

# Failure Analysis and Sub-critical Crack Growth (SCG) Characterization of Pt-Al<sub>2</sub>O<sub>3</sub> High Temperature Co-fired (HTCC) Ceramics

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# Presentation Outline

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## Introduction

**Implantable Medical Devices**

**HTCC- What is it, what it does, how it is made**

## Experimental Plans

**Mechanical Testing for Reliability**

**Test Vehicles and Approach**

## Results

**Iterative Materials Processing and Testing**

**- Phase I, II, and III**

**Strength, Stresses and Failure Modes**

## Conclusions

# Implantable Medical Devices



Hydrocephalus  
Sinus Diseases  
Sinus Augmentation  
Sleep Disordered Breathing  
Cervical Degenerative Disc Disease  
Thyroid Conditions

Parkinson's Disease  
Essential Tremor  
Dystonia\*\*  
Obsessive-Compulsive Disorder\*\*



Atrial Fibrillation  
Heart Failure  
Congenital Heart Disease  
Heart Rhythm Disorders  
Angina\*  
Coronary Artery Disease  
Heart Valve Disease

Otologic Disorders  
Meniere's Disease  
Aortic Disease

Scoliosis  
Spinal Fracture  
Lumbar Spinal Stenosis  
Degenerative Disc Disease  
Pelvic Trauma

Severe Spasticity associated with Multiple Sclerosis, Cerebral Palsy, Stroke and Spinal Cord and Brain Injuries

Peripheral Vascular Disease\*

Chronic Pain  
Nausea and Vomiting associated with Gastroparesis\*\*  
Diabetes  
Overactive Bladder and Urinary Retention  
Fecal Incontinence

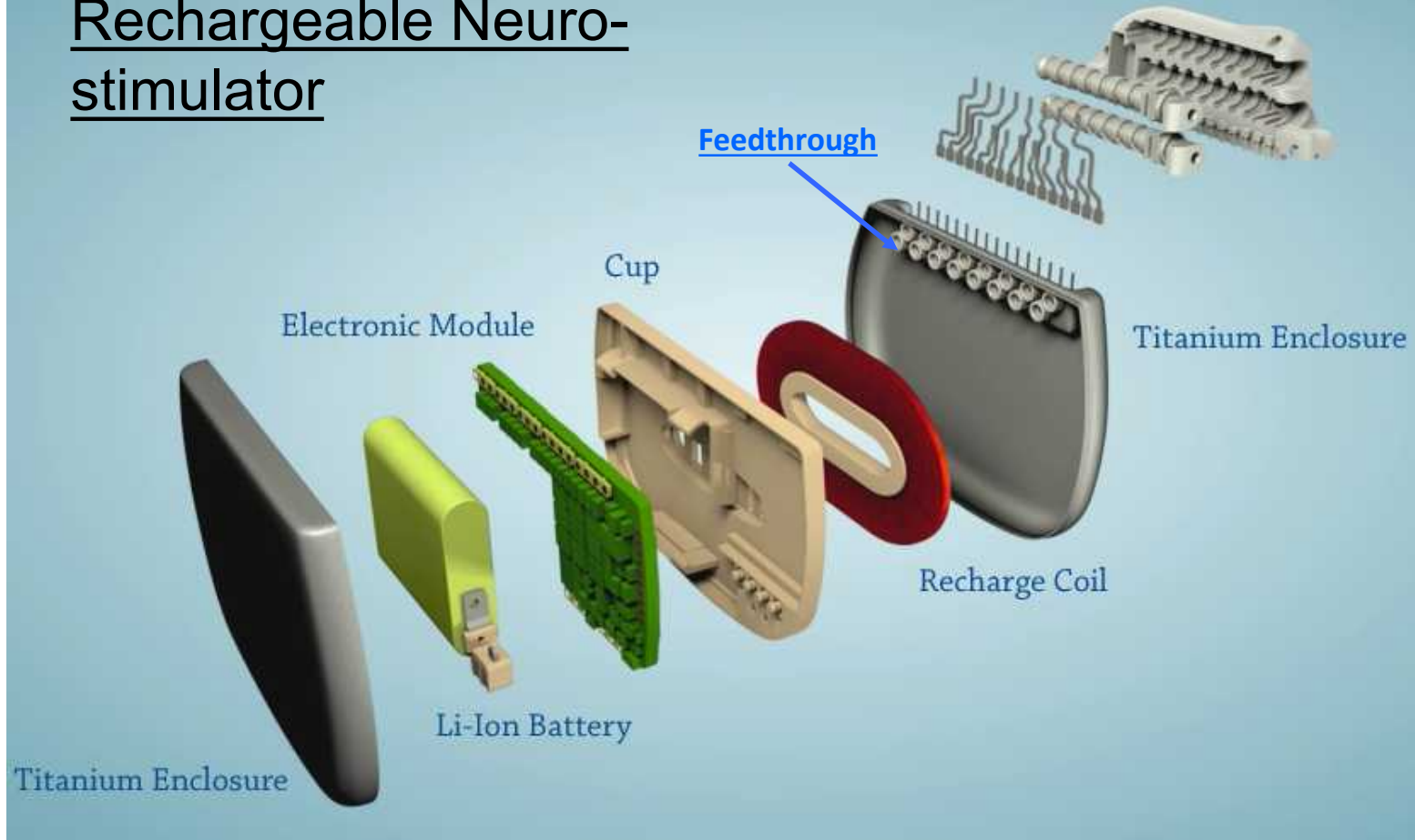
Tibial Fractures

\* Not approved for commercial distribution in the United States  
\*\* Humanitarian Device in the United States – the effectiveness for this use has not been demonstrated

