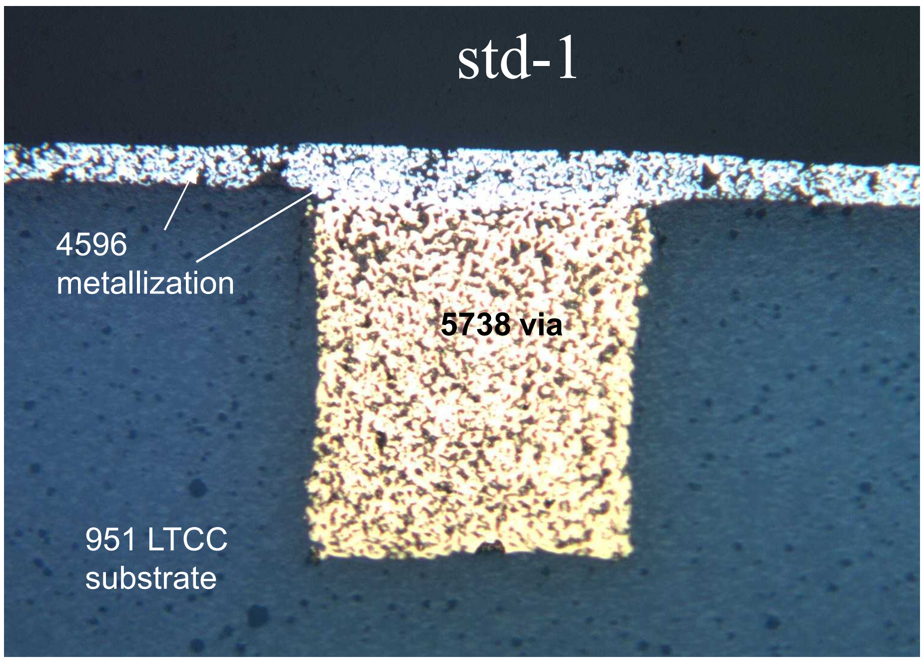
Low temperature co-fired ceramic (LTCC) parts were made with multiple layers of DuPont 951 green tape™ with stacked electrical vias

