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Sandia Stressed Glass Technology Overview

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Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company,
for the United States Department of Energy's National Nuclear Security Administration
under contract DE-AC04-94AL85000.

NNSA National Nuclear Security Administration

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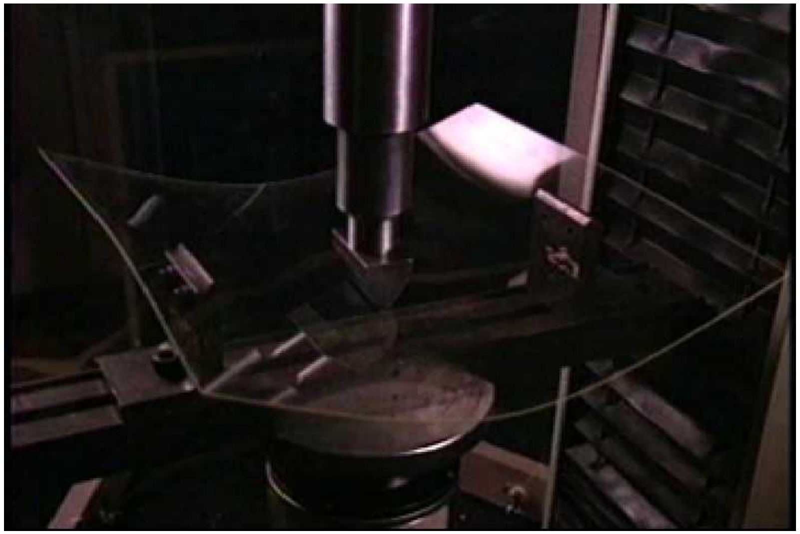
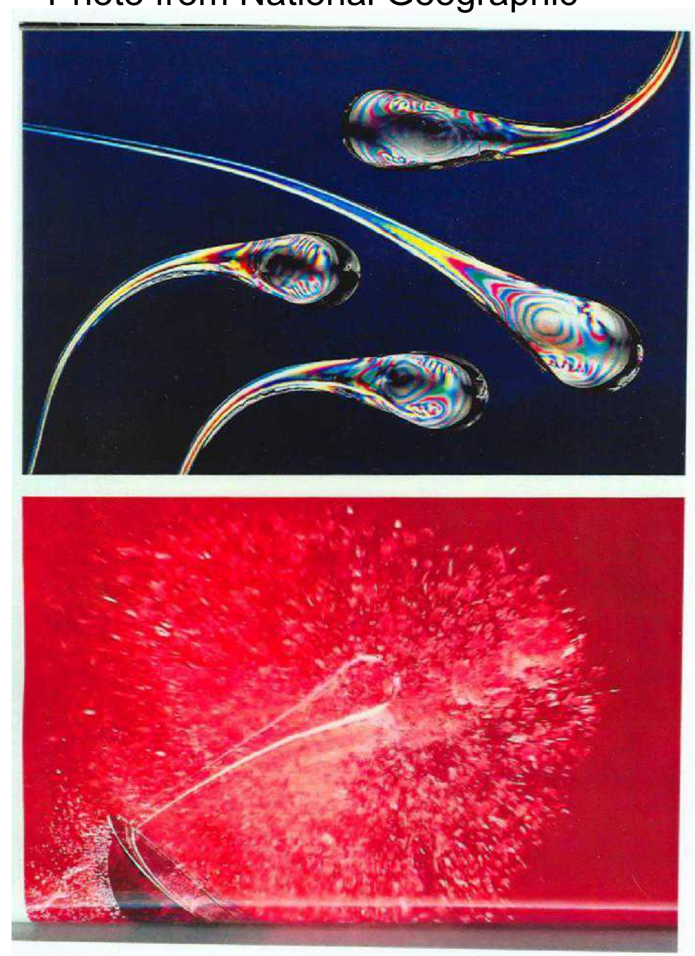
Deliberately Stressed Glass - Not Your Usual Weak Brittle Material!



Prince Rupert's Drops - Circa 1660
Photo from National Geographic



Outline

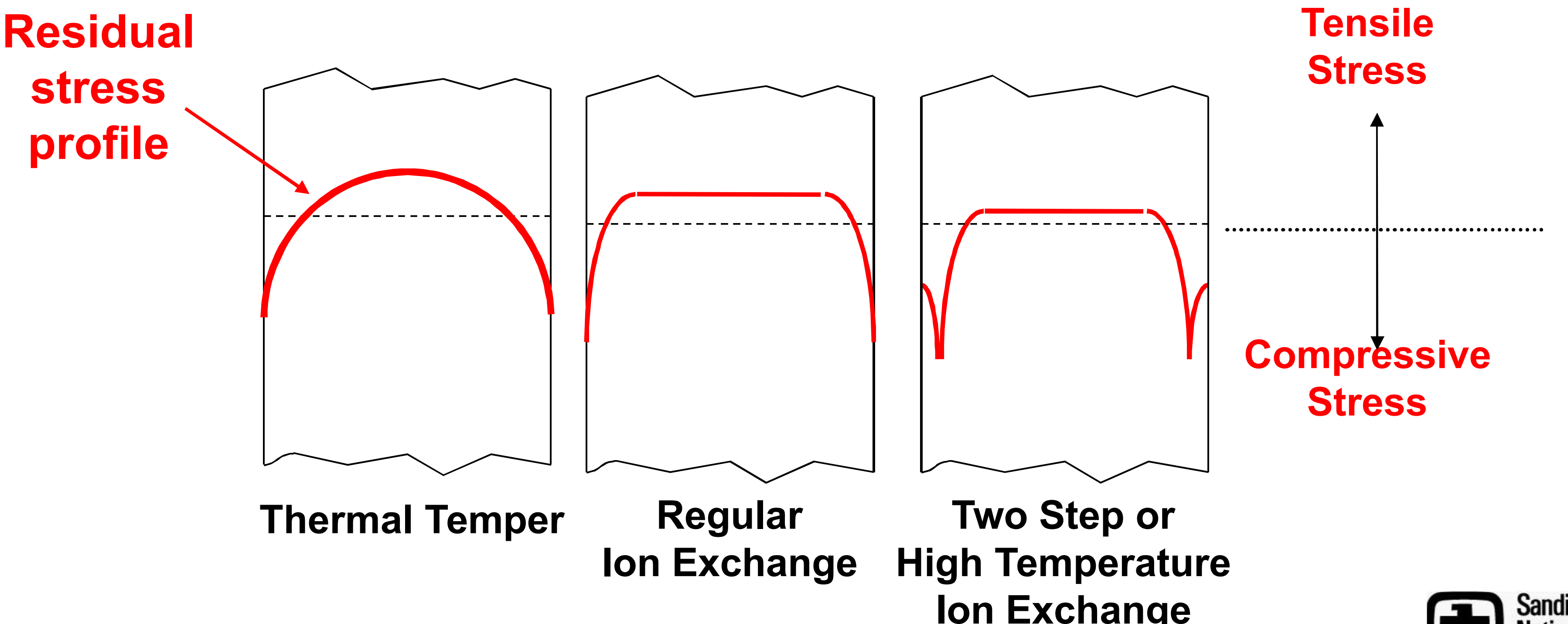
- **Introduction to Stressed Glass**
  - Glass processing
  - Mechanical properties and fragmentation behavior
  - Energy storage capability
- **Applications**
  - Sensors and materials for nuclear weapons weaklinks
  - Containers and barriers
- **Enhancement of Fragmentation Initiation**
  - Holes and masked regions
  - Protrusions
  - Directed laser energy
  - Load/unload indentation tests
- **Bonding techniques to enable fabrication of complex shapes**

INTRODUCTION

- Fabrication of stressed glass
- Mechanical properties and fragmentation behavior
- Energy storage capability

Stressed Glass is Made using Two Methods

- **Thermal Tempering (moderate strengthening)**
  - rapid cooling of hot glass produces residual stress profile
  - auto and architectural applications
- **Ion Exchange (extremely high strengths)**
  - stress profile produced by replacing small ions in surface with large ones at elevated temperature using a molten salt bath
  - personnel safety and optical applications