~70 B /per annum

# Implantable Medical Devices

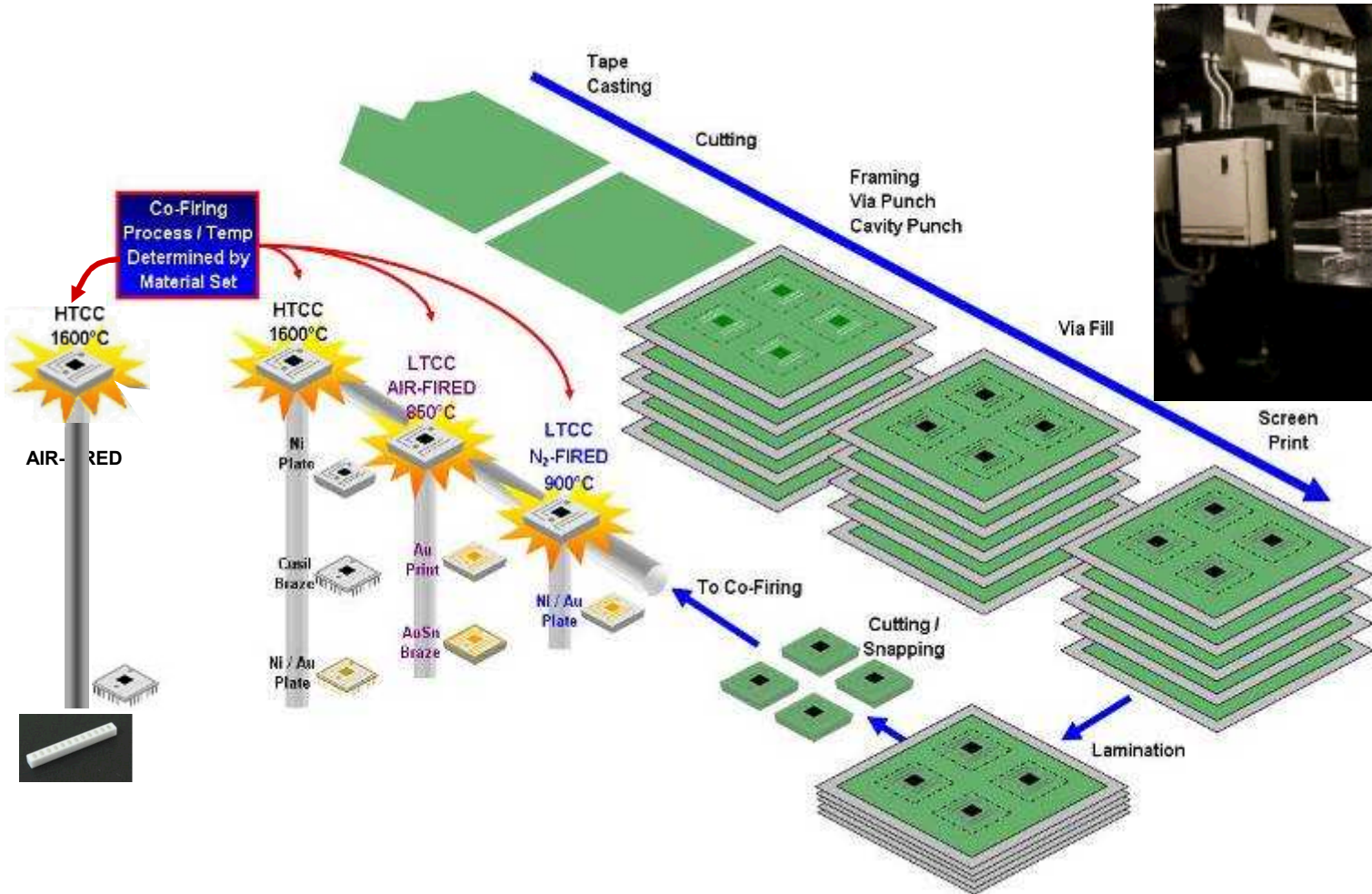
## Rechargeable Neuro- stimulator



# High Temp. Co-fired Ceramics (HTCC)

90-96% Alumina: W, Mo, Pt, Au, Ag via  
High Temperature Sintering

HTCC Belt Furnaces

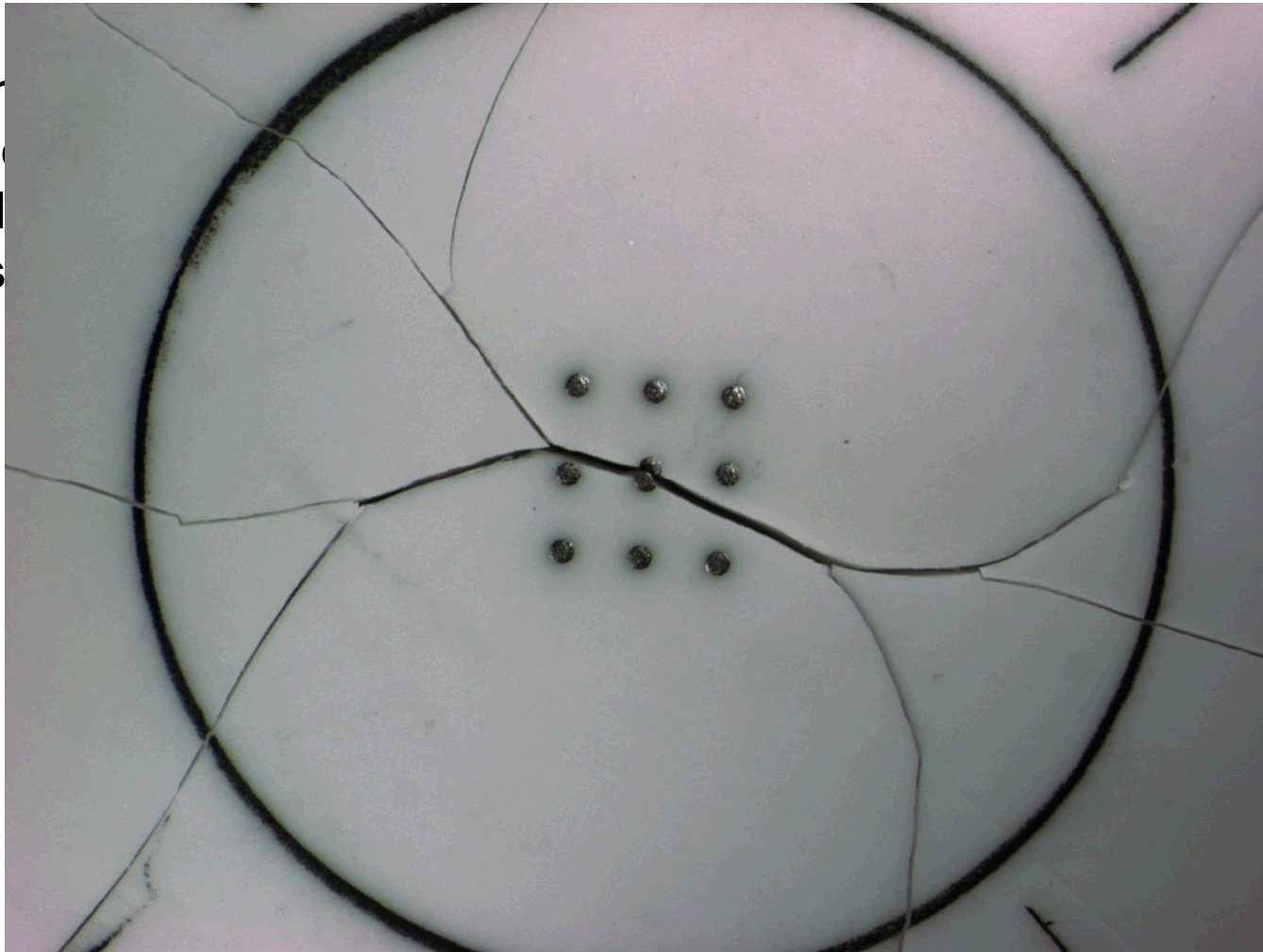


# Hermetic Feedthroughs for Medical Devices

## Alumina - High Temperature Co-fired Ceramic (HTCC)

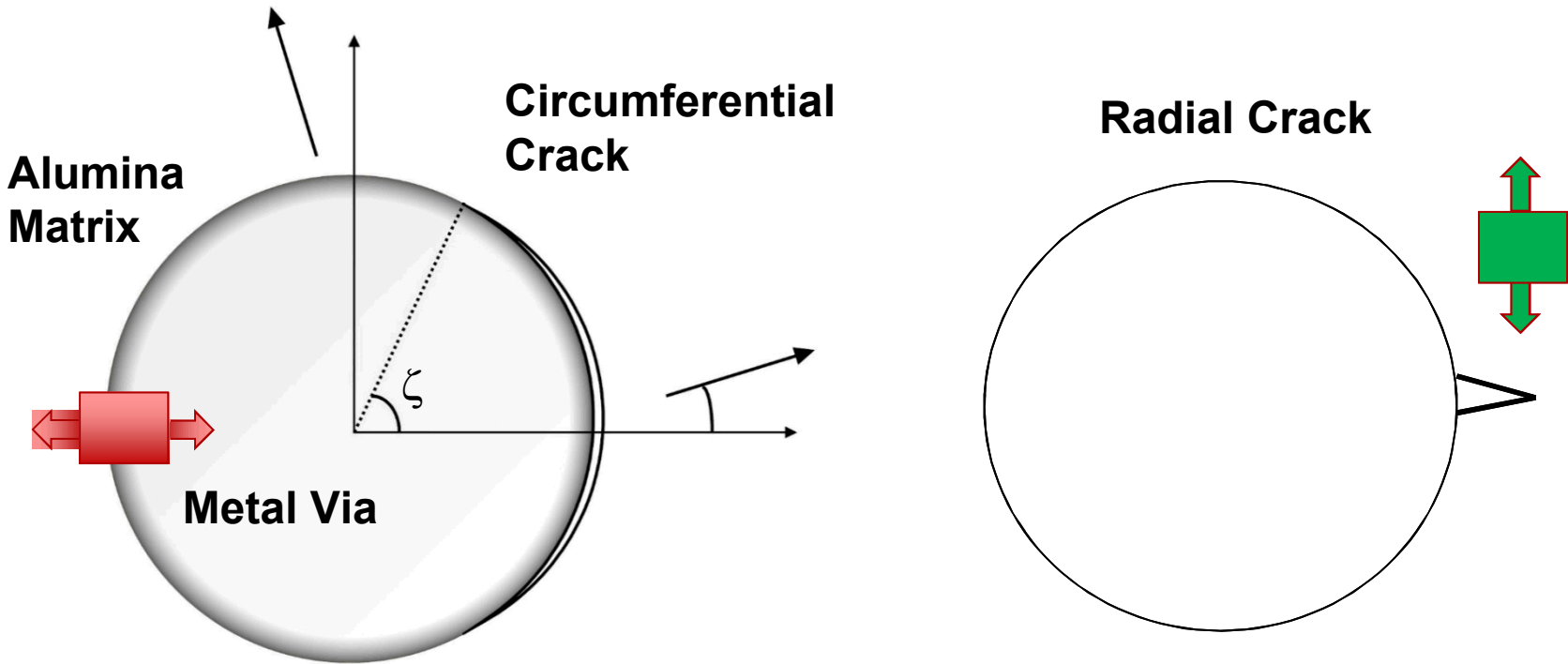
Physical and electrical characteristics for high-reliability applications:

- Mech
- High
- Easil
- Pass
- 



Antennas

# Stress State Around a Via Impacts Strength Sandia National Laboratories



Local residual stress – Due to thermal and elastic mismatches, modified by plasticity of via material

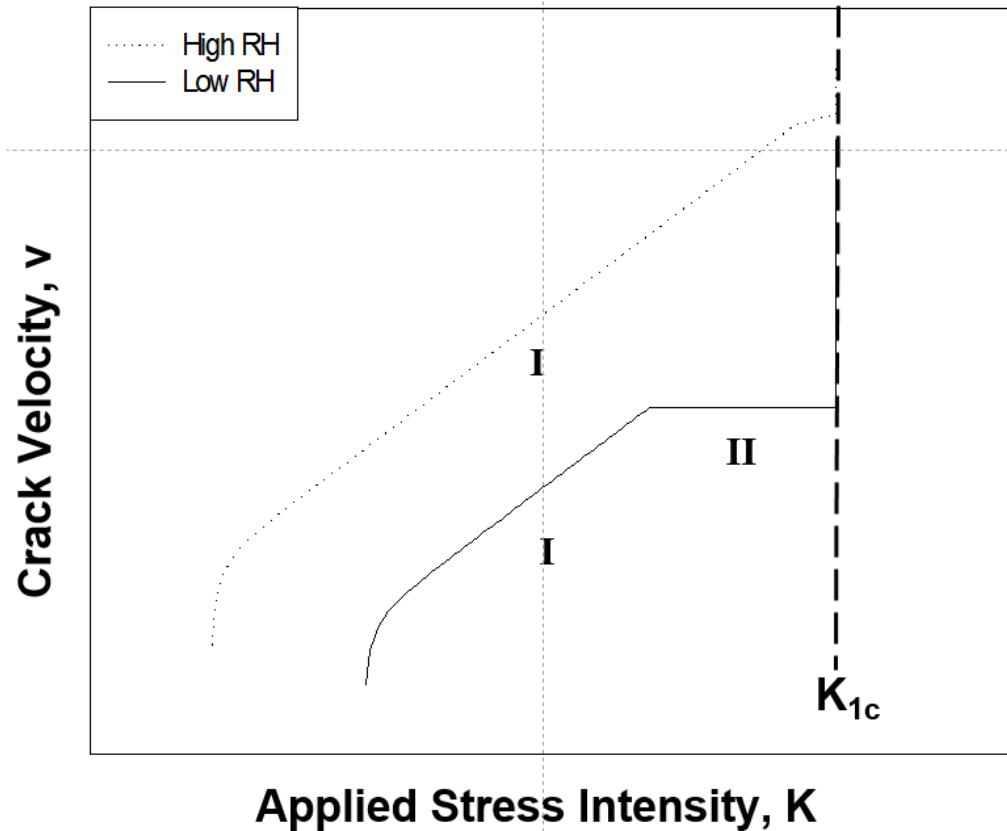
**Higher thermal expansion via**

**~Similar expansion via + plasticity**

Externally applied stresses – Thermal transients, & due to brazing/welding of material

Combination of these stresses can lead to Sub-Critical Crack Growth (SCG): can cause leaks, crack growth and failure

# Sub-Critical Crack Growth (SCG)



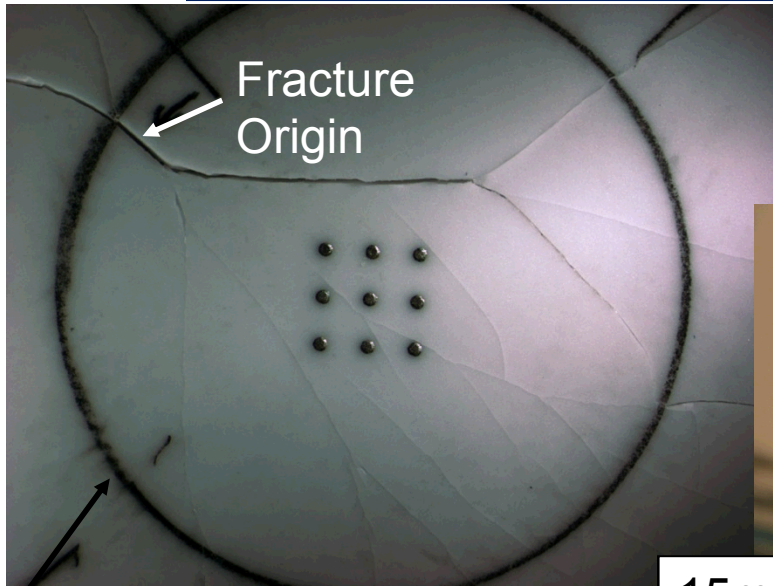
$$v = A \left( \frac{K_I}{K_{Ic}} \right)^n$$