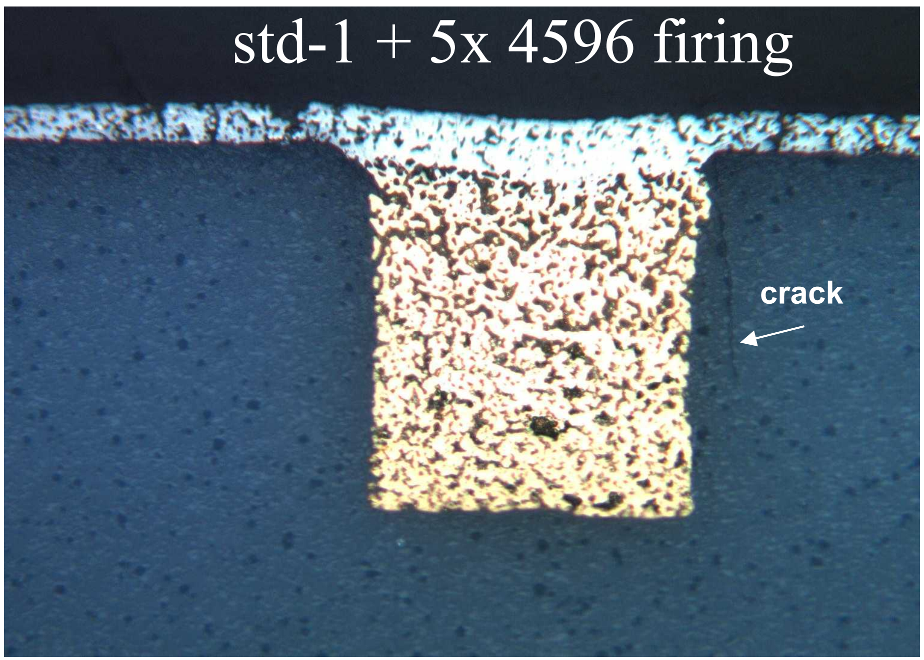


Vias through 4 layers of LTCC

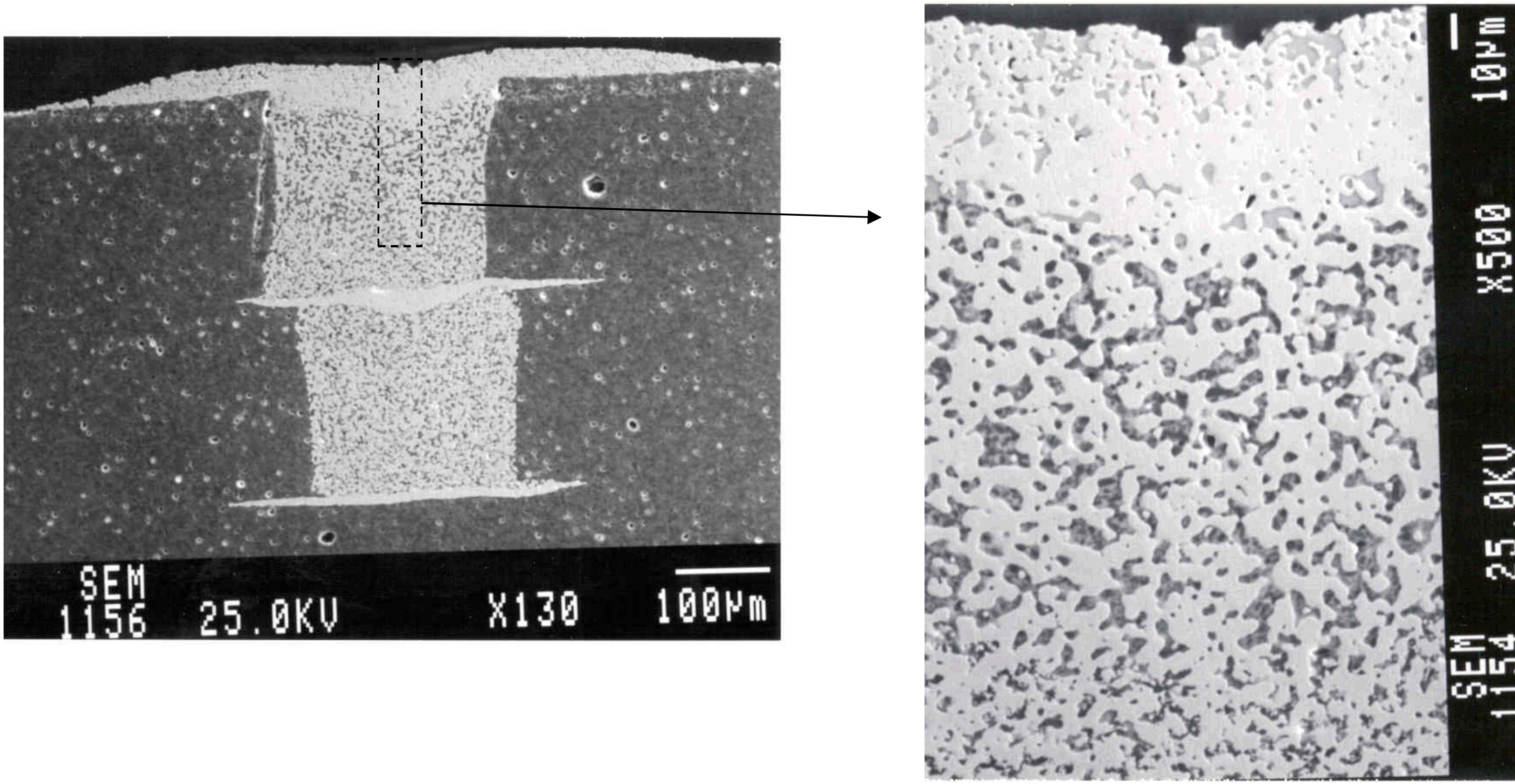


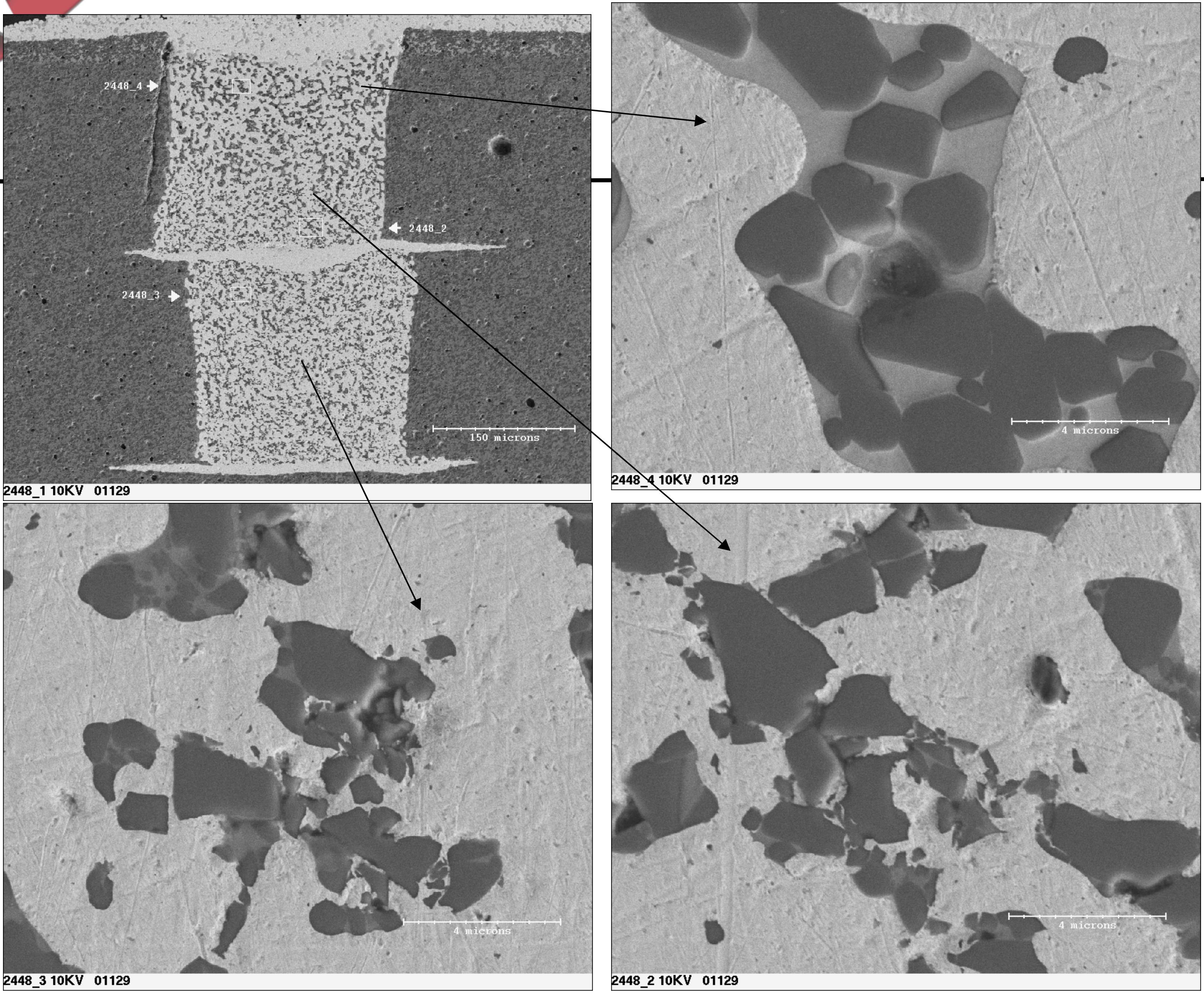
Longitudinal Sections Show LTCC, 5837 Via, and 4596 Metallization



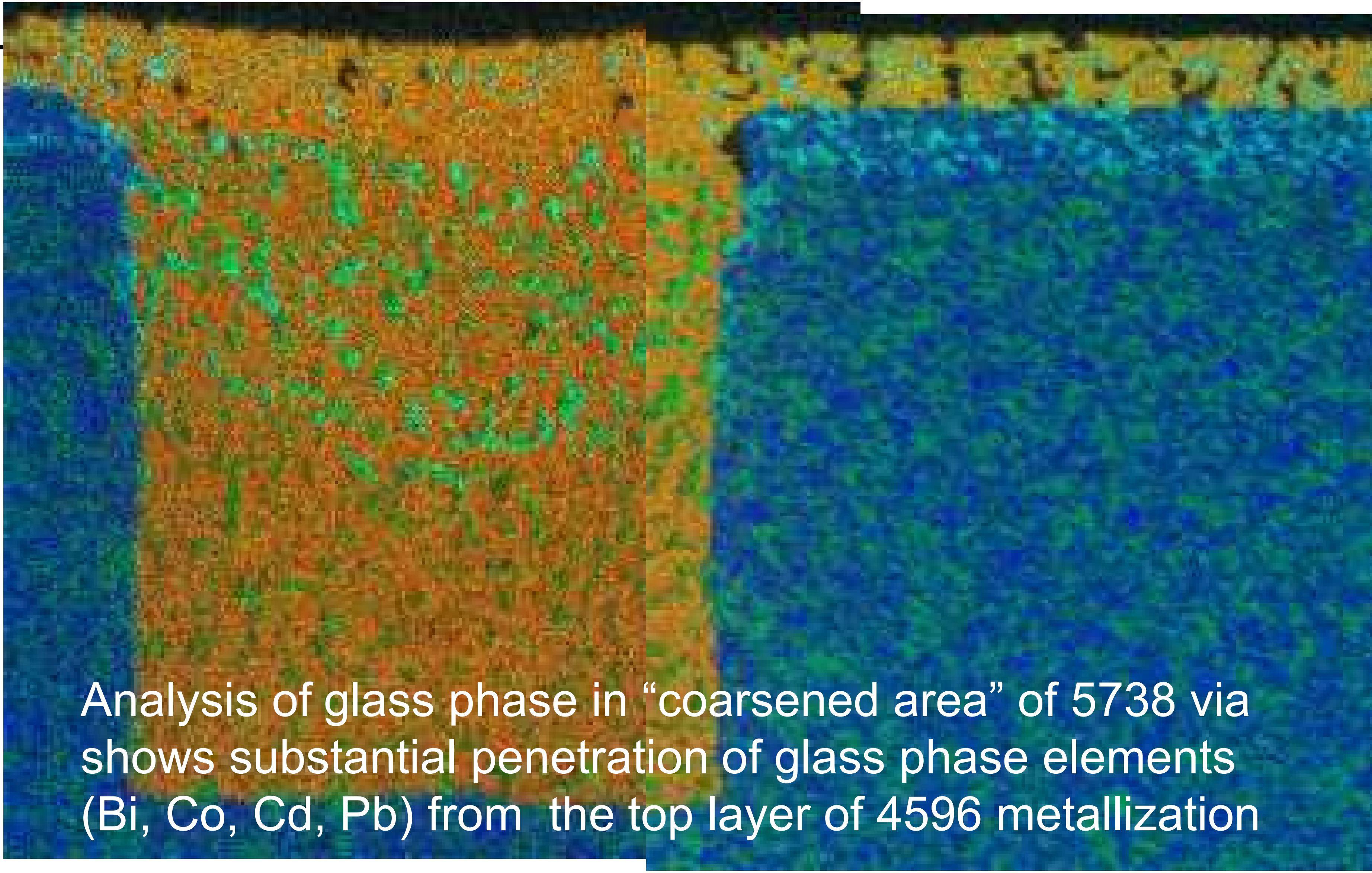


Electron Microprobe Shows Interactions Between Surface Metallization and Via Material