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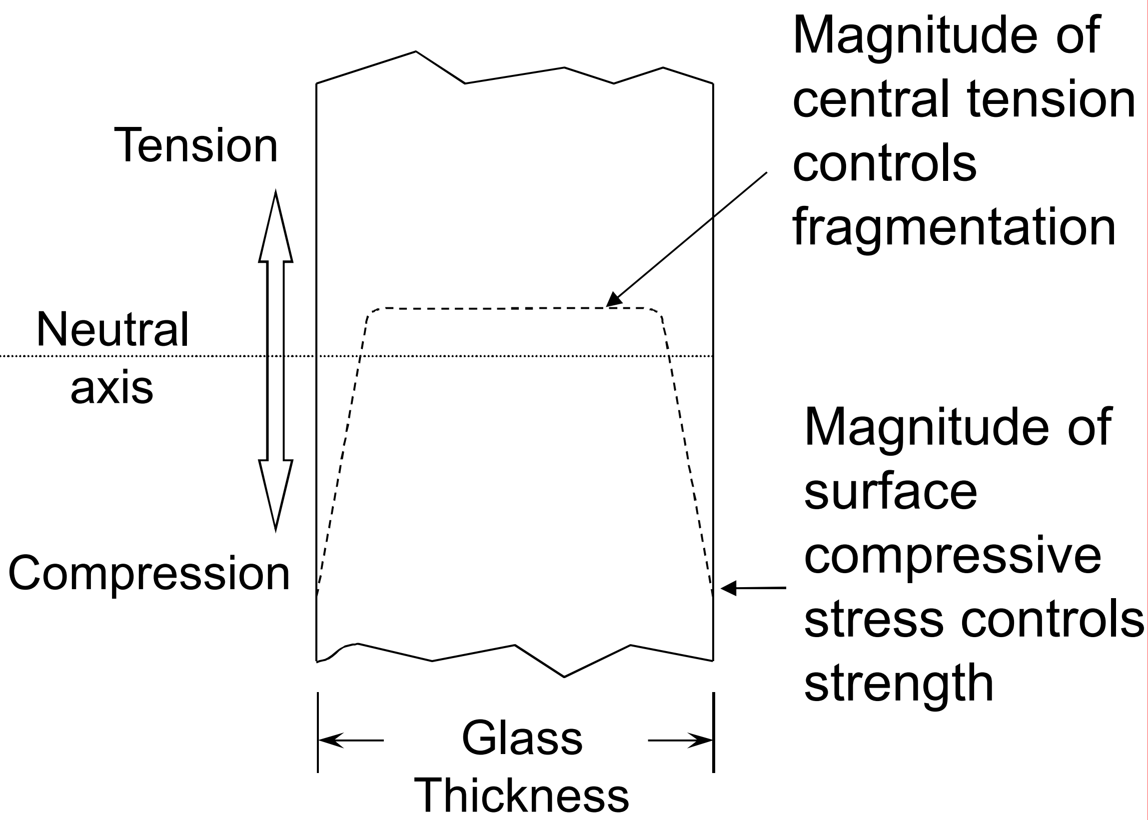
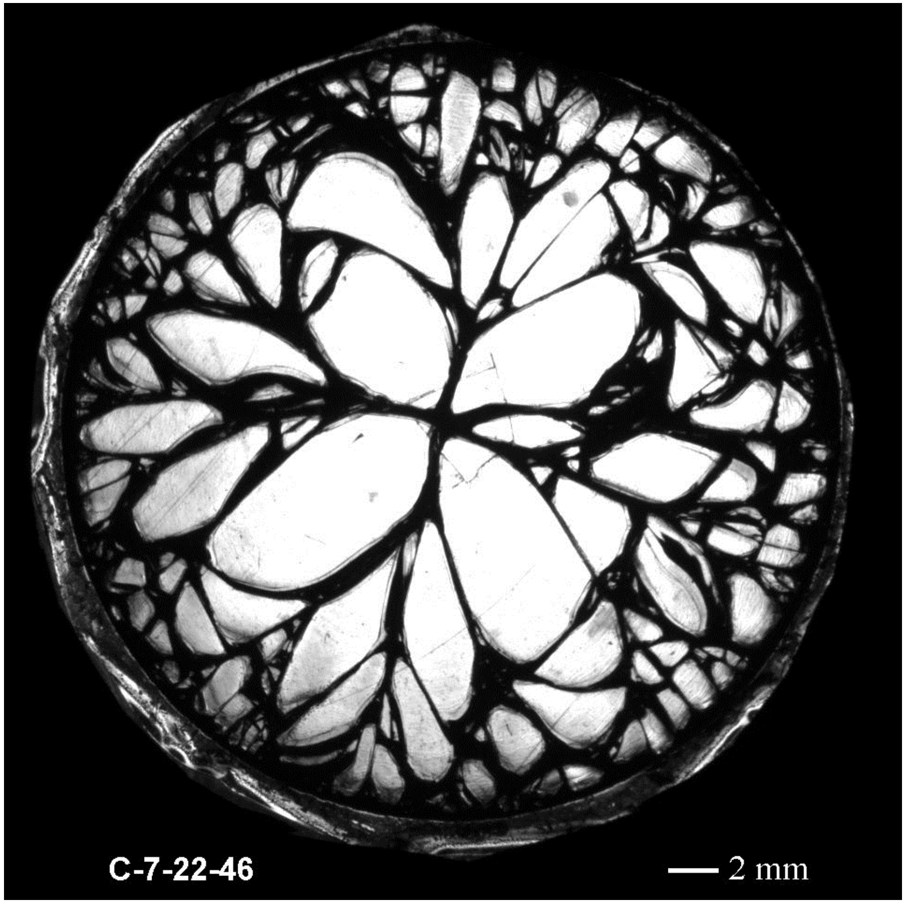
Ion-Exchanged Glass Has Remarkable Properties Because of its Residual Stress Profile



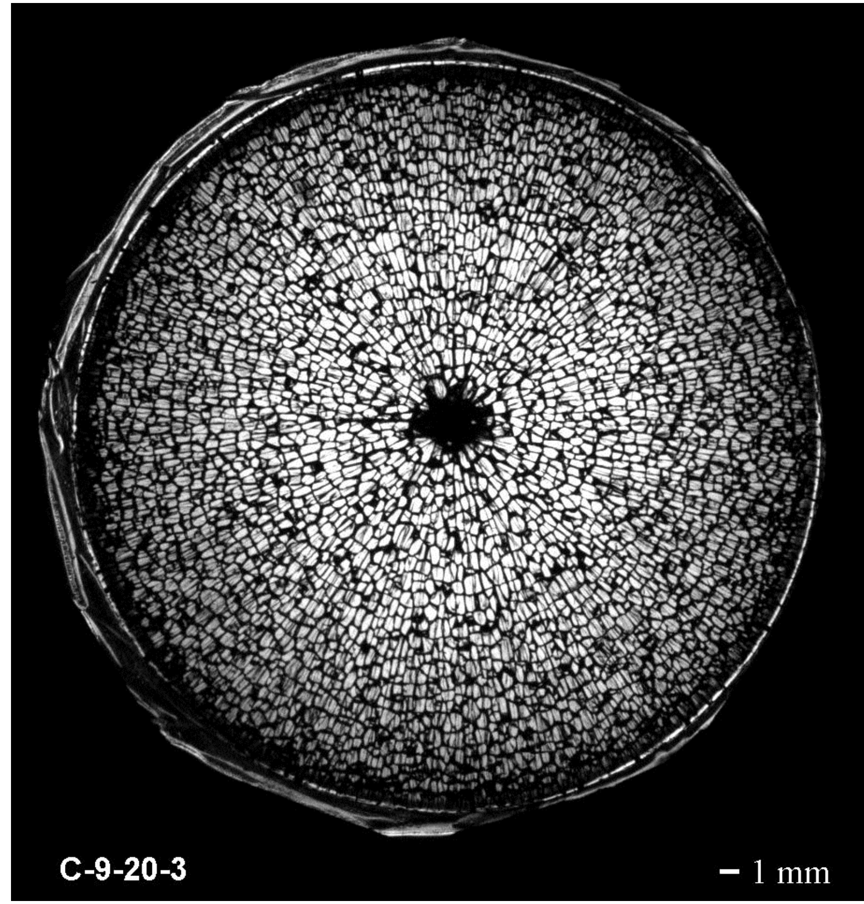
What's Possible?

- Areas from <1 mm to windows
- Thickness=0.5-3 mm (0.02-0.125")
- Strength=100-1000 MPa
- (14,500-145,000 psi)
- Central tension=5-200 MPa
- (725-29,000 psi)
- Lifetime=seconds to eternity
- Reliability: At 80% of σ_f, failure probability is 2 ppm
- Other: Coatings and patterns can be put on glass

We Can Control Glass Strength and Fracture Using Ion Exchange Time and Temperature



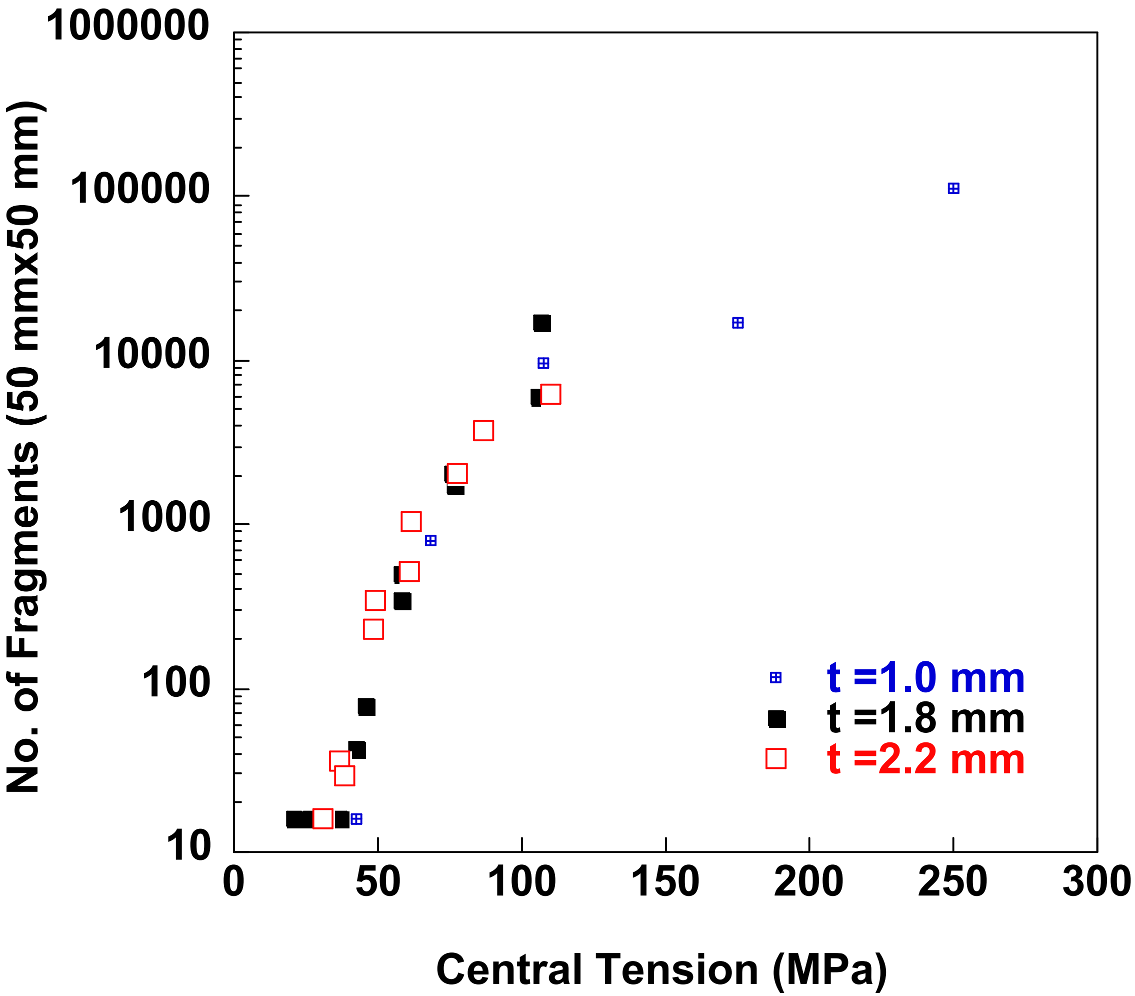
500°C, 24 hr exchange in KNO_3, thick.=1.8 mm



500°C, 96 hr exchange in KNO_3, thick.=1.0 mm

Fragmentation of taped glass disks was initiated with a diamond tip.

Number of Glass Fragments is Very Large



t=glass thickness

No. of fragments for a 50 mm x 50 mm plate

Once destruction of the glass is triggered, it is very thorough, messy, and irreversible (impossible to reassemble).