SCG behavior was quantified by conducting strength tests as a function of loading rates (*ASTM C1368: Test method for SCG Parameters*)

Materials with and without vias were strength tested in 20% RH, and in 0.9% saline (simulated body fluid)\*

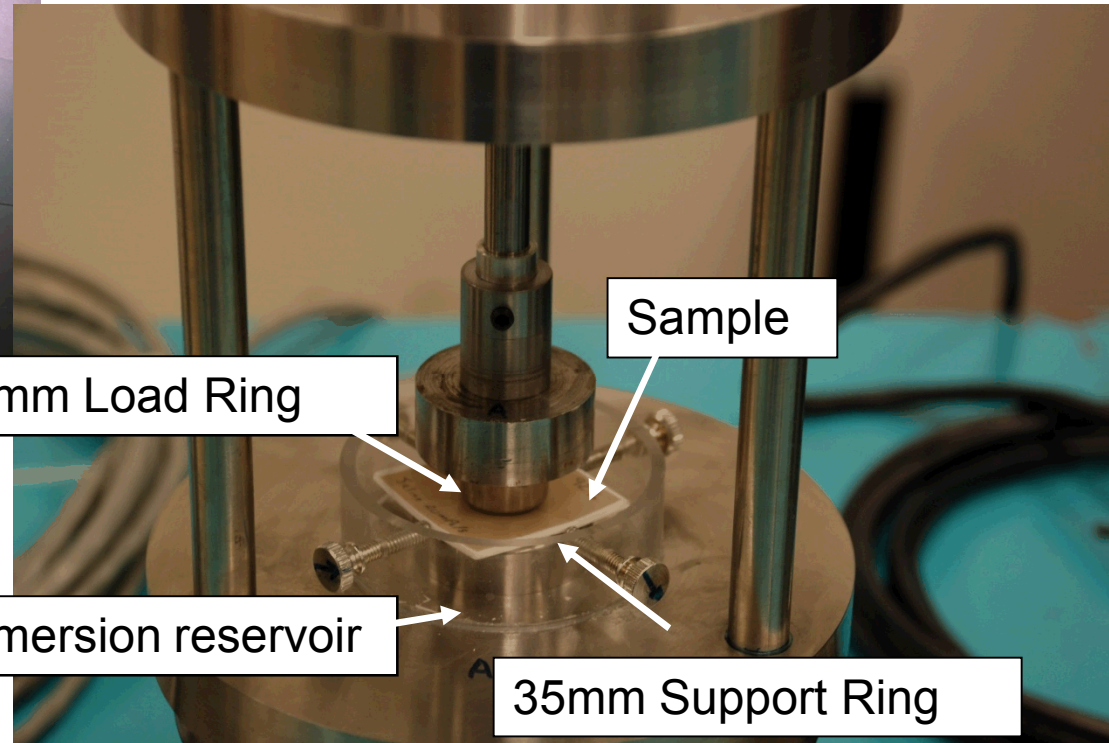
\*<https://www.ncbi.nlm.nih.gov/pmc/articles/PMC3683025/>

# Reliability of Devices



*(ASTM C1368 strength testing)*

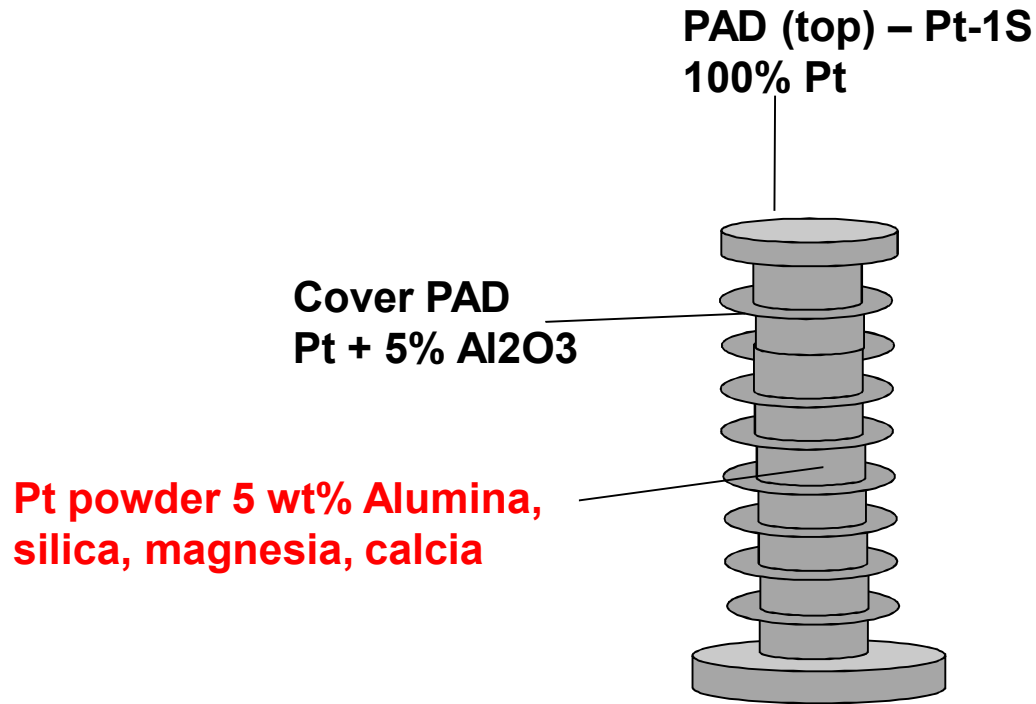
Tensile region  
(inside black circle)



**Sample Size: 40mm<sup>2</sup> x 2mm**

Loading rings made of Tefzel material

# Phase I: Via Materials Composition

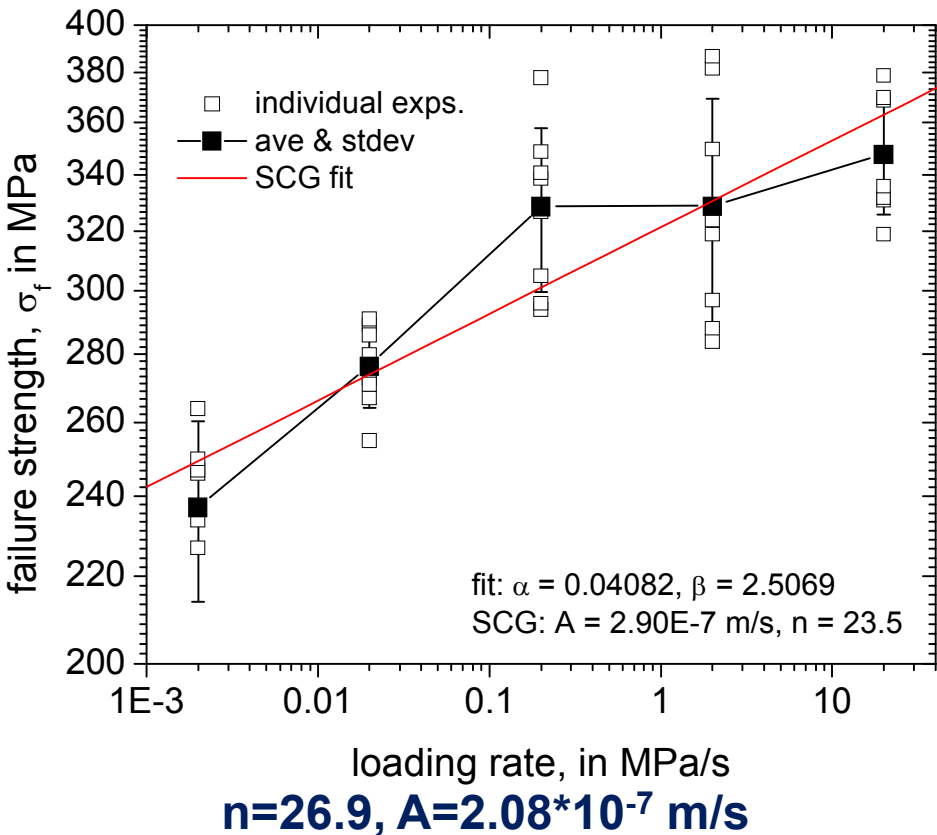


# Phase I – 20% RH Air

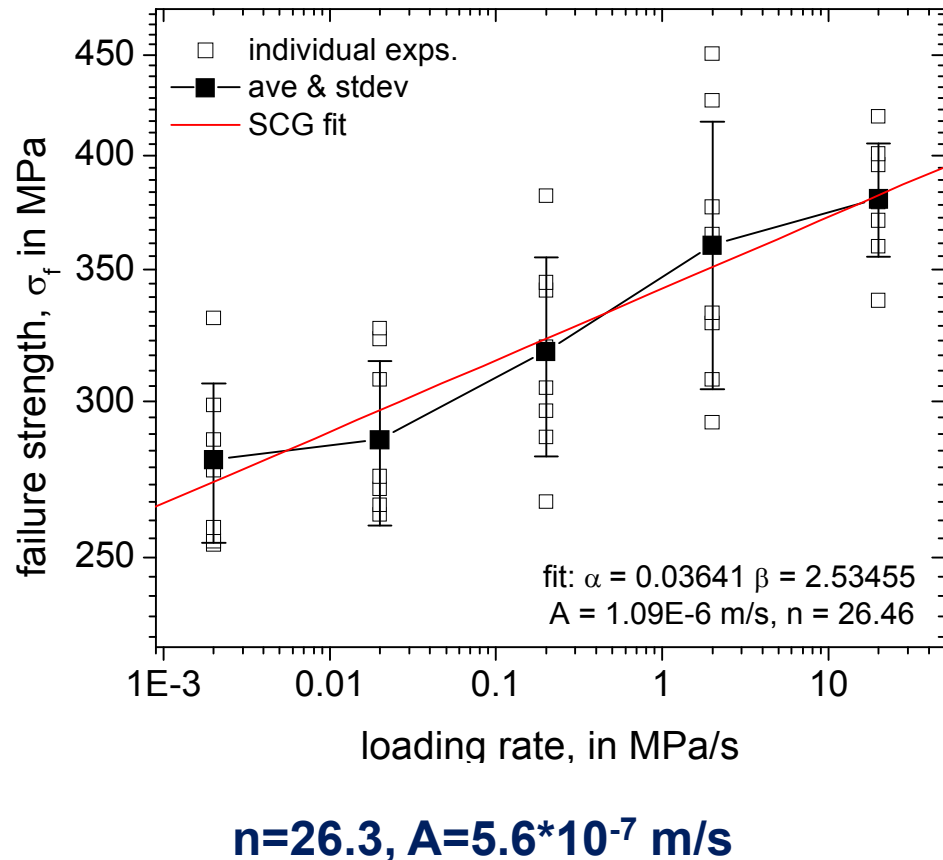
$$v = A \left( \frac{K_I}{K_{Ic}} \right)^n$$