2448_4
2448_2
2448_3
150 microns
2448_1 10KV 01129
4 microns
2448_4 10KV 01129
4 microns
2448_3 10KV 01129
4 microns
2448_2 10KV 01129

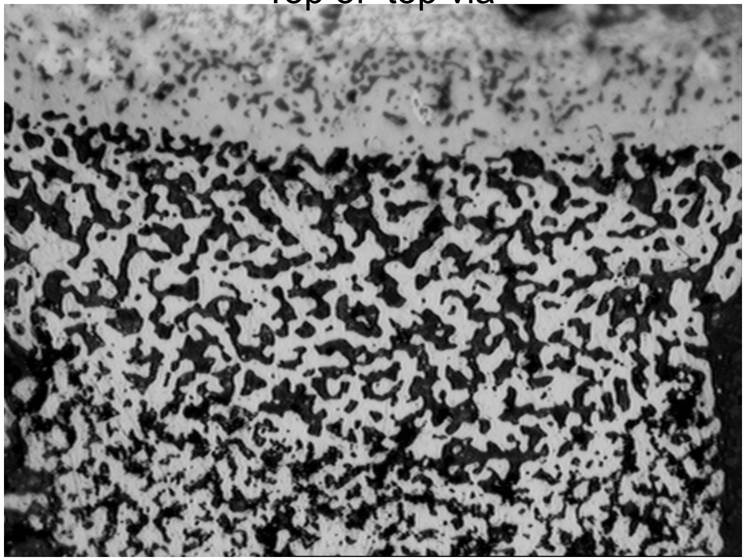
SEM Spectral Image Analysis of Std1_5xFires Parts



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

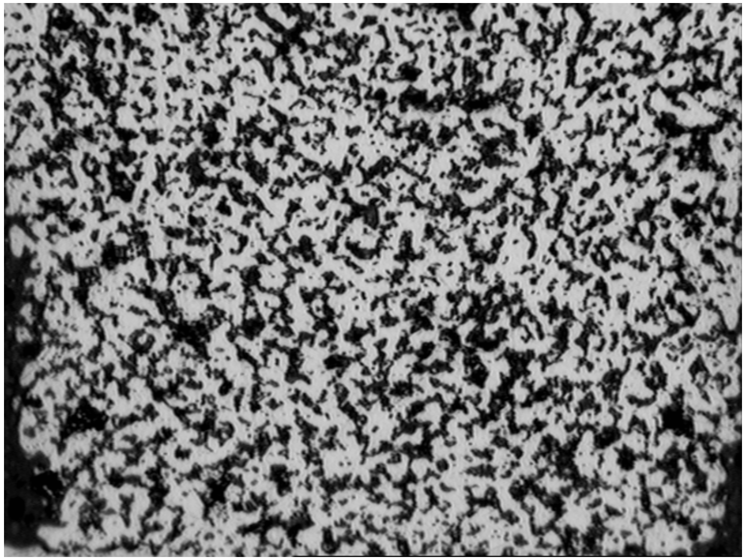
Estimated CTE for Via used in FEA Stress Predictions Based on Original Volume Fractions

Top of top via



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

Bottom of top via and internal vias



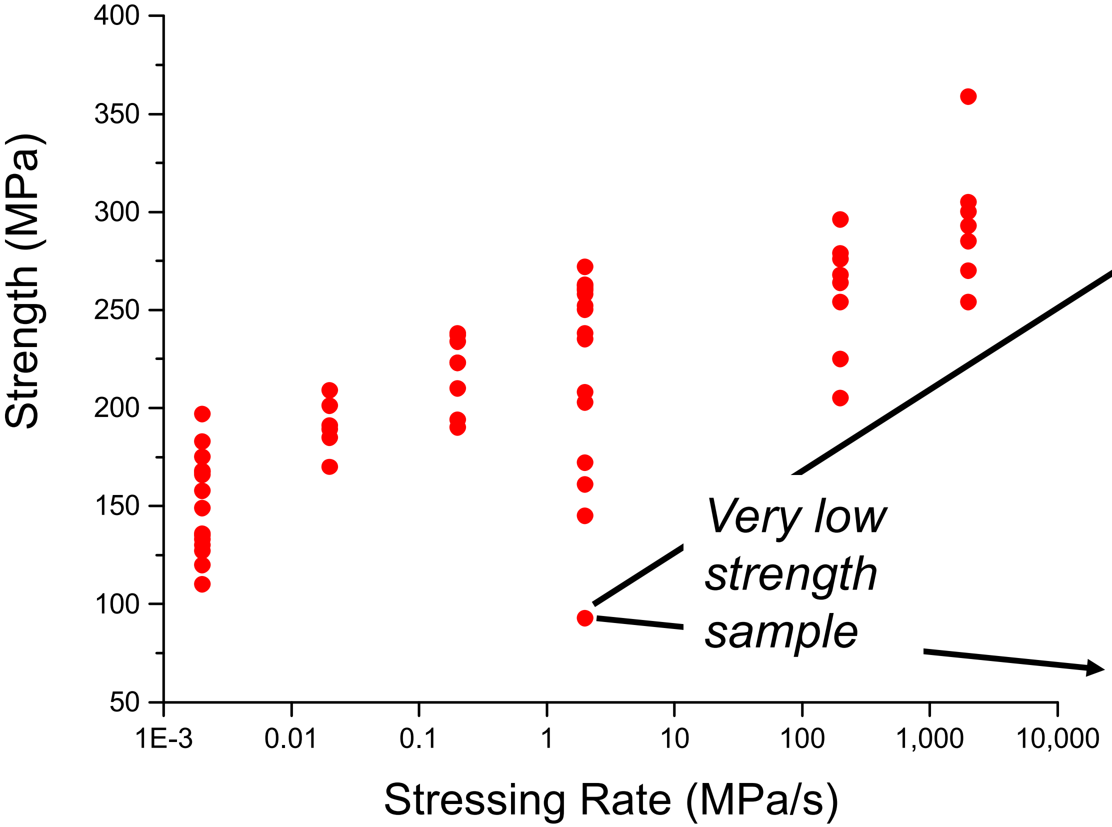
area fraction metal= 60%
area fraction other= 40%