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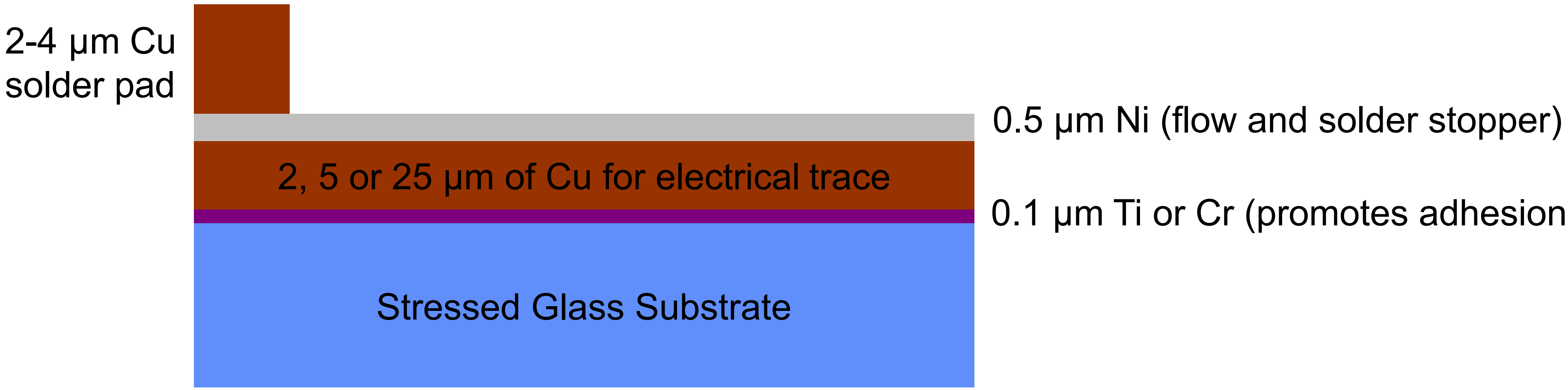
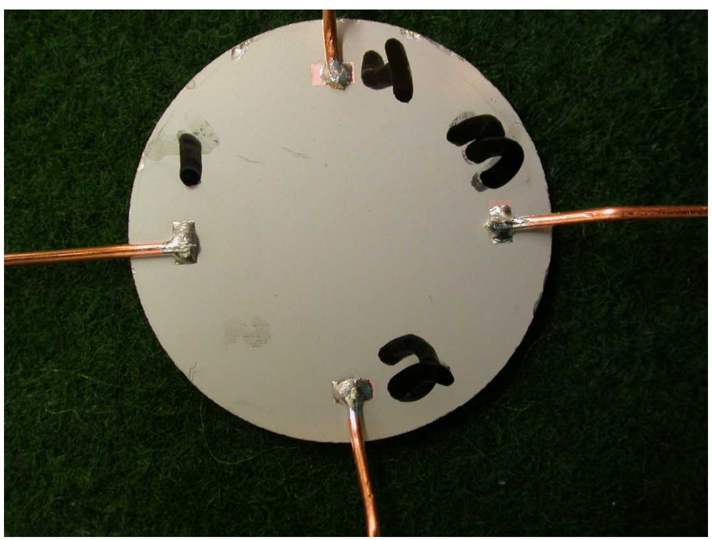
Stored Energy Available in Stressed Glass is Very Stable For Extremely Long Times

- Energy from the stress profile is available to do work or to initiate it
- Residual energy in glass and energy due to loading
  - Measurement of 0.06 J/g for residual stress
  - Calculations of 0.6×10^5 to 1×10^5 J/m^3 for residual
  - Calculations of 4×10^3 to 1×10^5 J/m^3 for loading
- Compares to:
  - 10^7 J/m^3 for shape memory alloys
  - 10^9 J/m^3 for batteries
  - 10^{10} J/m^3 for lead azide at 50% theor. density
- The **obstacle** to making use of the stable energy in stressed glass is the difficulty of breaking it to release the E

We Can Coat Stressed Glass with Conductive Coatings and Solder to Them





Top view

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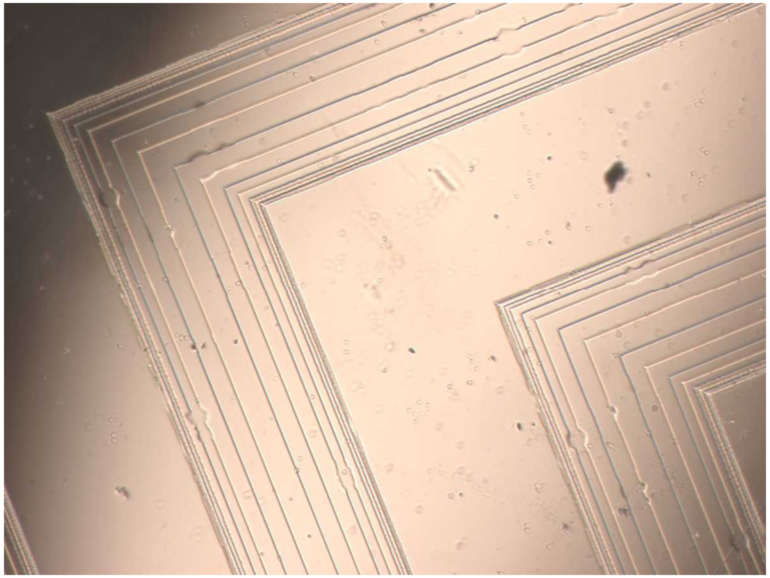
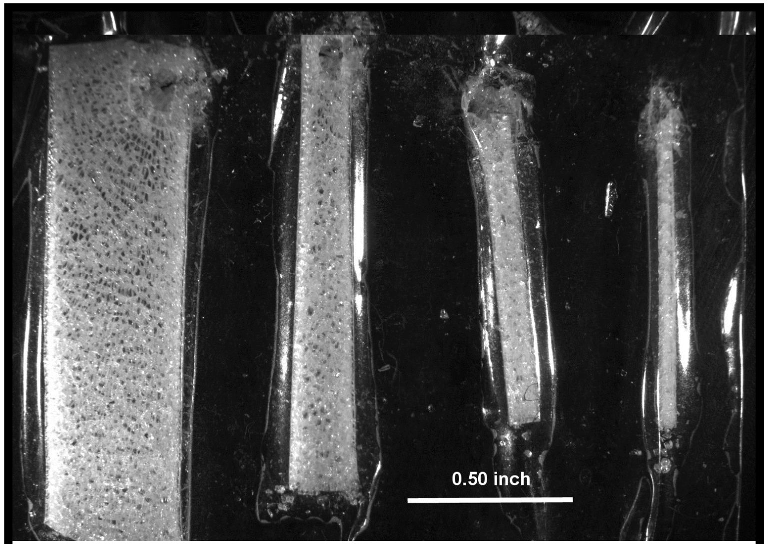
Glass Can be Patterned Before or After Ion Exchange and Maintain its Frangibility

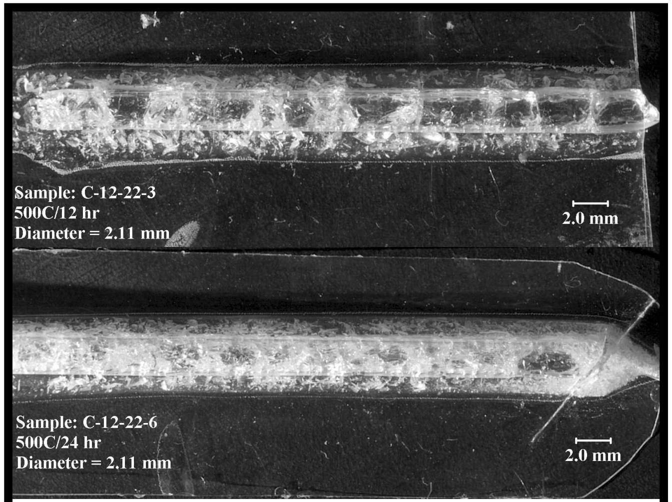
- **Masks**
  - Photoresist (PR)
  - Amorphous silicon with PR
  - Cr/Ni
- **Etches**
  - Plasma etch
  - Ion mill
  - Wet etch



Low Aspect Ratio Plates and Square Cross-Section Rods of Stressed Glass Will Fragment

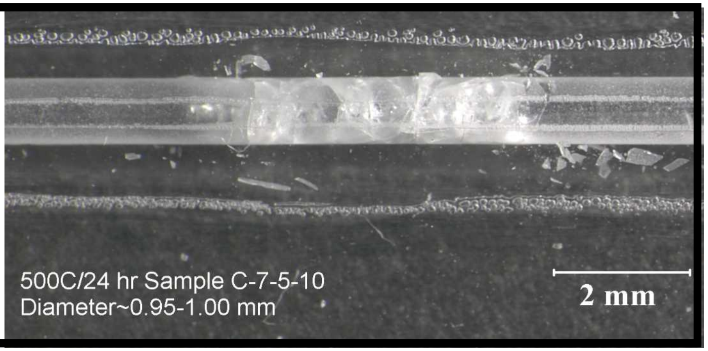


Stressed glass plates with decreasing width/thickness (w/t) ratios fragment completely.



Rectangular cross-section, stressed glass rods (dia.=2 mm) fragment completely.

Round cross-section rods and uniform diameter fibers of stressed glass do not fragment to completion. A crack starts to propagate but then terminates a short distance from the fracture initiation point.