## No Vias- Base 92% Alumina



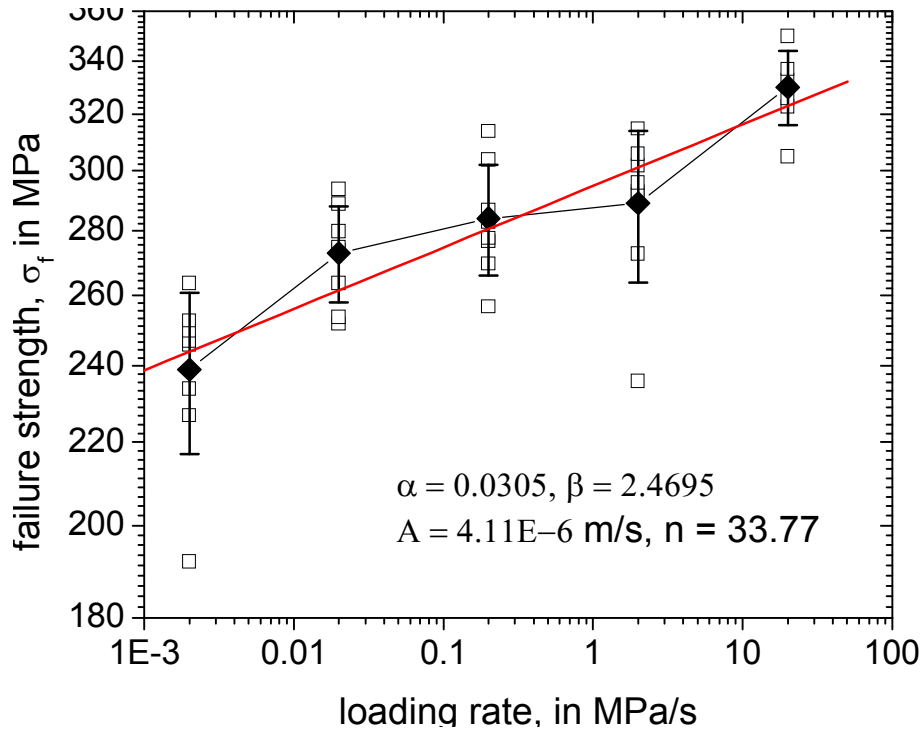
## Alumina with Vias



**$n$  values ~ those of alumina in literature**  
**Strength Response in air is not affected by vias**

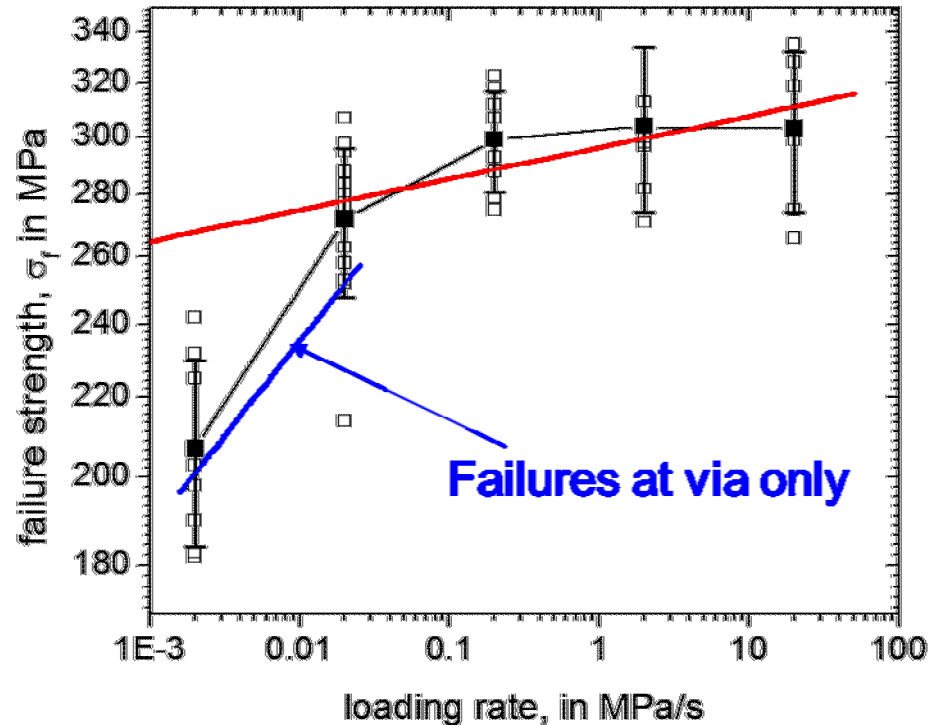
# Phase I- Saline Immersion (*in vivo* simulation)

## No Vias- Base 92% Alumina



$n=36.7, A=4.54 \cdot 10^{-6} \text{ m/s}$

## Alumina with Vias



$n=38.1, A=5.07 \cdot 10^{-8} \text{ m/s}$

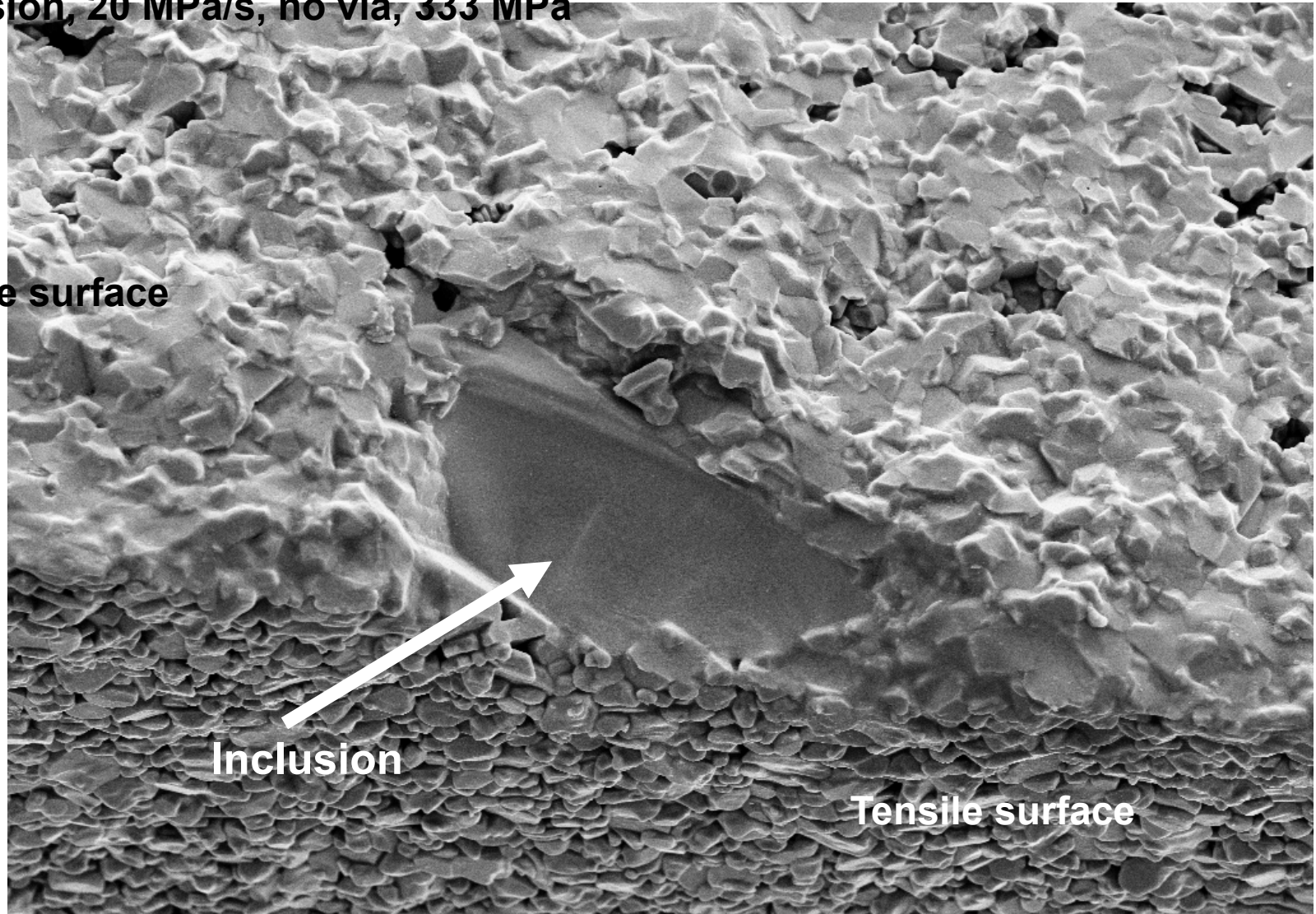
**At Via**  $n=9.2, A=1.0 \cdot 10^{-8} \text{ m/s}$