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

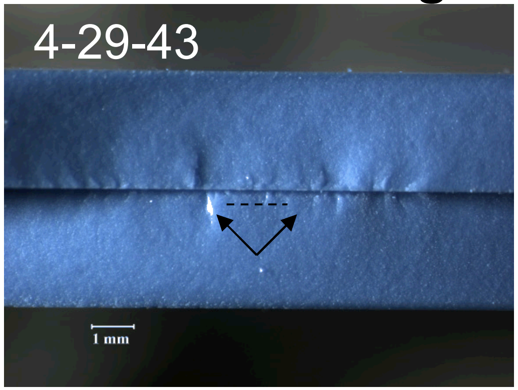
When $\sigma_{applied}$ or residual > $\sigma_f = \dfrac{K_{IC}}{Y\sqrt{c}}$

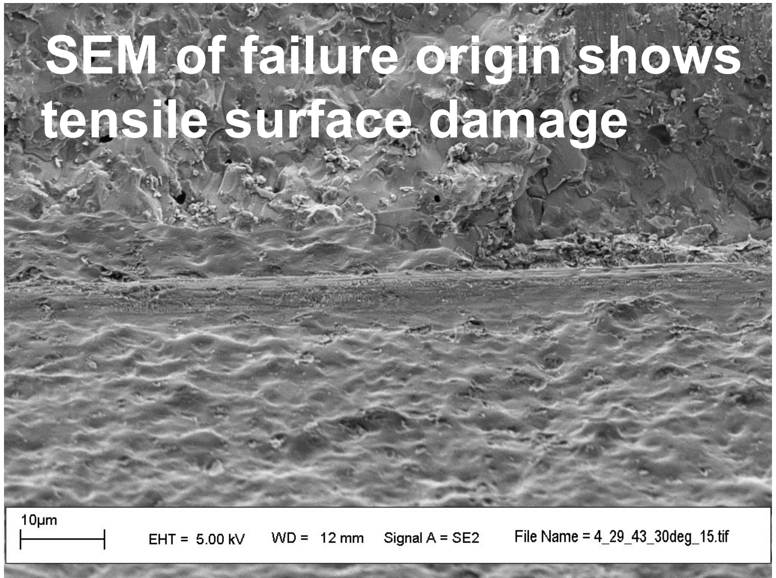
- Excessive stress
  - Excessive applied stress or macroscopic stress concentrations
  - ***Materials combination and/or processing that lead to excessive residual stress***
- Presence or introduction of defects
  - Intrinsic microstructural features
  - Processing related
  - Machining, in-service damage
- 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



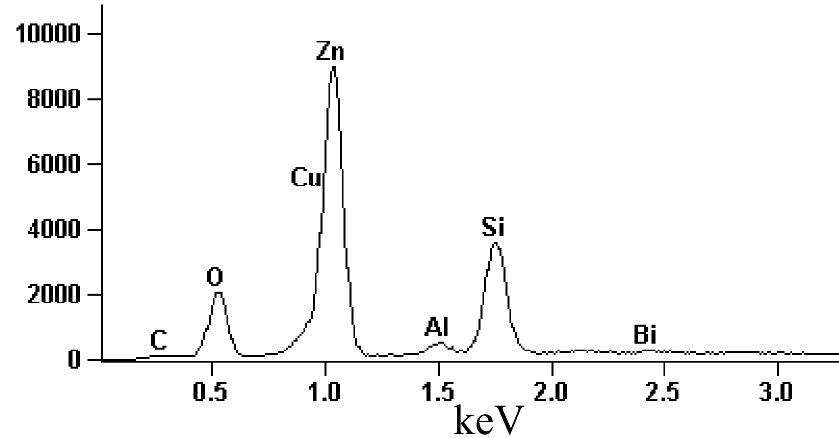


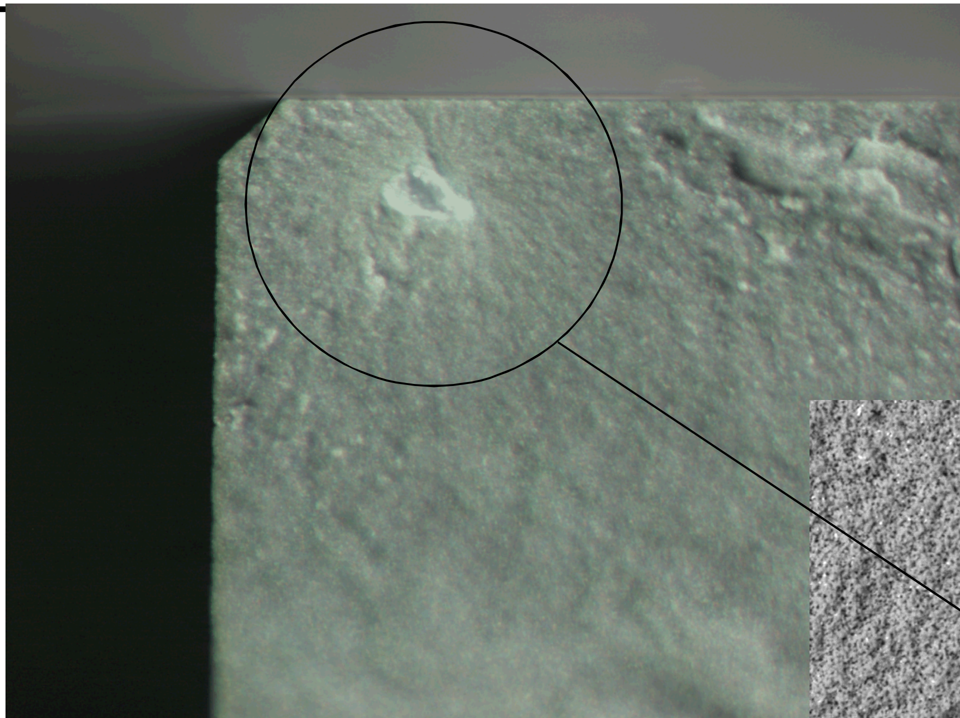
Ingredients for Failure of Low Temp Co-Fired Ceramic (LTCC) Strength Samples

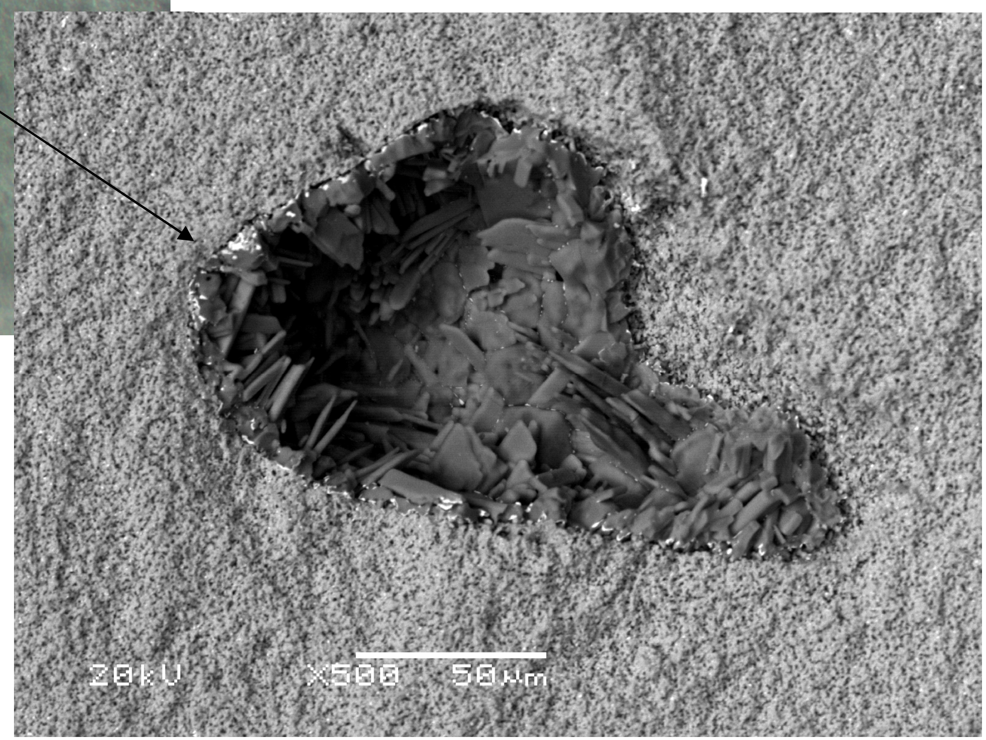
When $\sigma_{\text{applied or residual}}$ > $$\sigma_f = \frac{K_{IC}}{Y\sqrt{c}}$$