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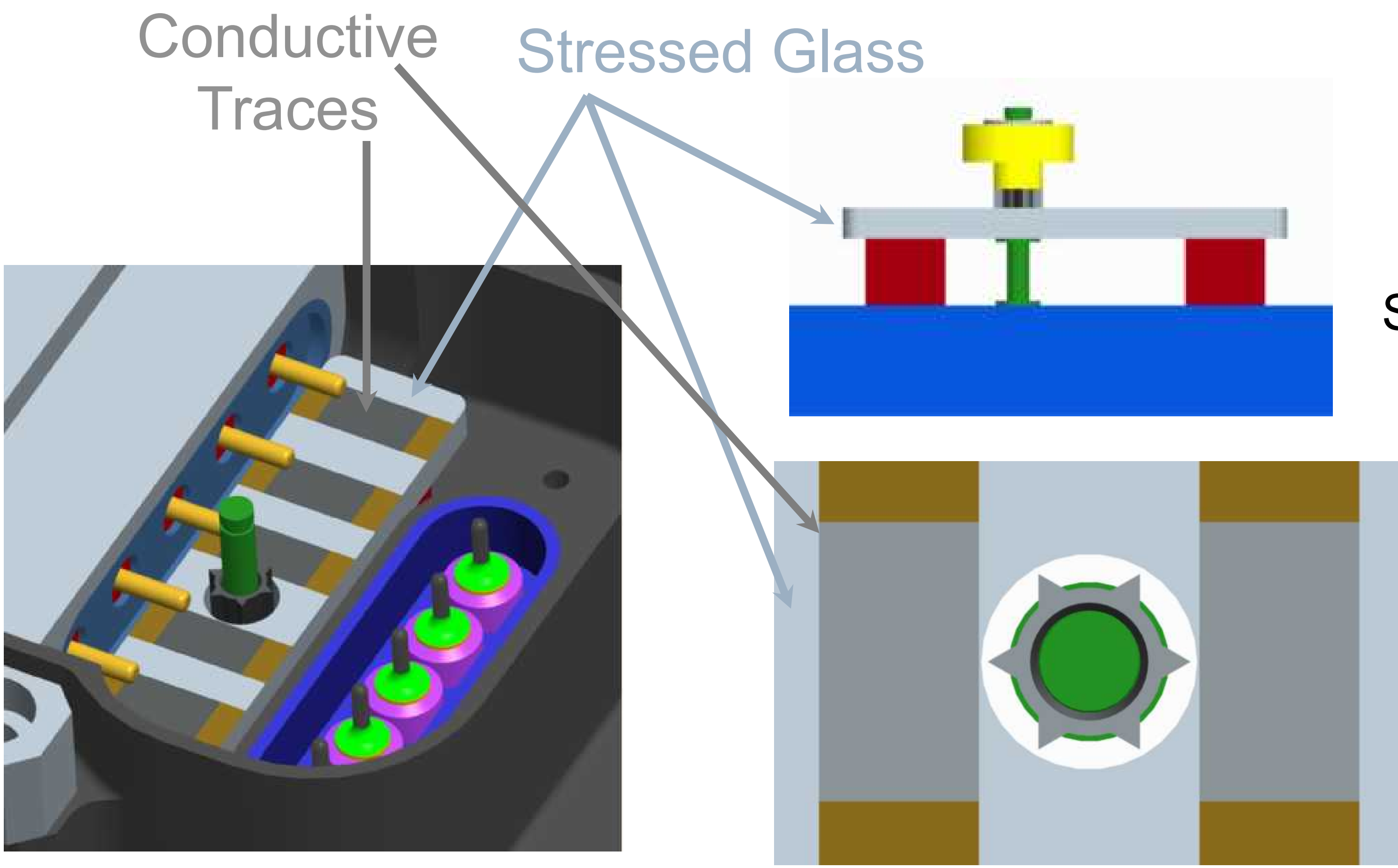
Applications

- **Sensors and nuclear weapons weaklink materials (can be used with conductive or other coatings)**
  - Mechanical shock weaklink
  - Crush weaklink
- **Containers and barriers**

1. Star Indenter Design Using Stressed Glass For a Mechanical Shock Weaklink or Sensor

1. Star Indenter Design - Deflection of Shaft Under Shock Loading Causes Glass to Fracture

Glass has conductive traces that carry necessary electrical signals.

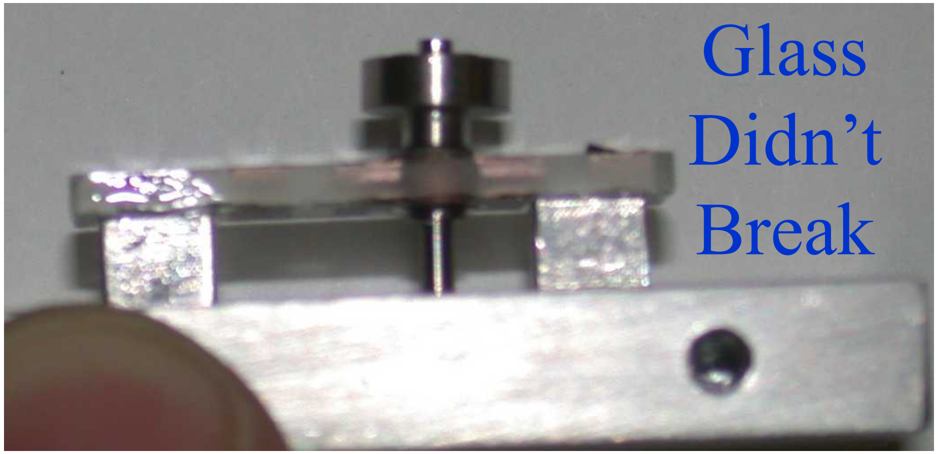


Side view

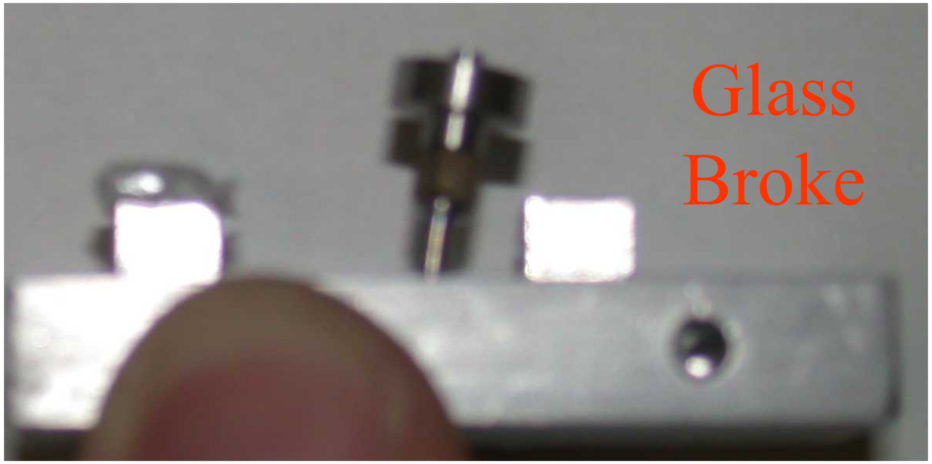
Top view and close-up of star indenter positioned in hole in glass

Shock Table Testing of Star Indenter Design Showed Stressed Glass Fractured at Expected G Loads