**Vulnerability: Slow rates and saline environment**

# Fractography- Hard Inclusion

Immersion, 20 MPa/s, no via, 333 MPa

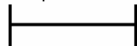
Fracture surface



Inclusion

Tensile surface

10  $\mu$ m



EHT = 10.00 kV

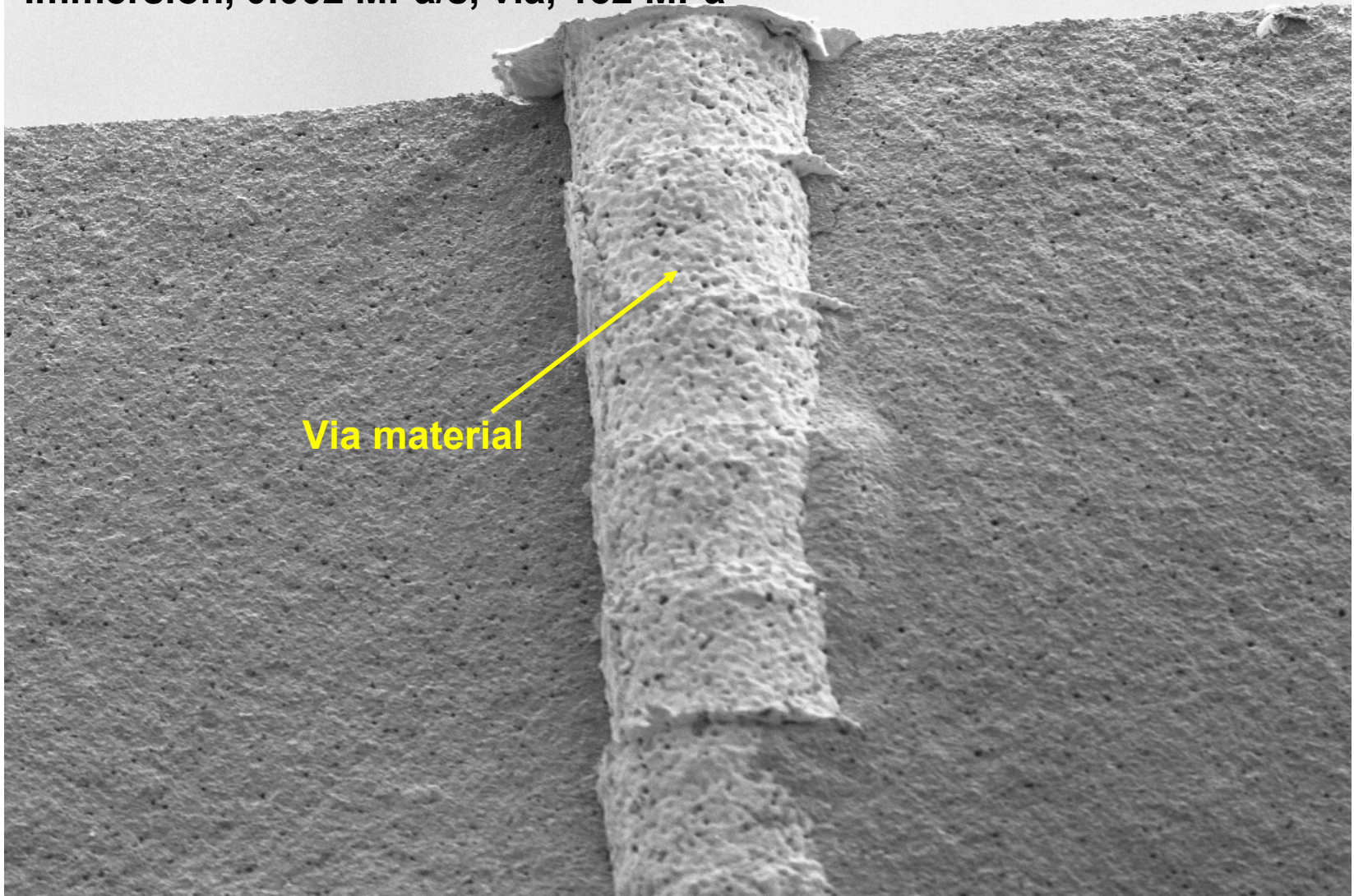
WD = 12 mm

Signal A = SE2

File Name = 04\_6\_03.tif

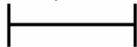
# Fractography – Via Separation - A

Immersion, 0.002 MPa/s, via, 182 MPa



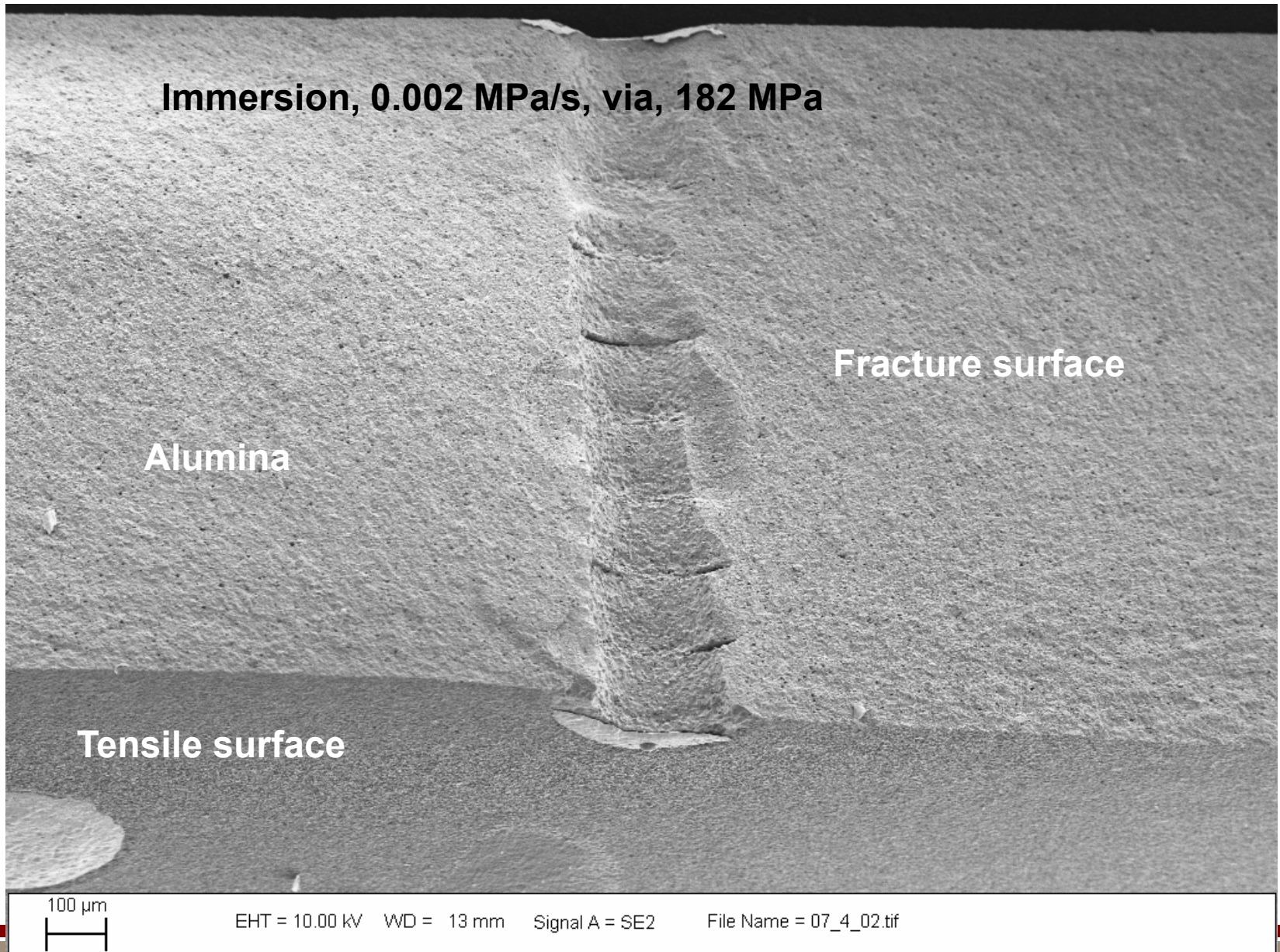
Via material

100  $\mu$ m



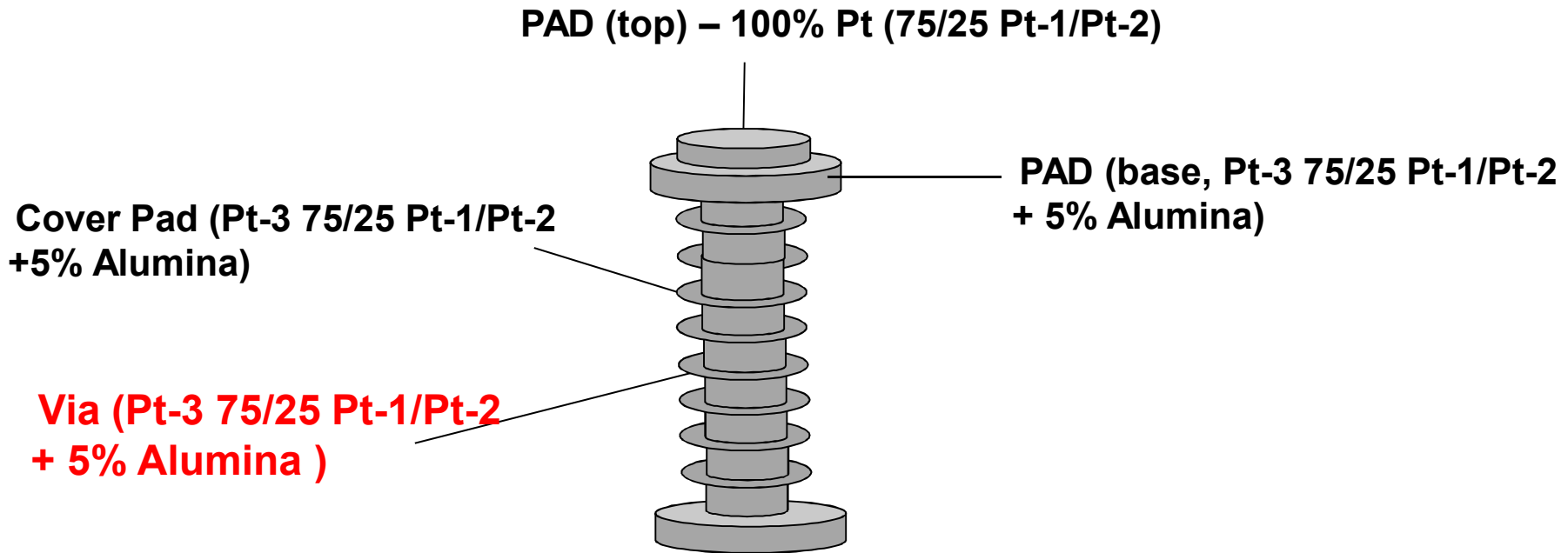
EHT = 10.00 kV WD = 13 mm Signal A = SE2 File Name = 07\_4\_05.tif

