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# Sharp Contact Damage in Ion-Exchanged Cover Glass

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# CORNING

## Sharp Contact Damage in Ion-Exchanged Cover Glass

Tim Gross, Jim Price, Scott Glaesemann

Functional Glasses: Properties and Applications for Energy and Information

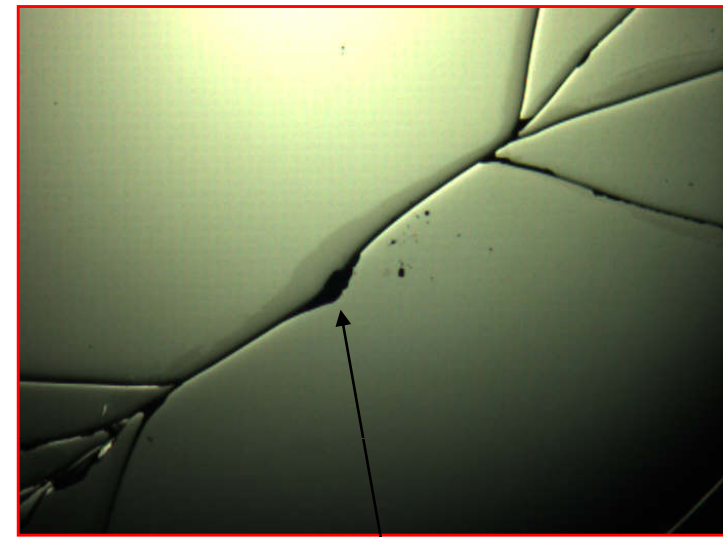
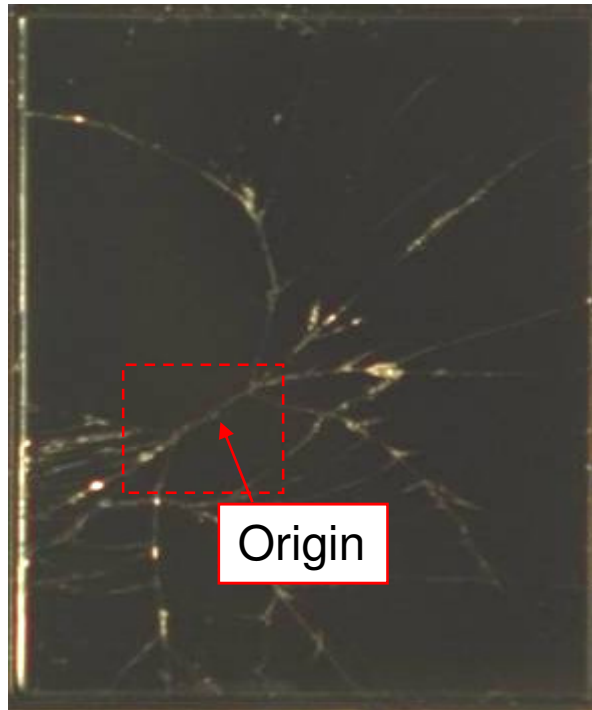
January 6-13 in Siracusa, Sicily, Italy

# Overview

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- Sharp point contact, the primary failure mode in ion-exchanged cover glass.
- Replication of field damage using diamond indenters
  - Role of indenter angle and rate of contact.
- Sharp scratch events, the source of highly visible cosmetic damage.
- Measurements of retained strength following sharp contact events.

## Primary failure mode in ion-exchanged cover glass is sharp contact and associated flexure

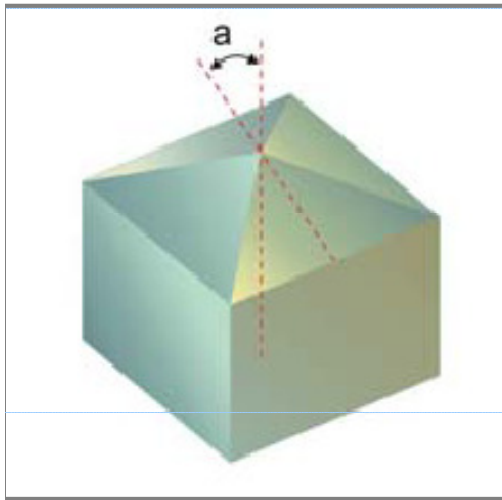


- Sharp contact deformation is defined by the glass response. It occurs when the contact load is distributed over small contact area and elastic limit is exceeded resulting in permanent deformation.
- Strength limiting flaw formation initiates within the permanent deformation region.
- Crack extension to failure occurs as contact flaws extend through the depth of compressive layer.