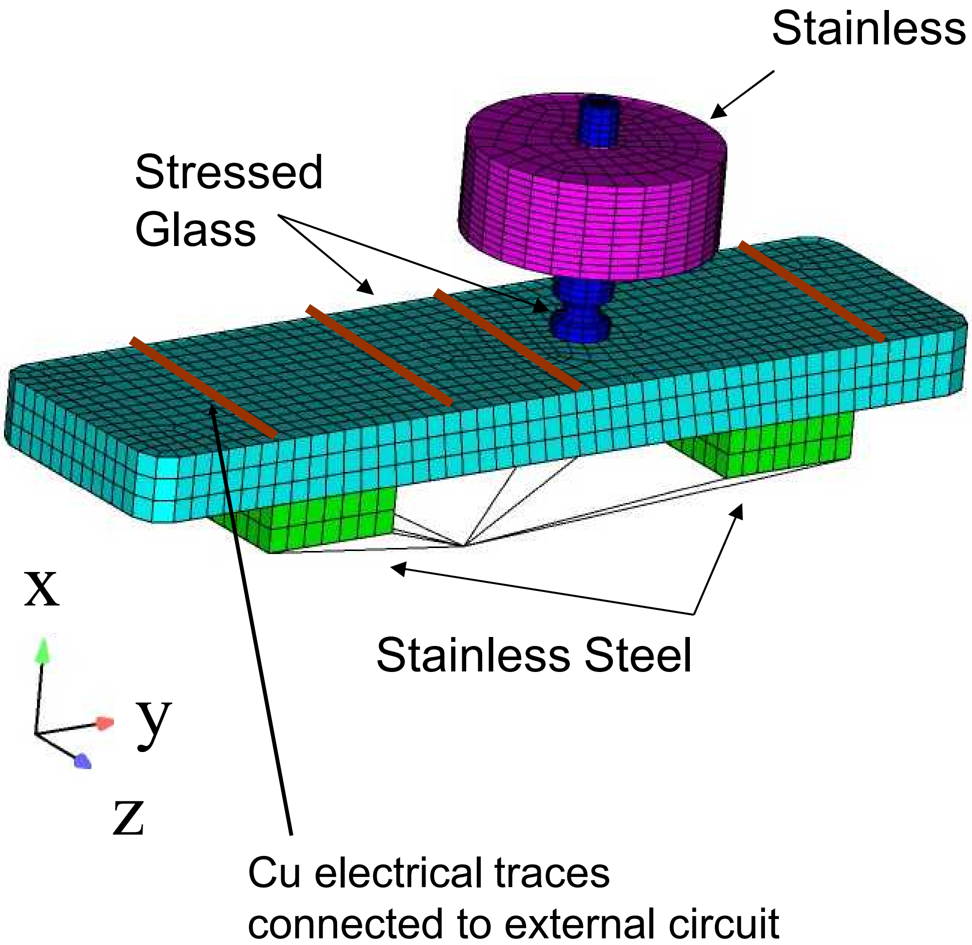
Test 5H

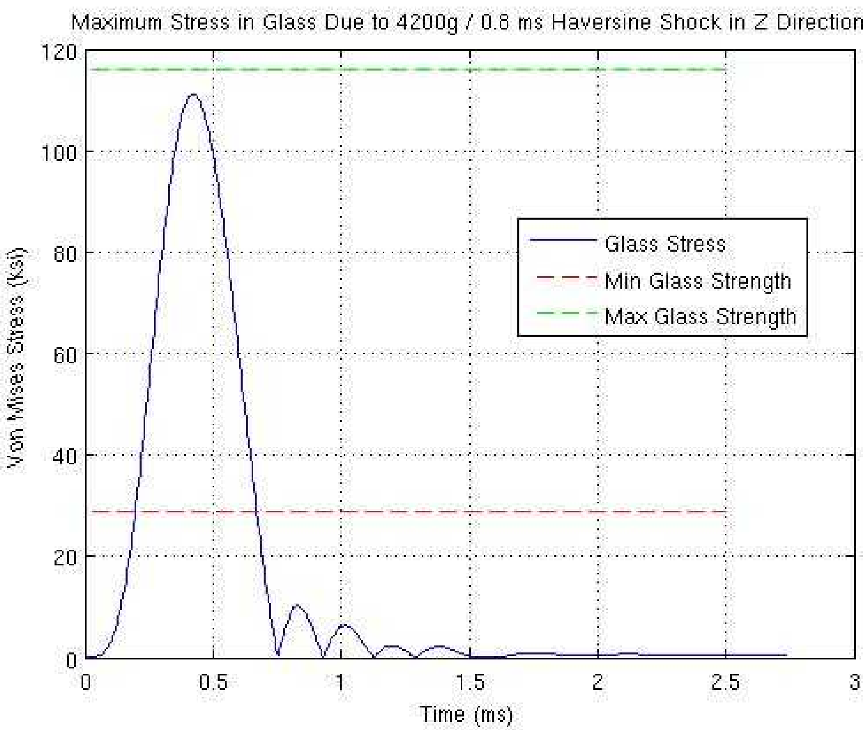


Test 4H



2. All-Glass (Rod and Plate) Design for Mechanical Shock Weaklink





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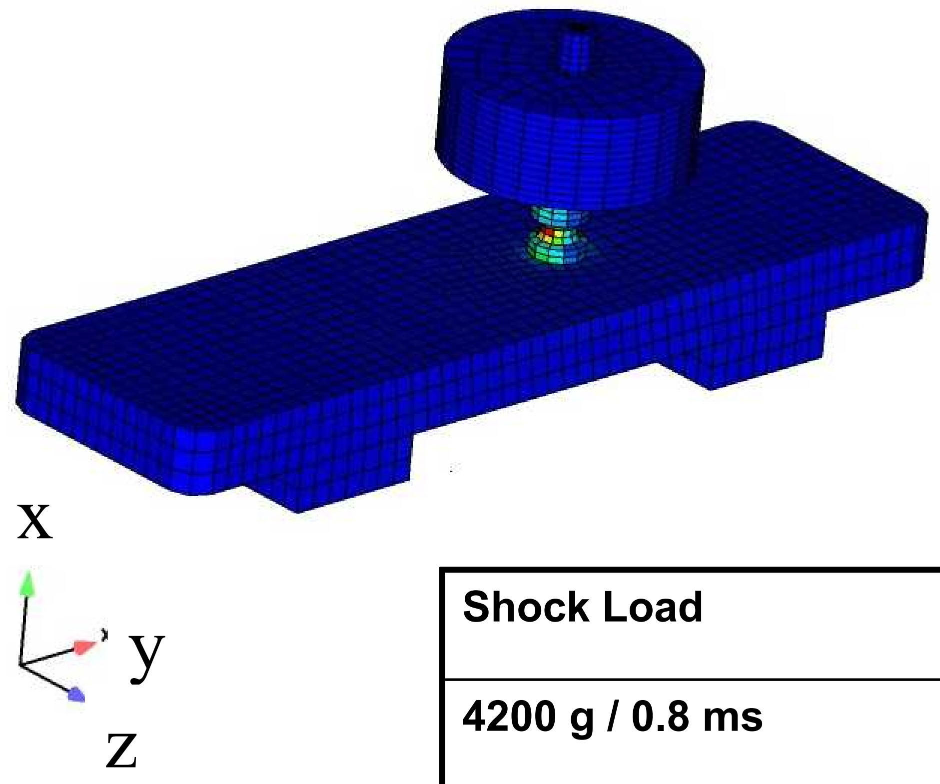
Applied Stress in Glass Shaft Due to G Loads - Able to Discriminate Different Directions

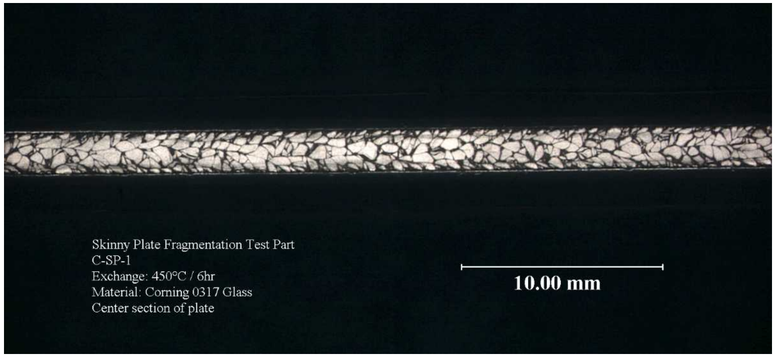


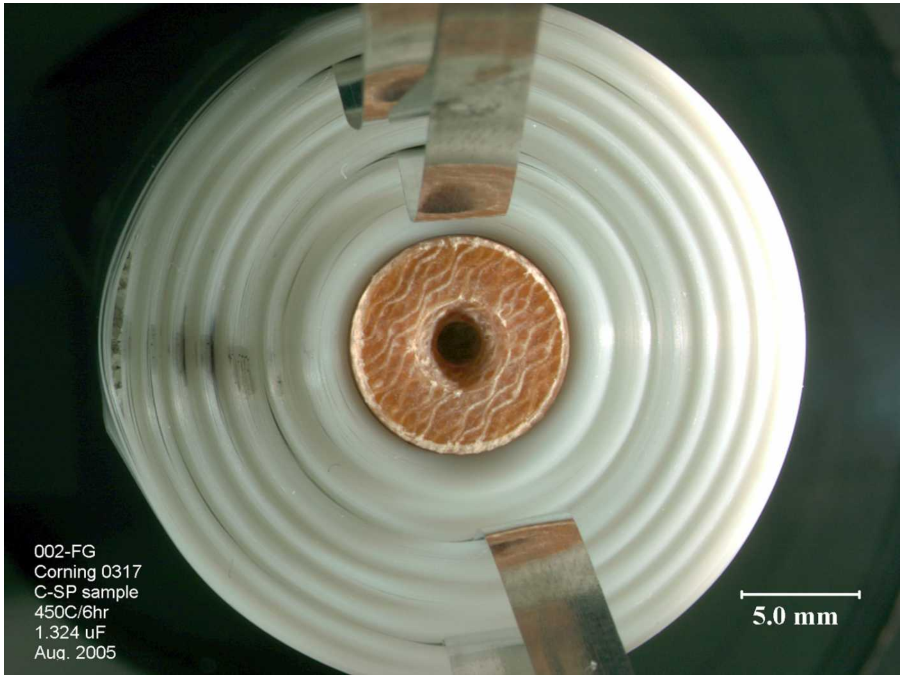
Glass strength can be tailored to be between ~ 29-116 ksi (200-800 MPa).

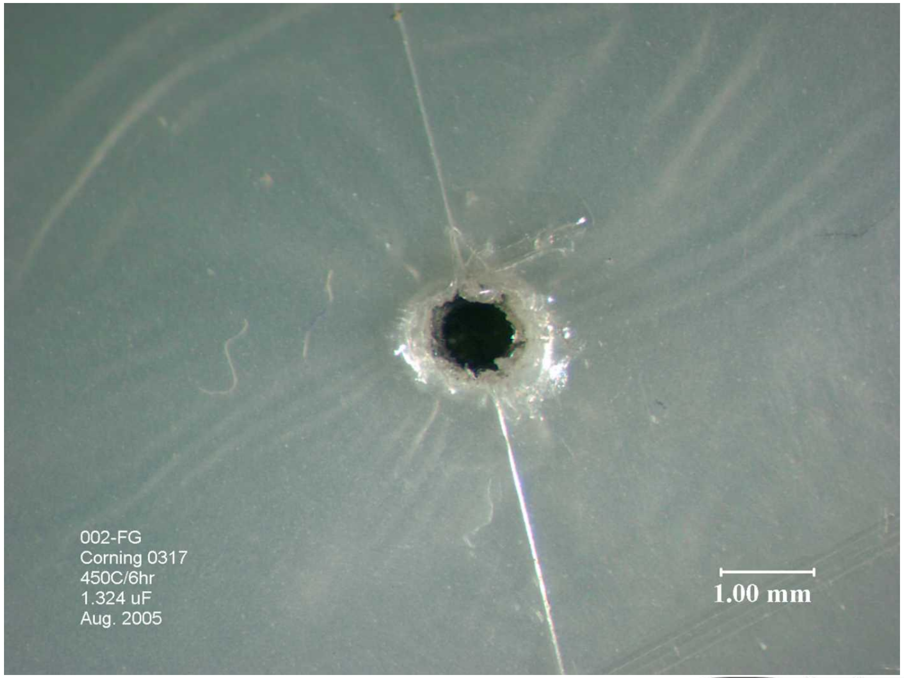
Shock Load	X	Y	Z
4200 g / 0.8 ms	5.45 ksi (38 MPa)	111.4 ksi (768 MPa)	111.2 ksi
1500 g / 0.33 ms	2.00 ksi (14 MPa)	45.6 ksi (314 MPa)	45.5 ksi

3. Embedded Stressed Glass Strip Demonstrated in a Capacitor as a Crush Weaklink

Fracture of the embedded glass strip (right) caused an electrical short and puncture in the rolled Mylar capacitor (bottom right)







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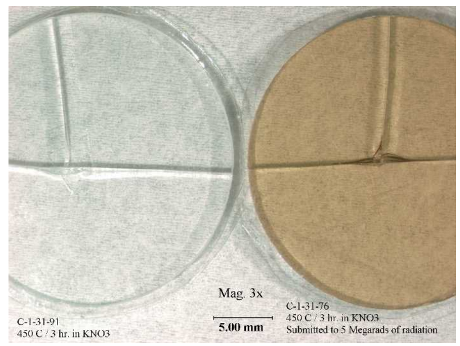
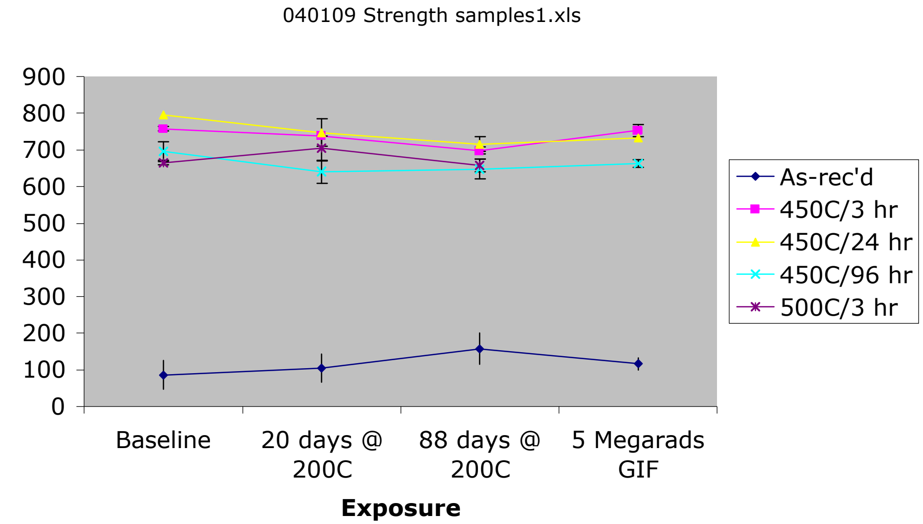
4. Irradiation and Thermal Aging Have No "Significant" Effect on the Mechanical Behavior

- Strength and fragmentation behavior unchanged for gamma irradiation
- Stress profile near surface may have been slightly modified
- Obvious color change from clear to brown