# Fractography – Via Separation - B



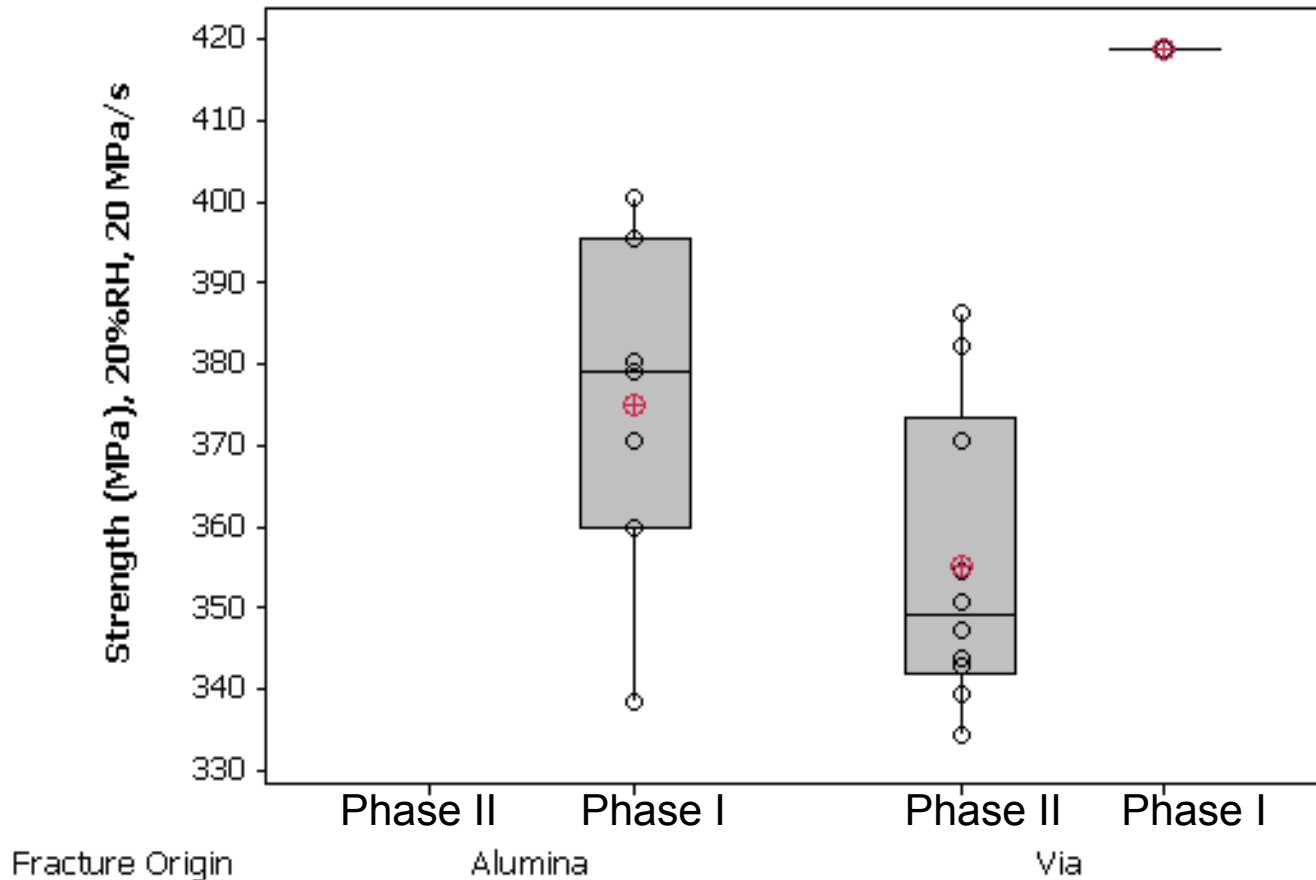
# Phase II

Significant modification to  
process to improve via interfaces



# Phase II – 20% RH, Alumina with Via

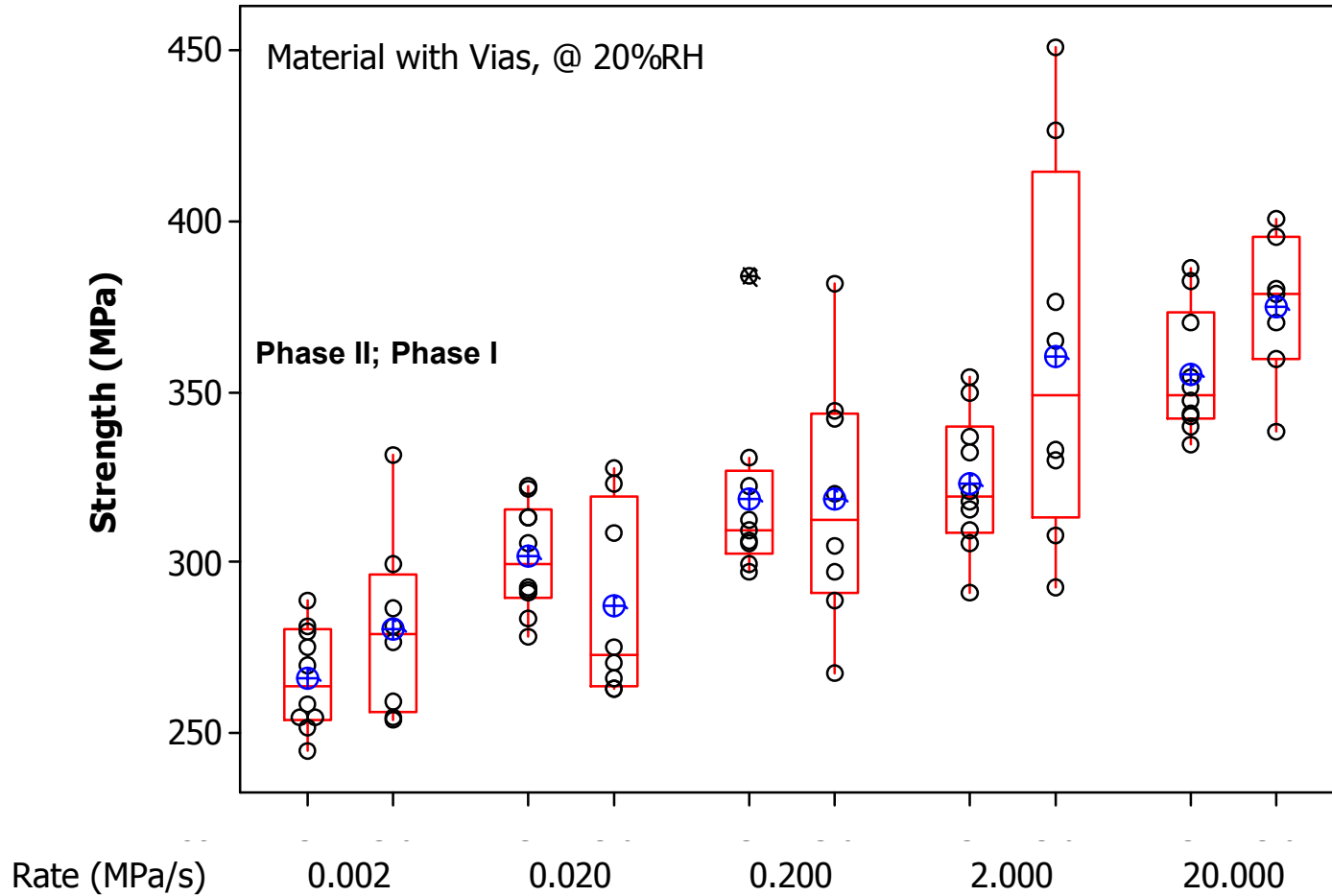
Fast testing rates (20 MPa/s), and 20% RH



The new batch of samples have lower mean strength.

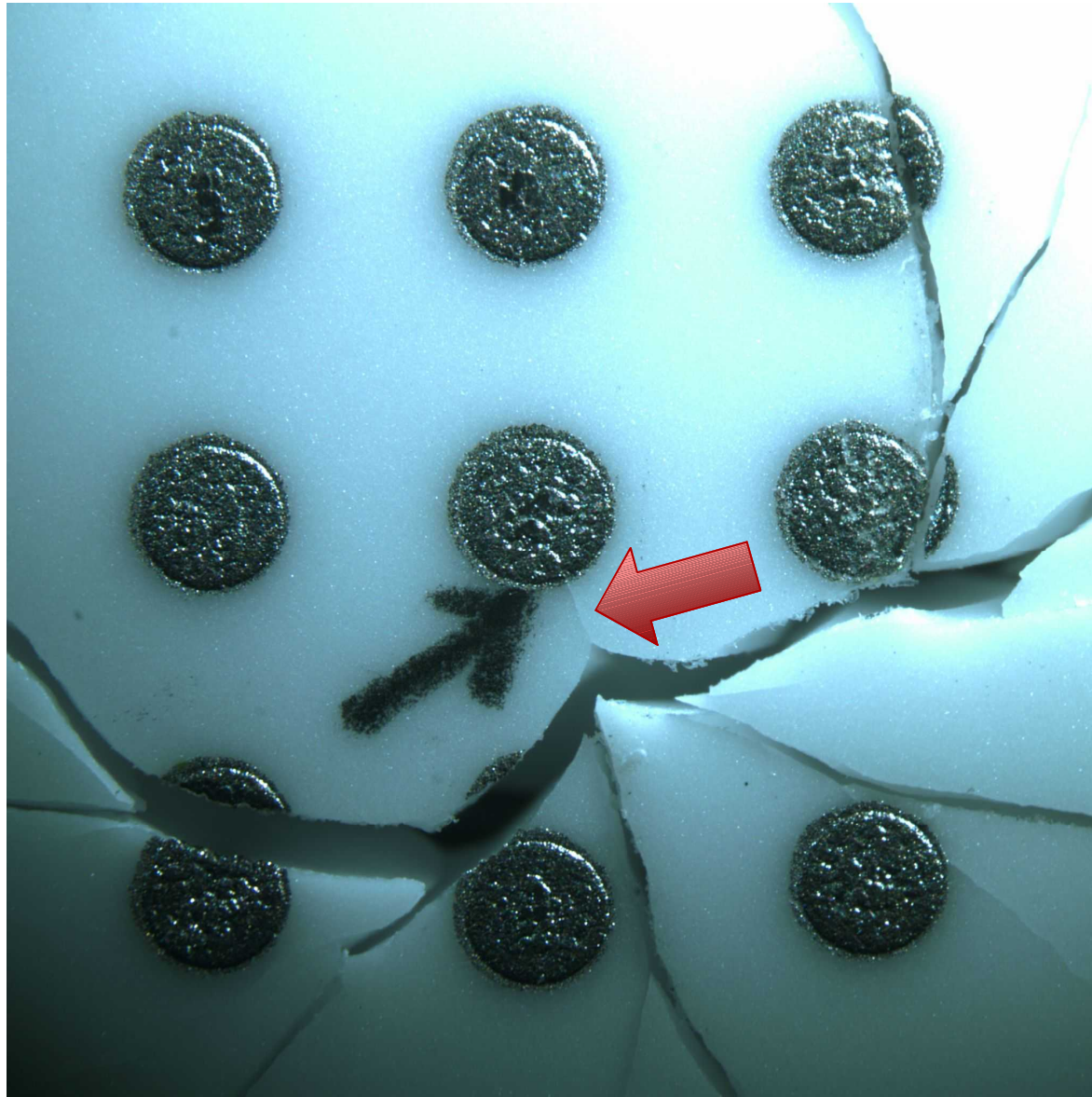
**The failure origin has shifted: All failures are from the via**