- 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}

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

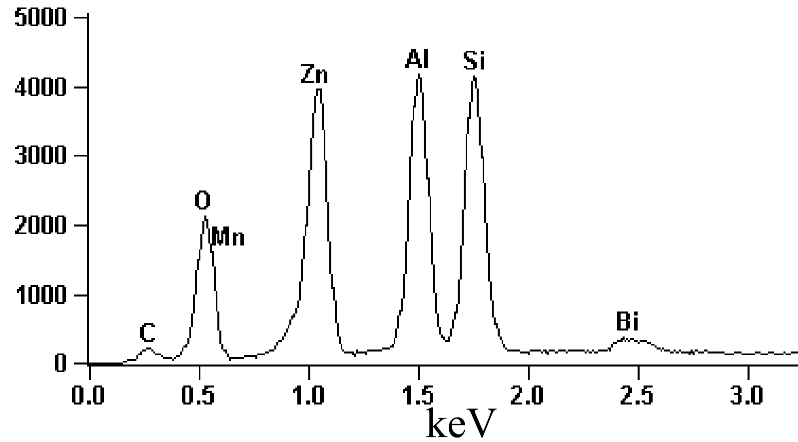


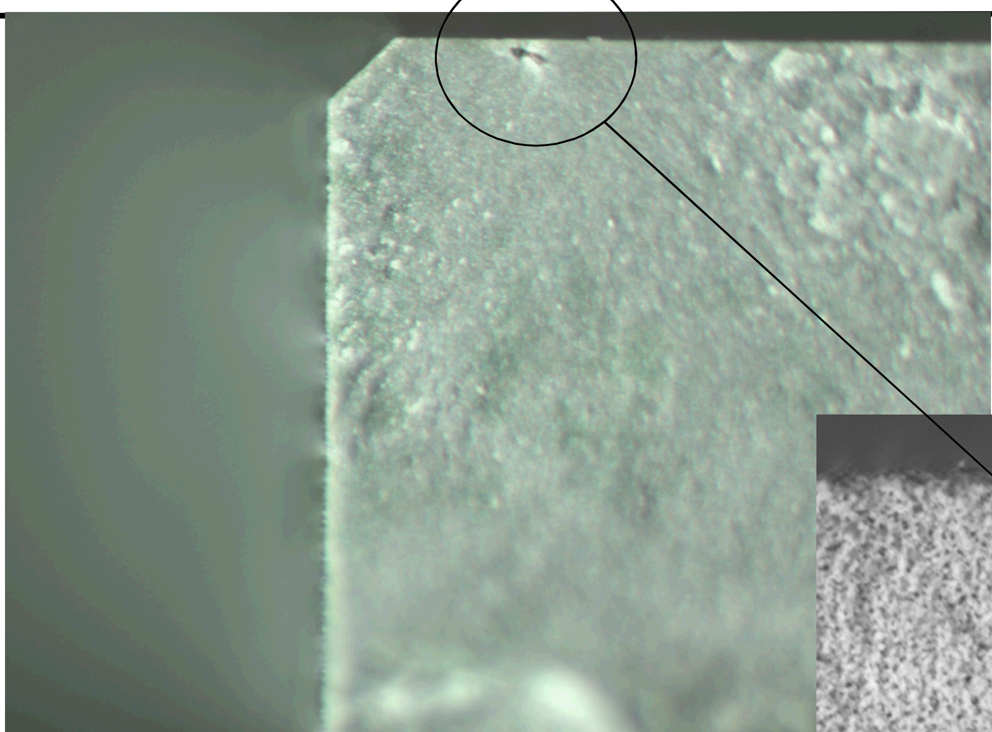


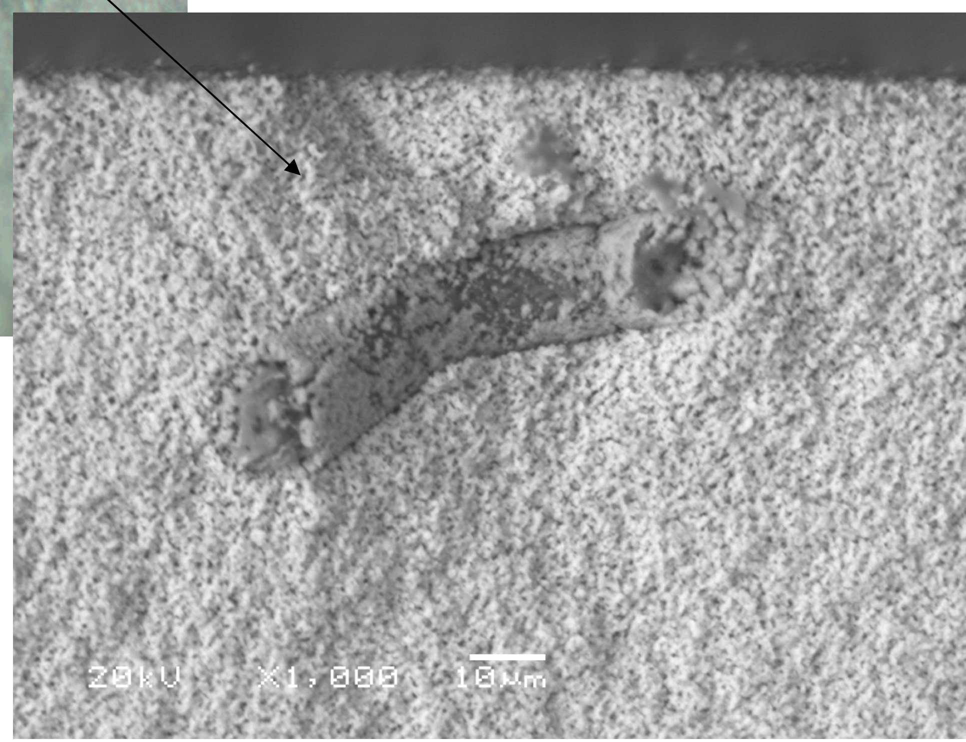


Si rich inclusion

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



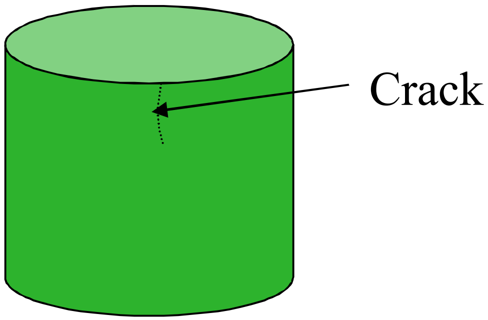


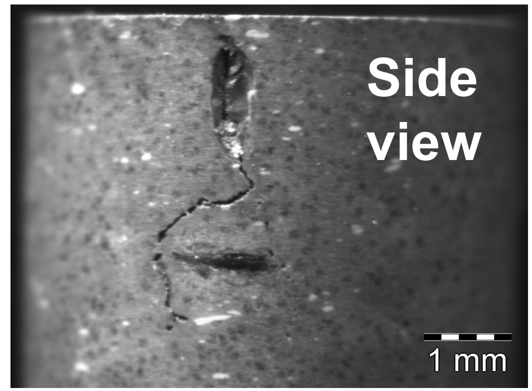


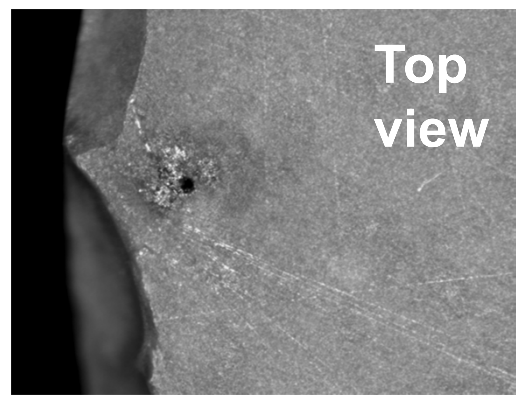
Mn, Al, Si rich inclusion

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

Prior to electrical pulse testing







- Cracks parallel to current flow produce early breakdowns