Gamma (GIF) (before/after)



Neutrons (after)



080507 SNL Stressed Glass Overview.ppt from R. Tandon and S. J. Glass

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Applications

- Sensors and materials for nuclear weapons weaklinks
- **Containers and barriers**

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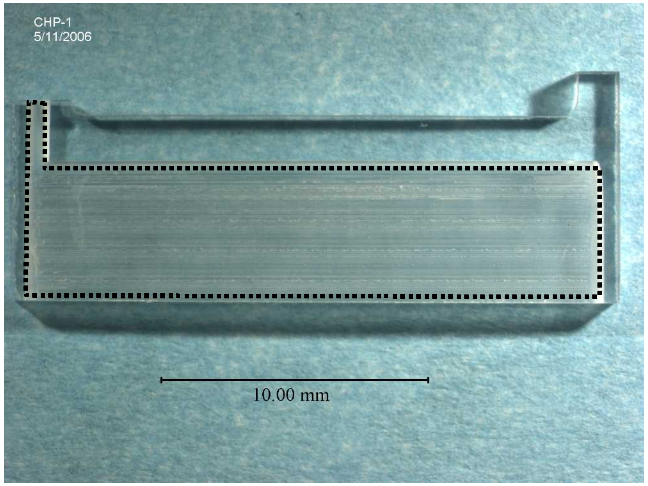
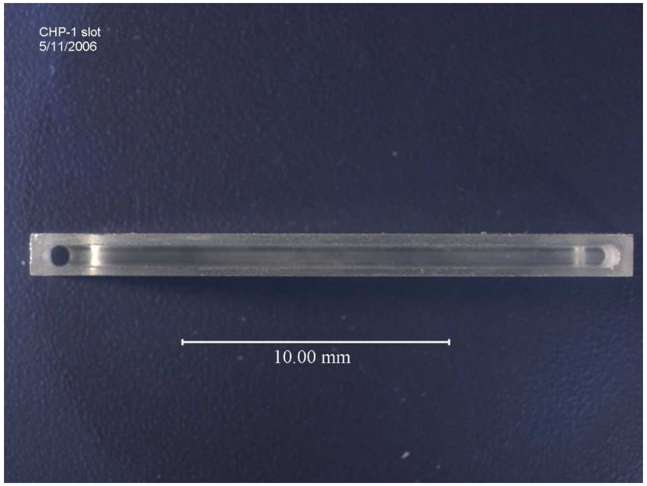
Applications - Three Approaches were Investigated to Enable the Fabrication of Open Containers

- Molding of glass plates in a furnace - unsuccessful - Corning 0317 glass does not deform readily even at elevated T's - use of a frit may be a better approach
- Traditional glass blowing - unsuccessful because high melting T of C0317 glass makes it hard to work - it also appears to offgas under certain conditions
- Machining of a cavity in glass plate - successful; however, container size (thickness) is limited to available glass thickness

Side view - access hole (top left) and cavity shown with dotted line



Bottom view of cavity in glass plate



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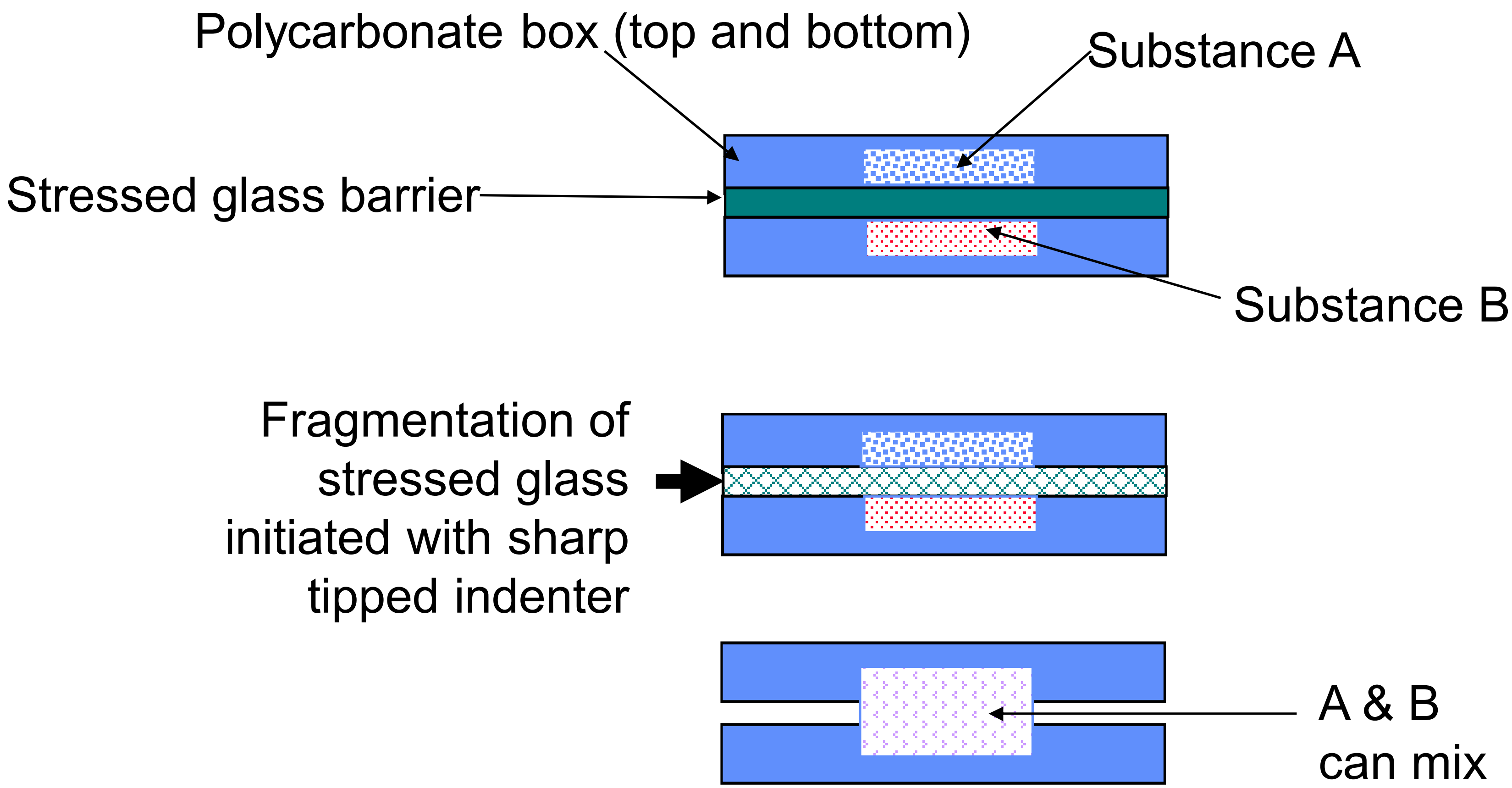
Prototype Frangible Barrier Demonstrated



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Enhancement of Fracture and Fragmentation Initiation of Stressed Glass

The high surface compression at the surface of ion-exchanged stressed glass makes it very difficult to crack and initiate fragmentation. To overcome this we investigated:

- **Creating or taking advantage of vulnerable regions in the glass**
  - Holes and masked regions
  - Edges of plates
- **Fabricating glass parts with small cross-section protrusions that fracture easily under small loads**
- **Using directed laser energy (femtosecond) to initiate fracture in the tensile region of the glass**
- **Load/unload indentation tests**

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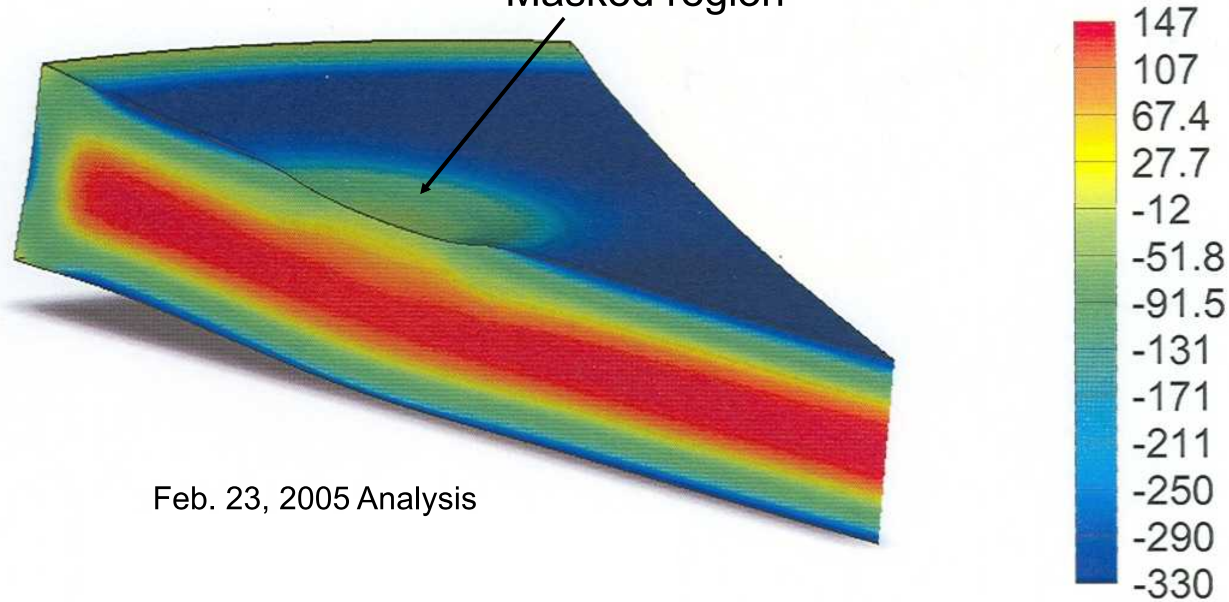
Masking Investigated as a Way to Reduce Force Needed to Break Glass in Selected Regions

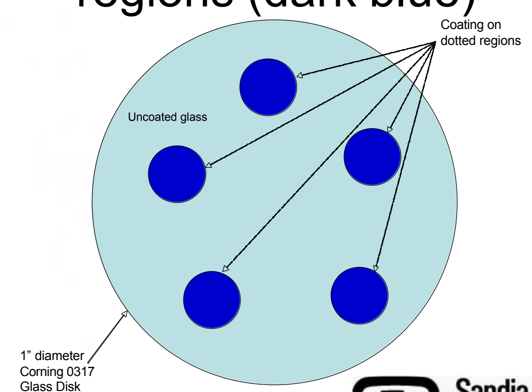
- **FEA used to predict stress profiles for disk with masked region**
- **Blue shows max compression at surface, red is max tension in the interior**
- **Masked regions and <u>edges</u> have less compression and are therefore weaker**
- **Some success achieved with masking circles using Al_2O_3 and TiN coatings prior to ion exchange**