# Phase II – Strength Lower

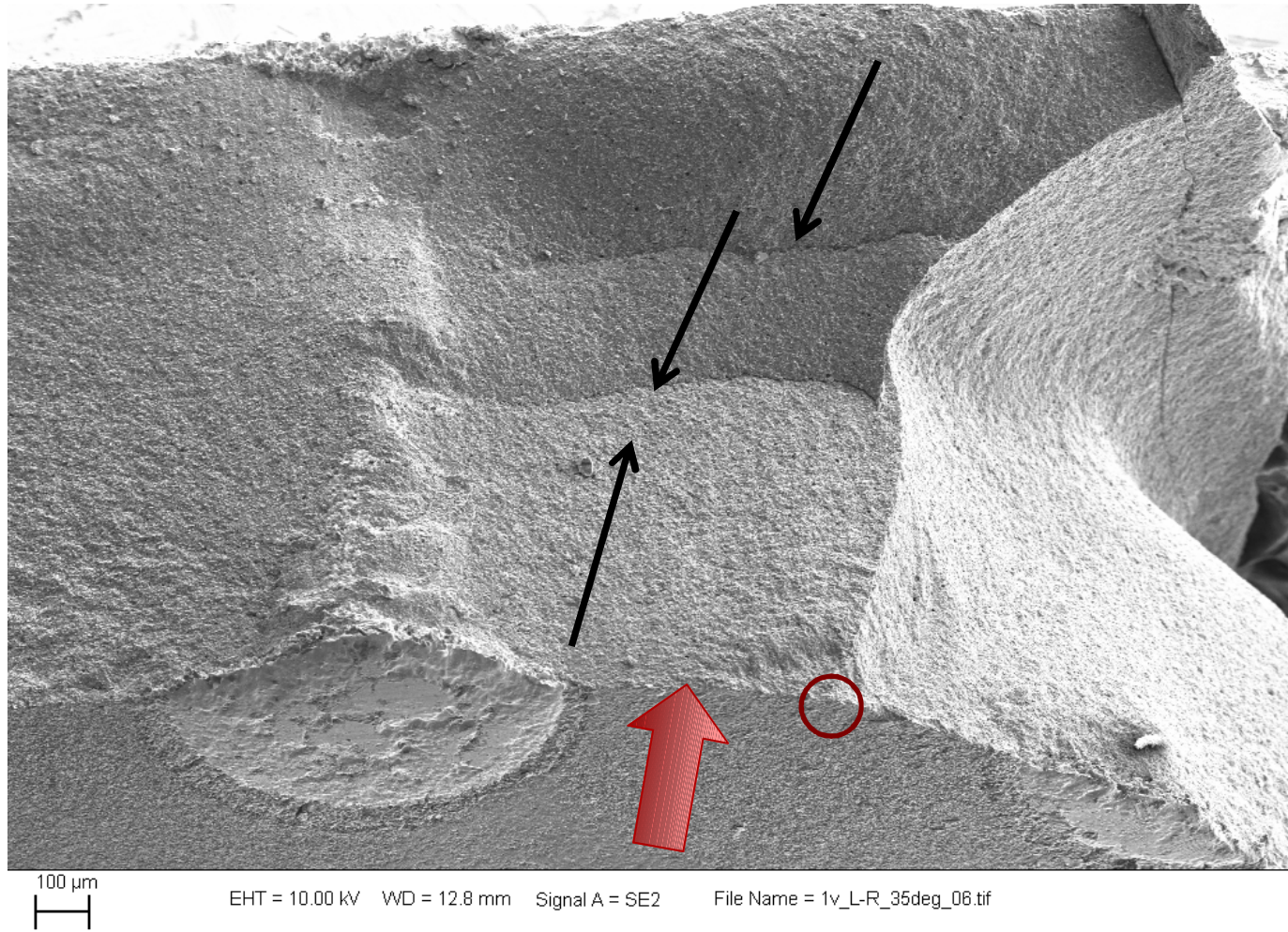


# Phase II – Fractography (radial cracks)

Via 20 MPa/s  
20% RH



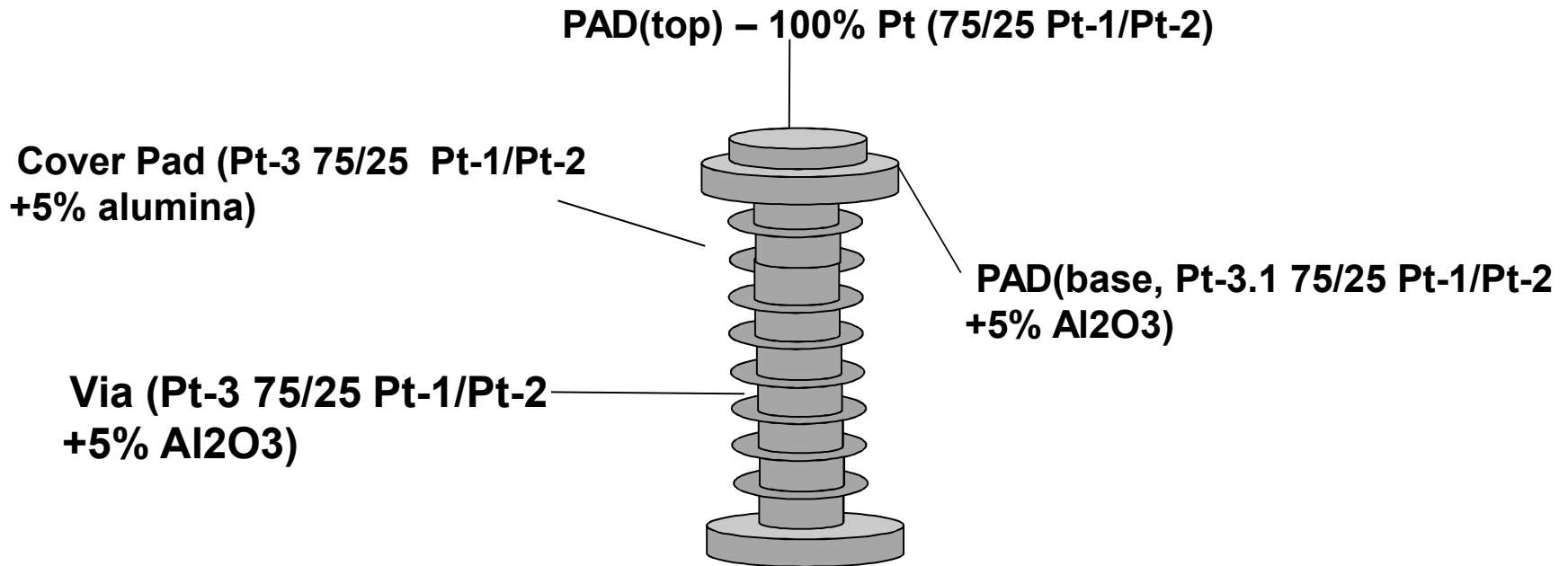
# Phase II – Fractography (radial cracks)



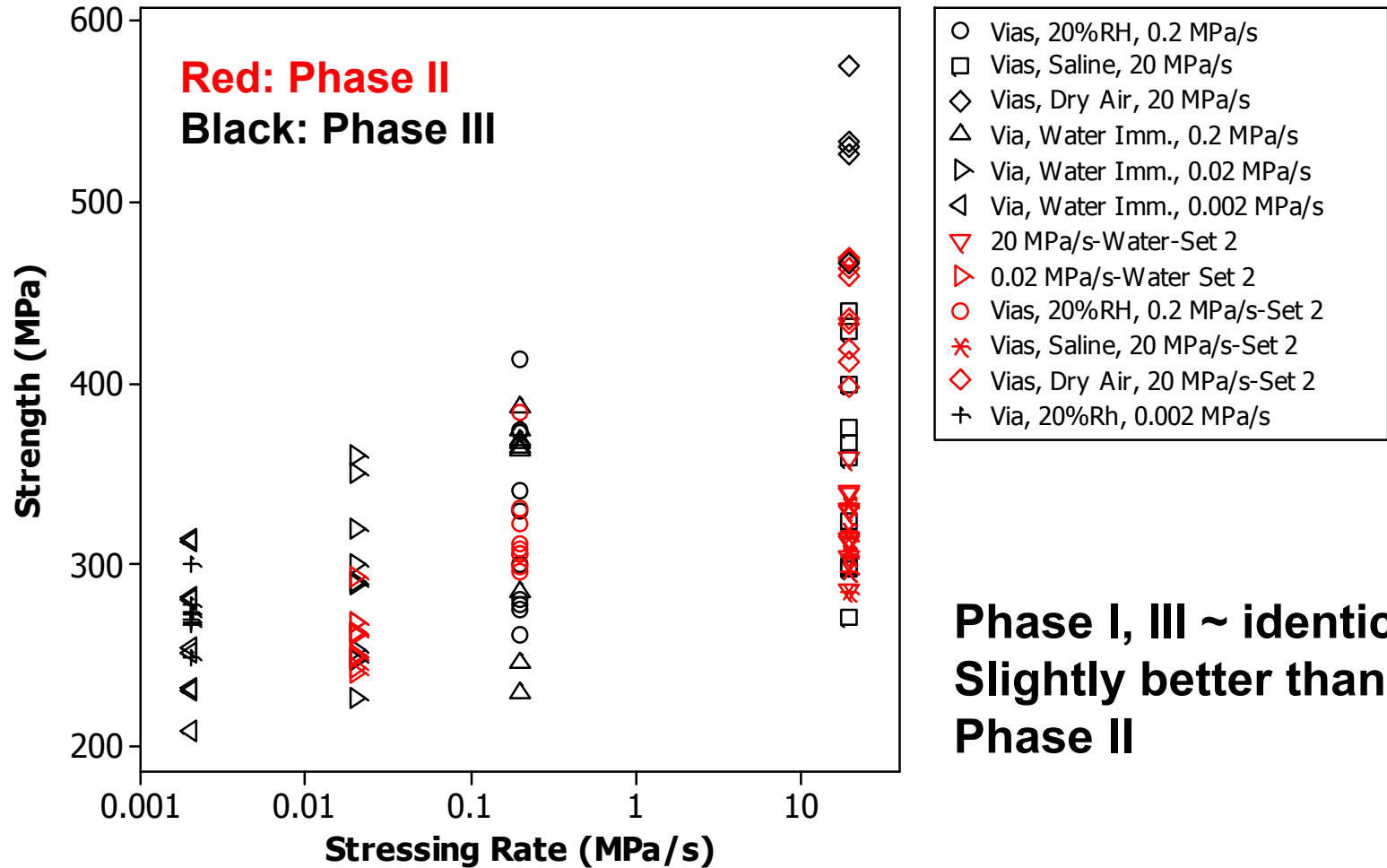
**These samples must have a small circumferential tensile stress near the vias: Radial cracks and slightly lower strength Lamination ?**