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

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

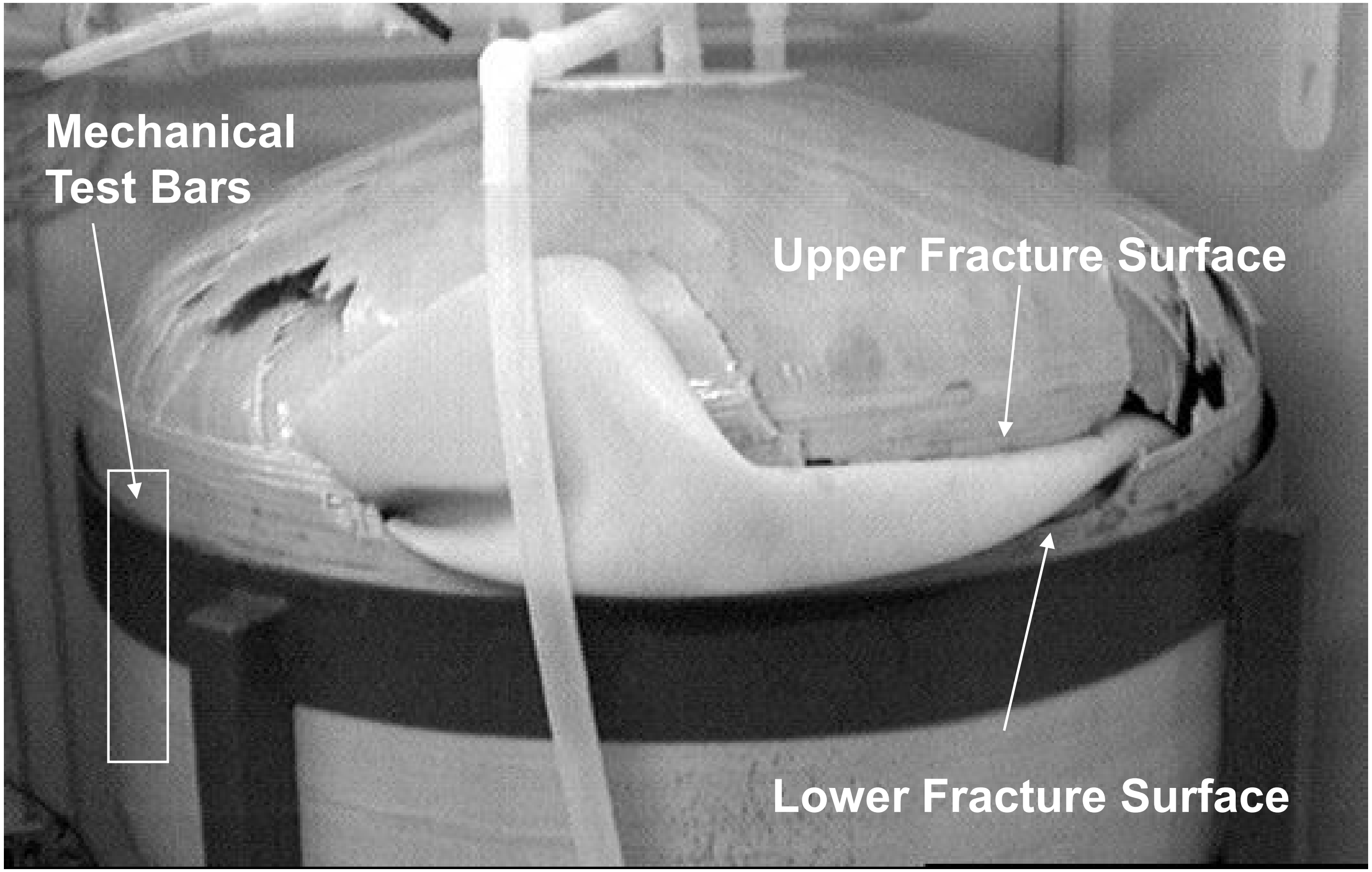
Excessive stress
- Excessive applied stress or macroscopic stress concentrations
- Materials combinations and/or processing that lead to excessive residual stress

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}

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

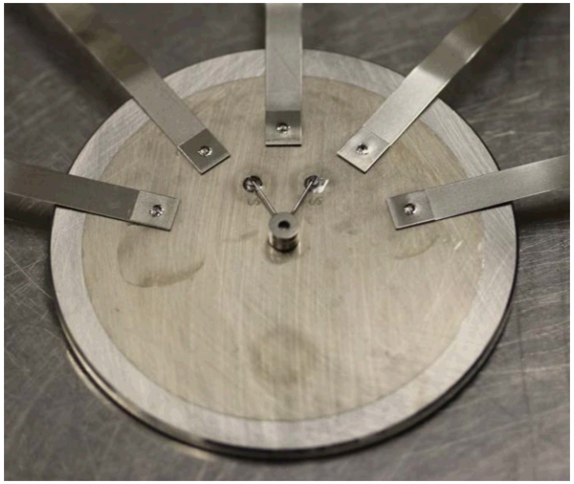
Ingredients for Failure of a Fiberglass Reinforced Composite (FRC) Tank

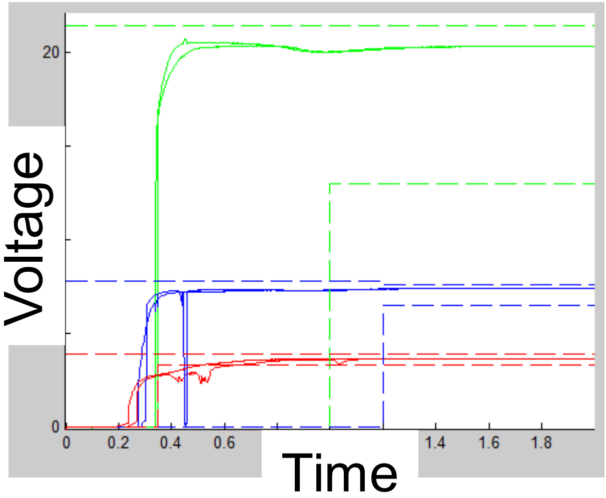
When $\sigma_{\text{applied or residual}}$ > $$\sigma_f = \frac{K_{IC}}{Y\sqrt{c}}$$