Stress Magnitude (MPa)
(+ve=tension, -ve=compression)



Feb. 23, 2005 Analysis

Top view of glass disk with TiN or Al_2O_3 coated regions (dark blue)



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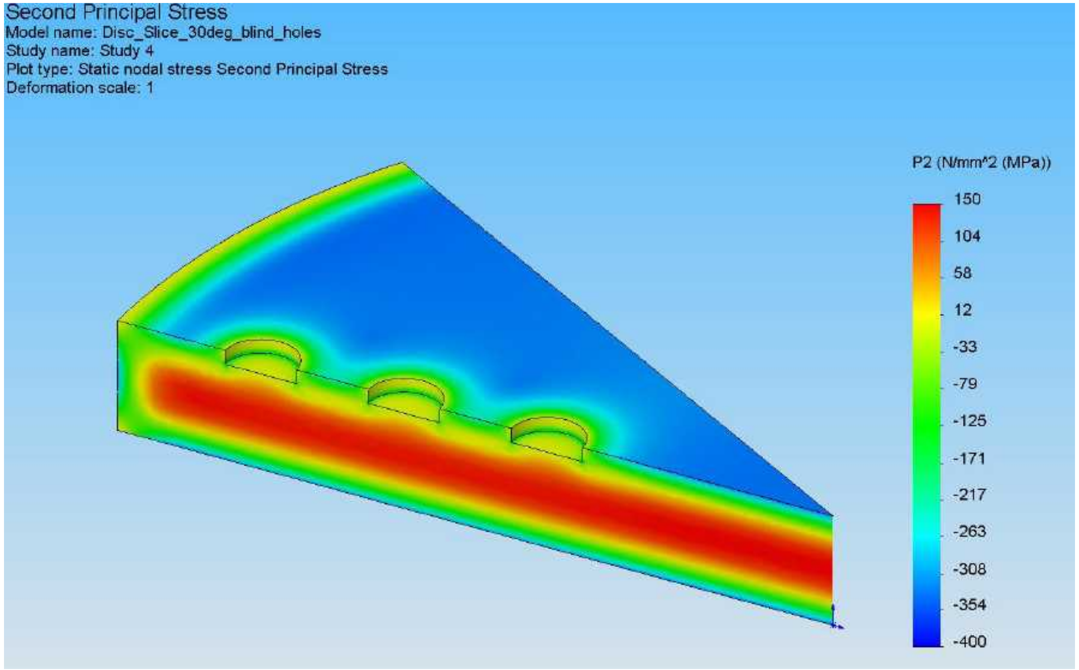
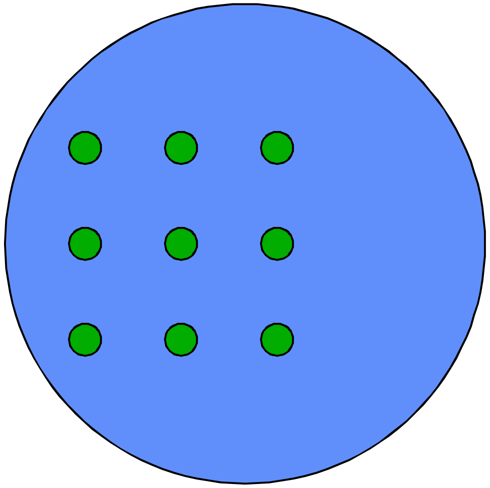
Selectively Ion Exchanging Samples Made with Holes Produces Weaker Regions Near the Holes

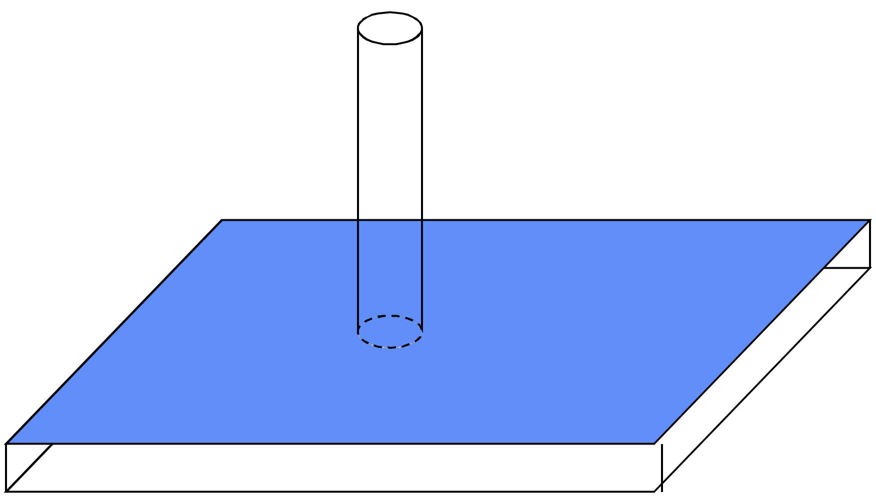
- Samples were fabricated with holes
- The holes are blocked to prevent ion exchange
- FEA predictions show a weaker region between holes
- This concept has not been evaluated experimentally

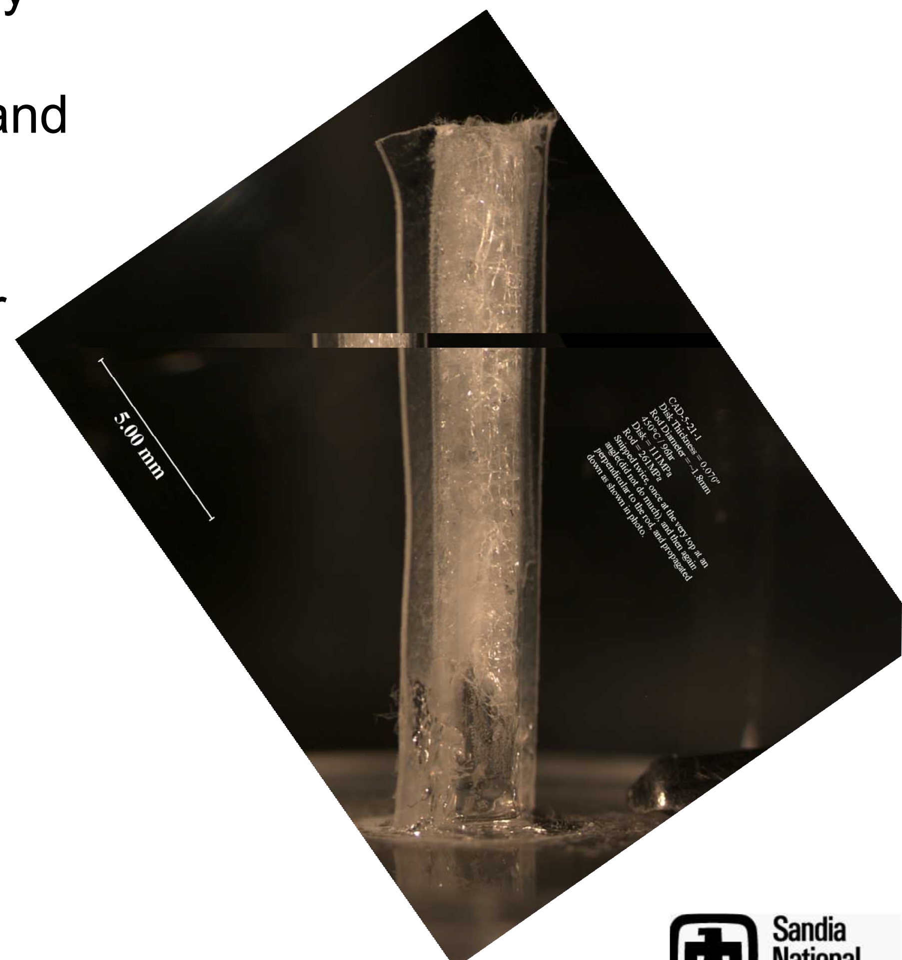




Thin Cross-Section Protrusions Have Been Fabricated on Glass Plates to Enable Fracture

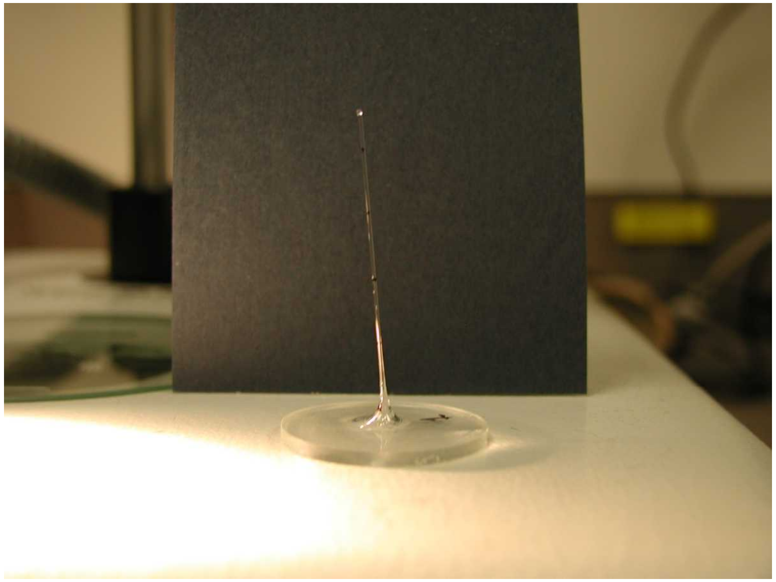
- Glass blowing techniques used to bond rods to glass parts - the joined assembly is then ion exchanged
- More work needed to obtain complete and reliable fragmentation - round cross-section rods may not be best choice to achieve desired fragmentation behavior