# Phase III

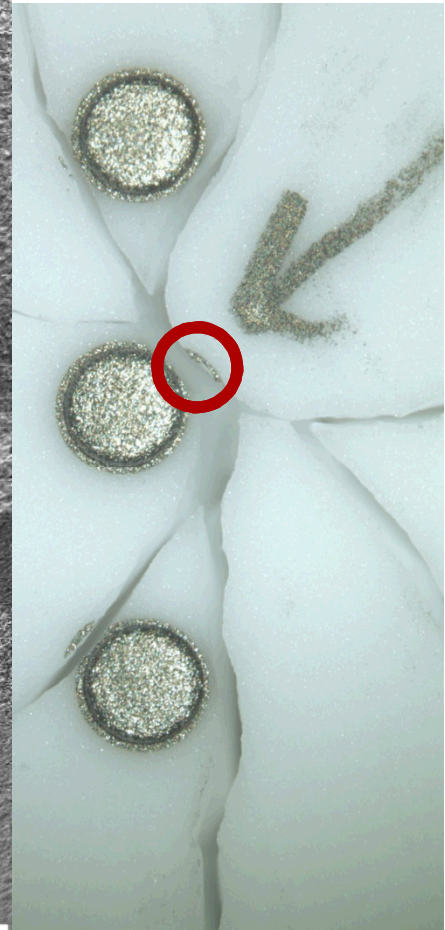
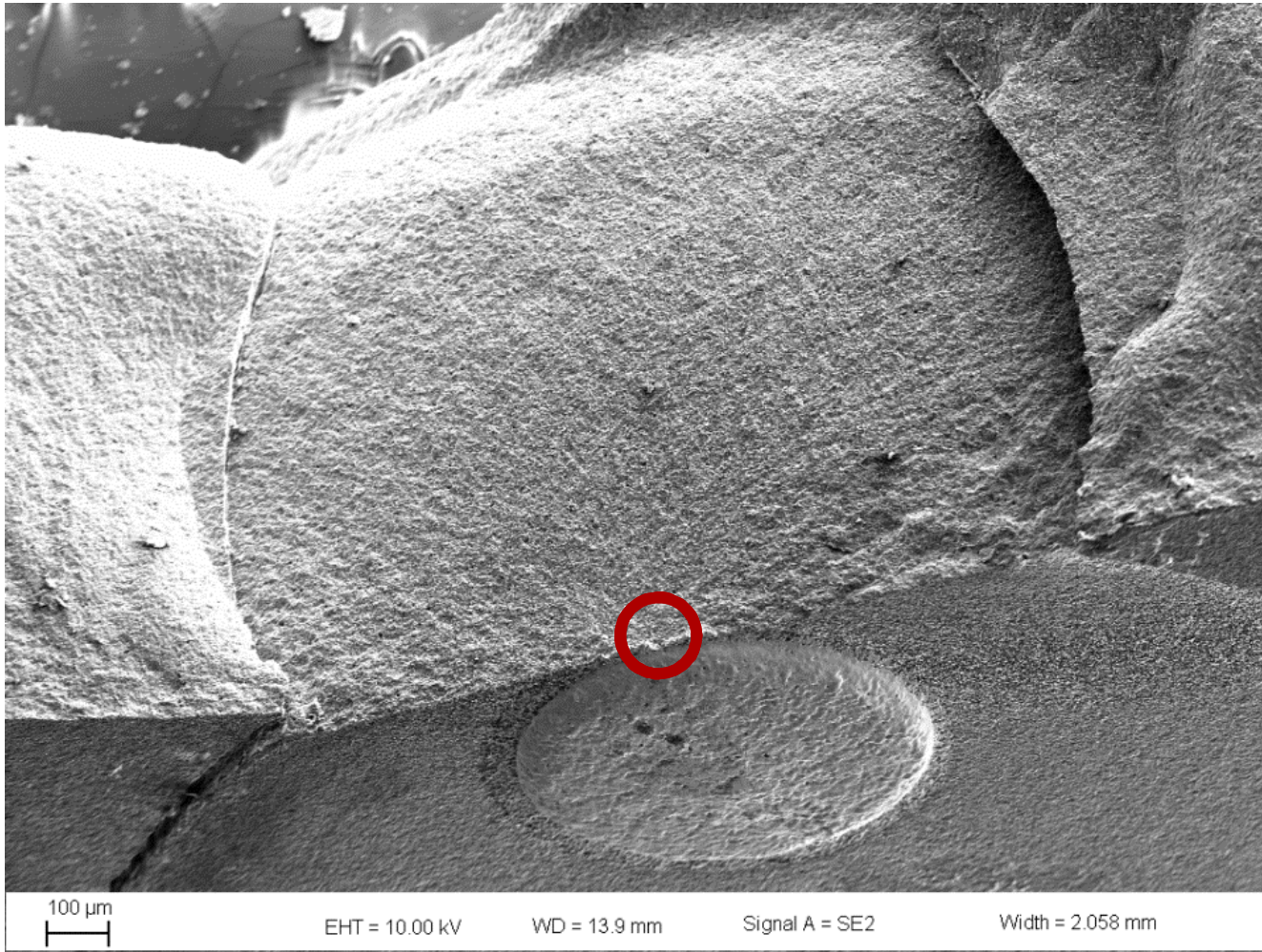
Modification of Pt ratios in pastes  
Modified paste solvent vehicle  
Changed lamination procedure



# Phase III – Strength ~ base alumina



# Phase III - Fractography



# Conclusions

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## Strength:

Materials in Phase I, III had ~ identical strength; Phase II slightly lower

### **Notable exception of saline immersion, Phase I**

- **Vulnerability must be considered, tested for, and designed out prior to use**

## **Stress State around the via (plasticity and thermal expansion considerations):**

Phase I has a slight radial tension, and failure along interface in depth

- Removal of higher expansion glass phase led to redress of interfacial failure mode

Phase II has a slight circumferential tension, and radial cracking

Phase III has ~ 0 radial tensile stress

## Lifetime:

Parameters extracted from these tests, and stresses use geometry, were used to calculate lifetimes > 20 years

## Overall:

**We can obtain material with vias that have similar lifetimes as base alumina material.**

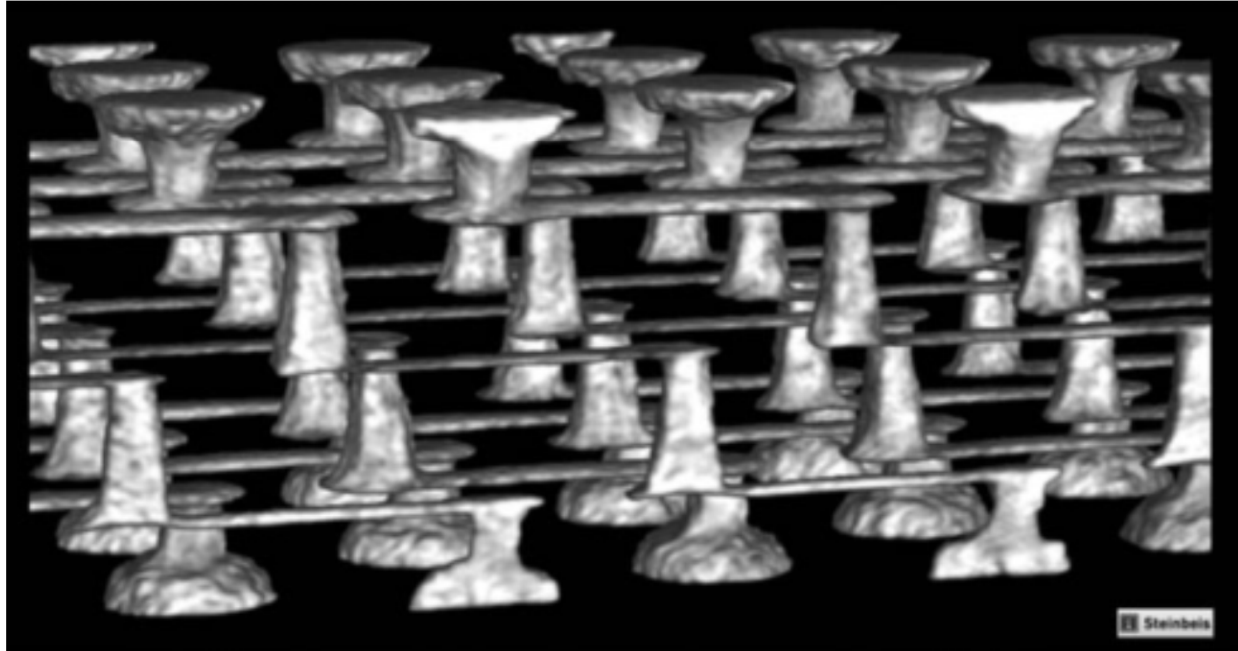
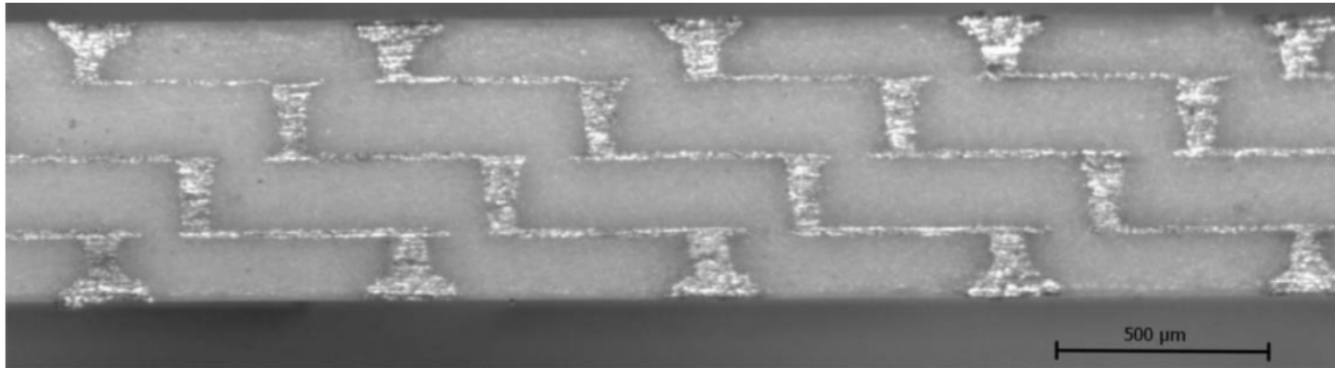
## Title: Failure analysis and sub-critical crack growth (SCG) characterization of Pt-Al<sub>2</sub>O<sub>3</sub> high temperature co-fired (HTCC) ceramics

Abstract: Miniaturization and integration of multiple functions into one component is desired for device downsizing. HTCC alumina, with its high strength and bio-compatibility, provides an option for integration and higher reliability medical devices. A 92% alumina-platinum via HTCC material was characterized for failure modes and sub-critical crack growth, and modifications to processing were implemented to obtain a material with strength and SCG characteristics similar to bulk alumina. In phase I development, this material had strength equivalent to bulk alumina, except at low stressing rates in saline, body fluid-like environments, where a novel failure mode (circumferential cracking) was encountered. Processing changes implemented for Phase II materials led to failures emanating as radial cracks from the vias with slight loss of strength. Changes made to Phase III materials led to strength and SCG characteristics that were undistinguishable from the base materials. Fractographic evidence indicates that cracking originates tangentially to the via in Phase III material

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# Via Geometry, Layouts



**Guenther et al., Journal of  
Biomedical Materials Research, 2013**

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