- 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}***

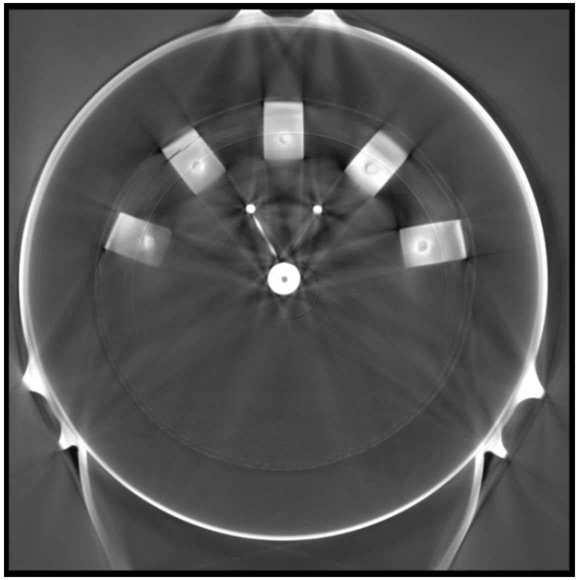
All of the above!

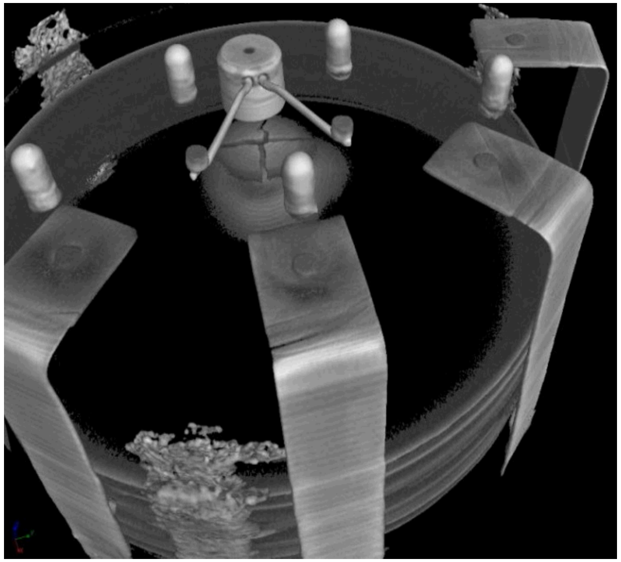
Ni Lead Failures in Thermal Batteries During Testing in Various Vibration Environments



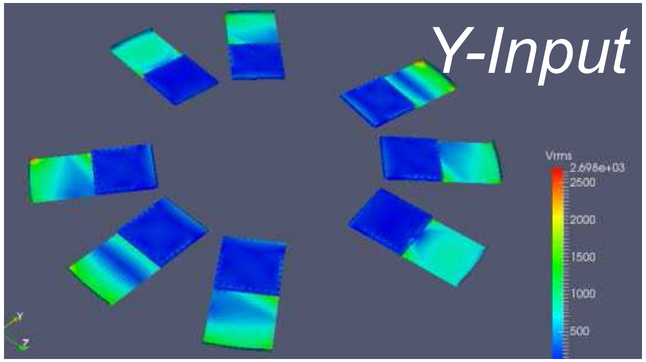


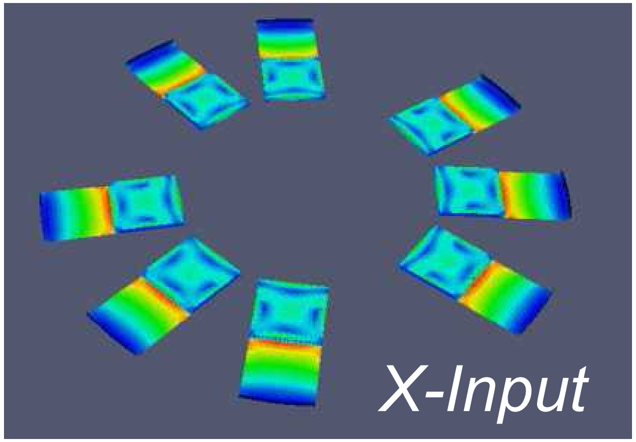
Electrical testing of a thermal battery in a vibration environment showed unacceptable drops in voltage vs. time.



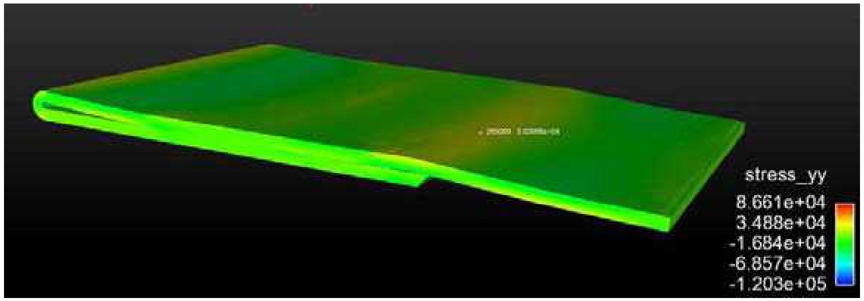


Stress Analyses of Ni Leads For Vibration Tests Showed Stresses were Highly Axis Dependent