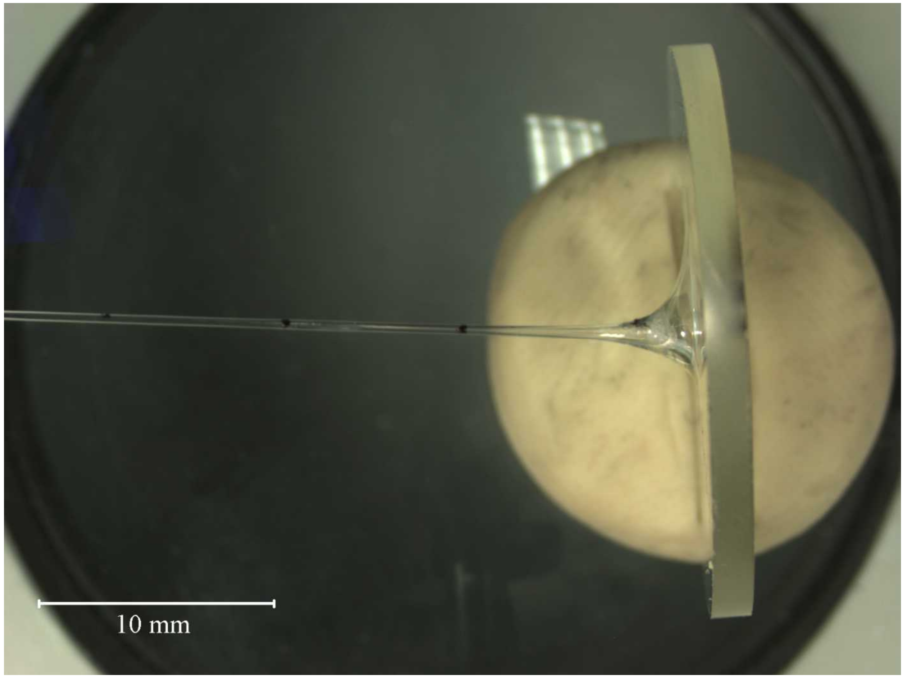




Thin Cross-Section Protrusions Have Been Fabricated on Glass Plates to Enable Fracture

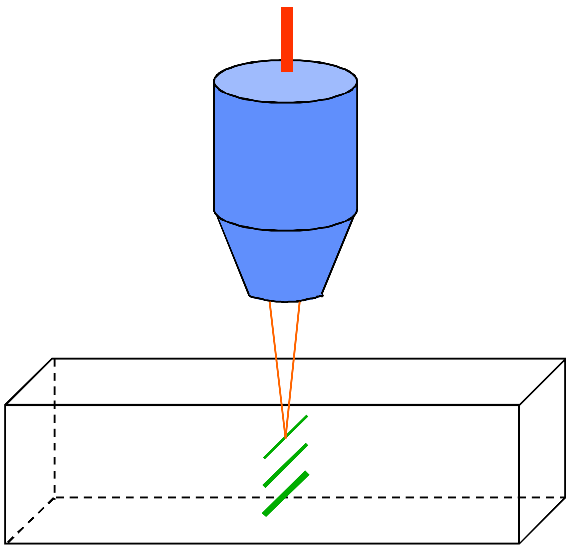
- Glass blowing techniques used to fiber-like protrusions to glass parts - the joined assembly is then ion exchanged
- A fracture that was initiated in the fiber propagated into the base plate - further work needed to understand and take advantage of this behavior

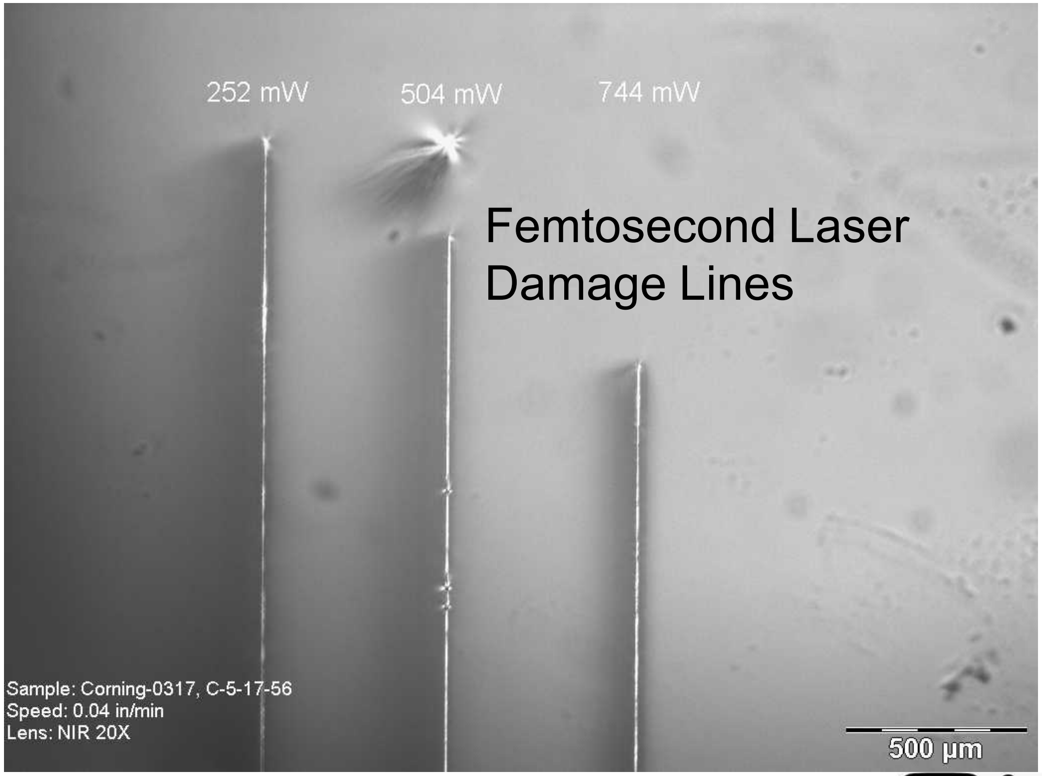




Directed Laser Energy Used to Create Damage and Initiate Fracture in Tensile Region of Stressed Glass

- Subsurface damage lines were written in the weak central tension region of ion-exchanged glass samples using a femtosecond laser
- Above a critical power level and line length the damage led to instantaneous and complete fragmentation of the glass





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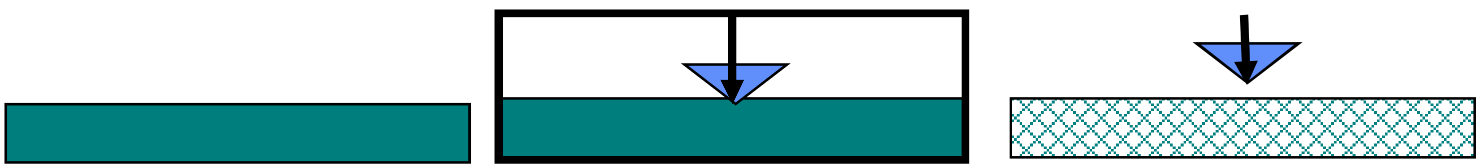
Load/Unload Indentation Tests used as a Demonstration of an Approach to Initiate Fracture

- Sharp-tipped diamond indenters are pushed into the surface of stressed glass using high loads (>300 N) to initiate fracture
- Fracture and fragmentation occur when the diamond tip is removed from the glass surface, not when it is contacting the surface under load
- To produce <u>easy</u> fracture the glass could be pre-loaded with a diamond tip - at a later time the tip could be removed from the surface to initiate fracture
- We conducted a series of tests to determine the contact load required to ensure glass fracture on removal of the tip

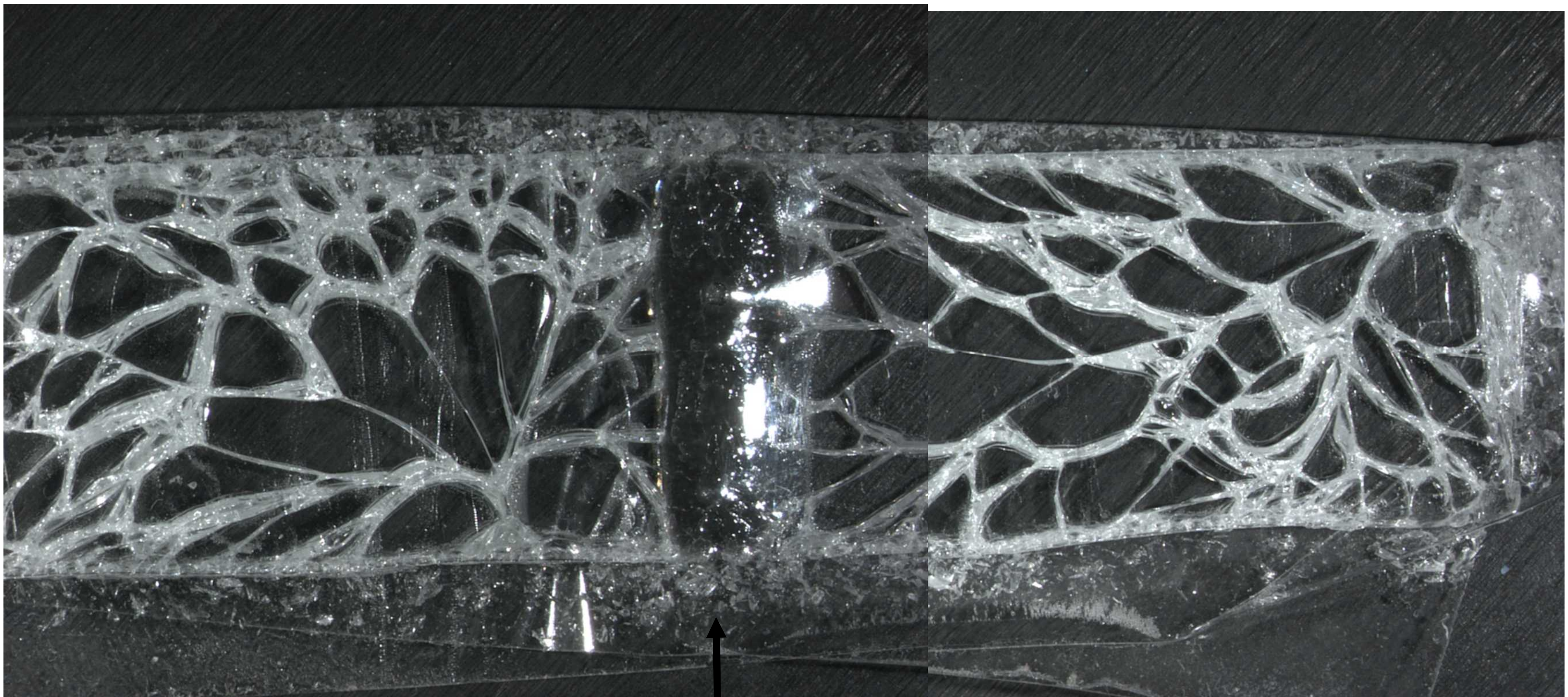


Side view of stressed glass disk

Side view of fixed loading (300 N) of a stressed glass disk with a diamond tip

Side view of glass after diamond tip is removed - instantaneous and complete fracture of the disk has occurred

More Complex Geometries Can be Made by Joining with Low Temperature Frit



Fracture transfer across interface made from sealing glass with appropriate coefficient of thermal expansion