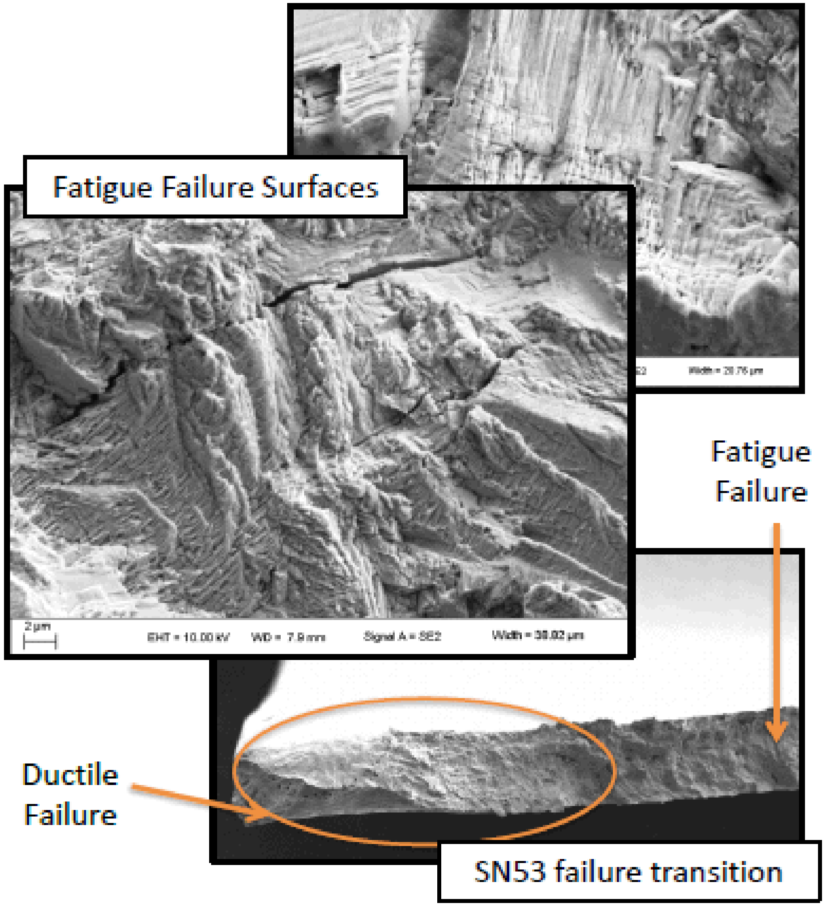




Fracture Surface Analysis Confirmed High Cycle Fatigue Failure

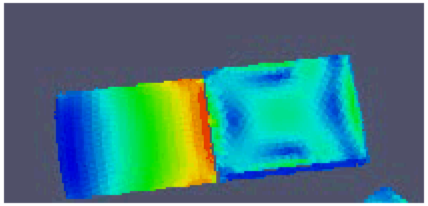


- All three failed Ni leads show surface that appears brittle, with tightly spaced striations throughout
- Based on these features, failures are consistent with low amplitude, high cycle fatigue (transportation vibration environment)
- Remainder of lead was 'failed' in lab environment – portion exhibited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

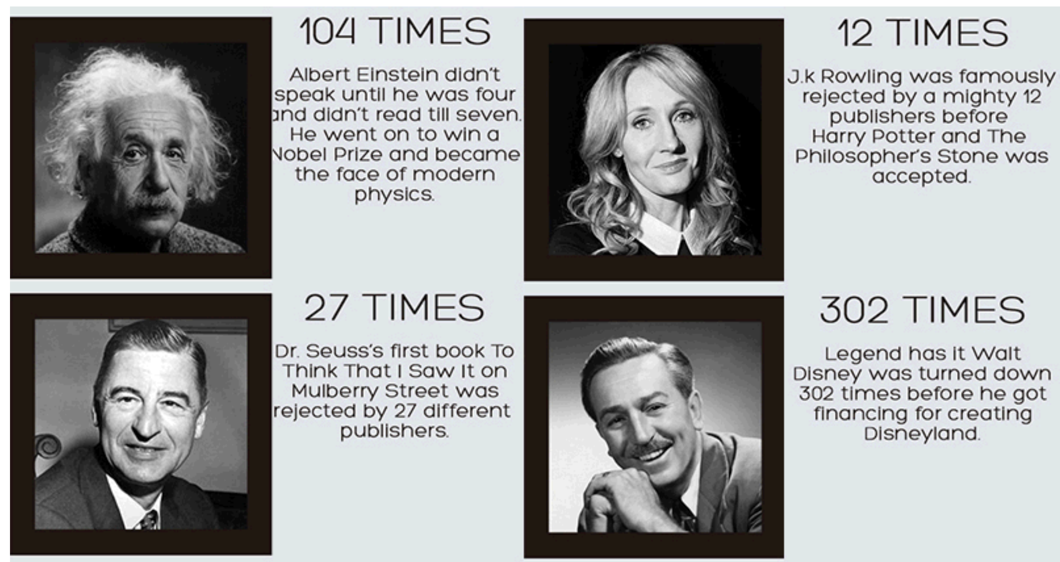


Making the Most of Our Failures

- 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



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

Thank you!

Failure Quotes

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

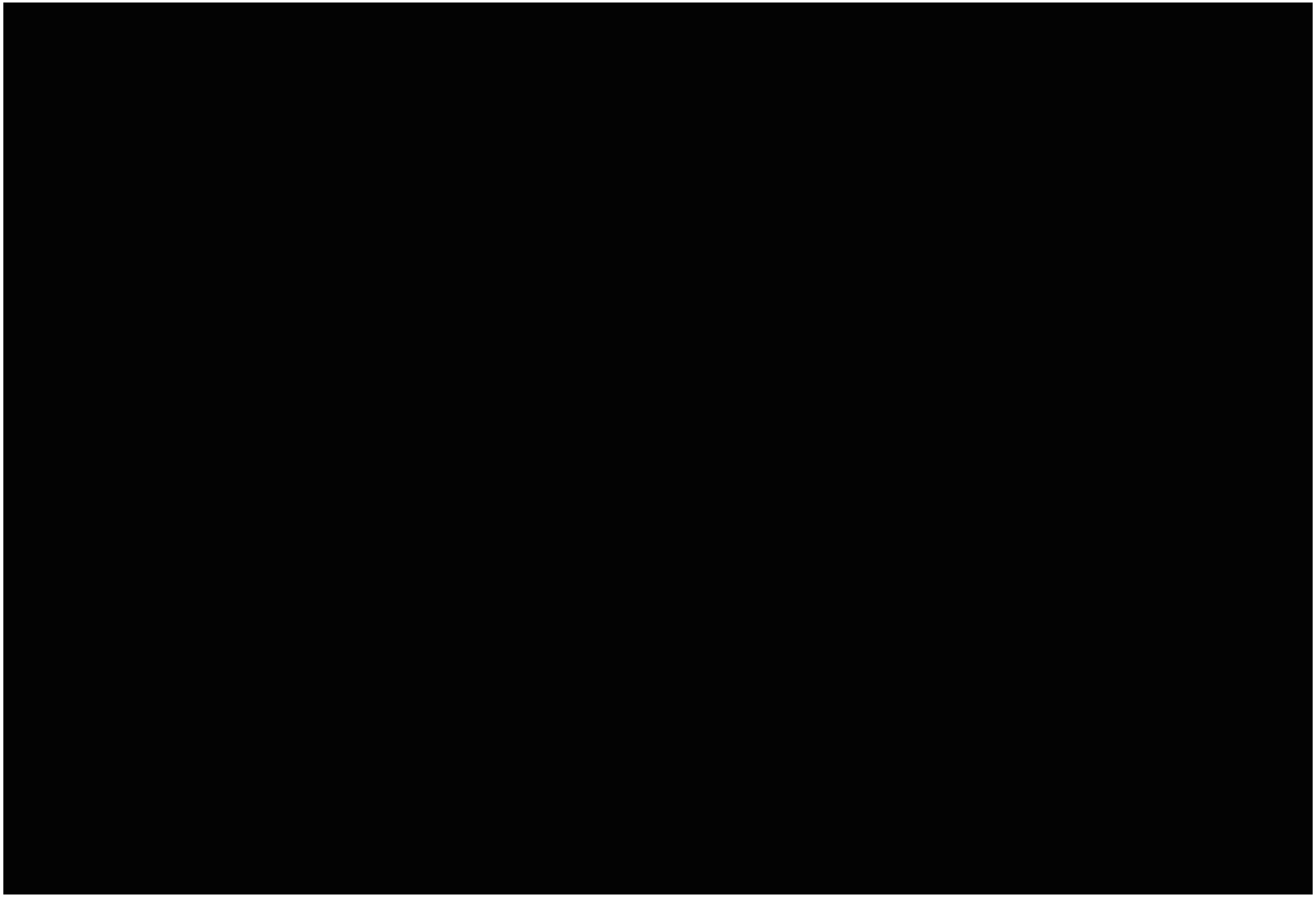
"All major weaknesses have been exposed, analyzed, and replaced with new weaknesses."   Bruce Leverett

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

Back-Up Slides

"Safety Movie"

Glass Failure Movie



Optical Examination Revealed Unusually Flat Fracture Surfaces

- Few fibers protruded from fracture surfaces of failed tank