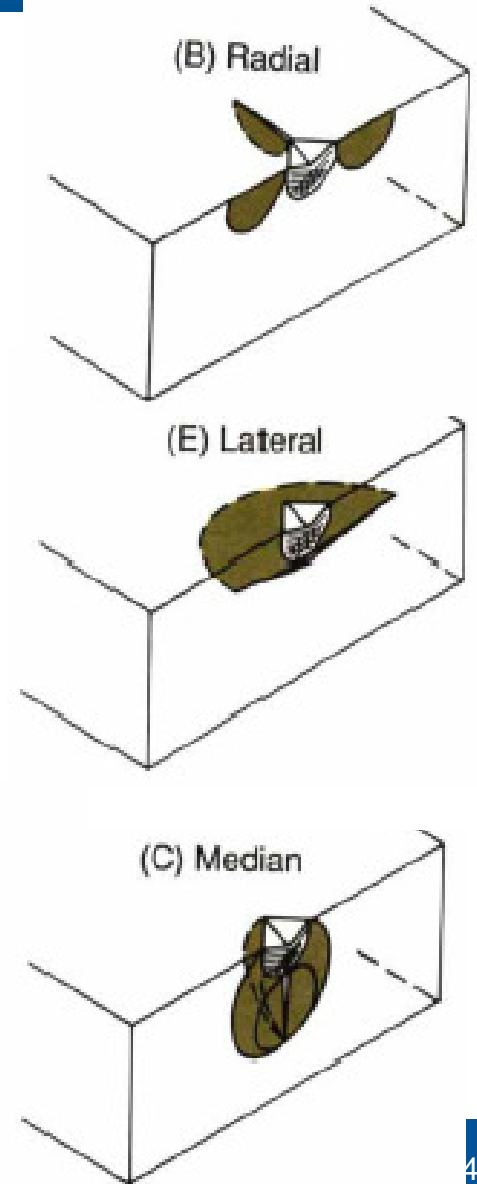
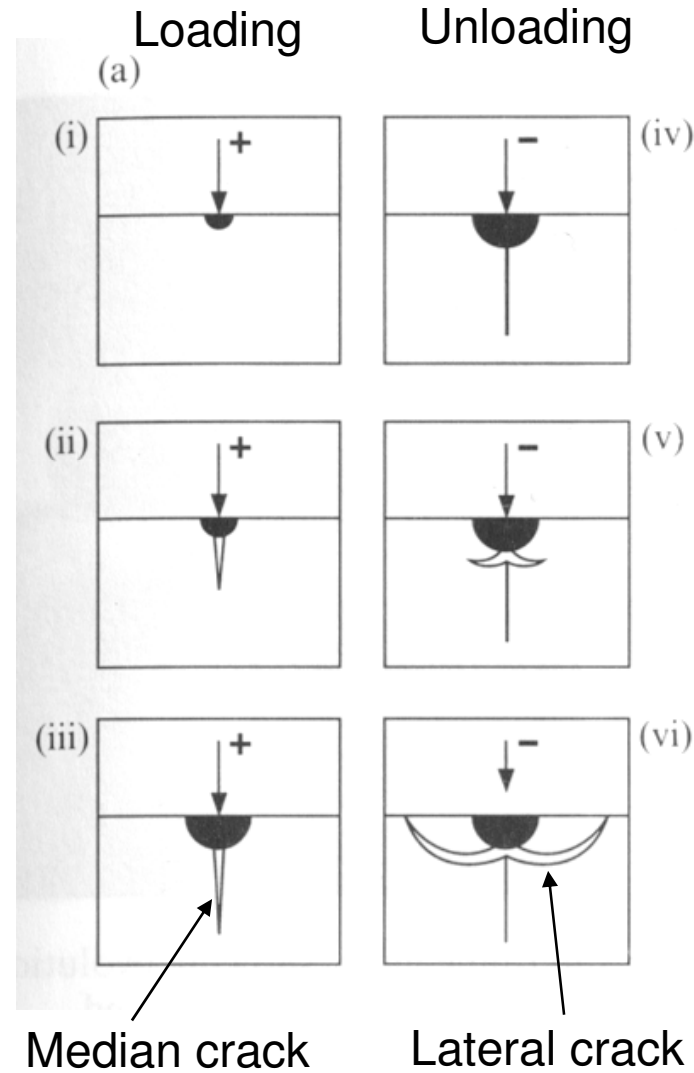


# Using diamond indentation to mimic the response from sharp contact

B. Lawn, Fracture of Brittle Solids, Cambridge Univ. Press, 1993.

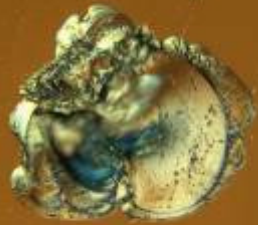


The Vickers diamond indenter is a 4-sided pyramid with angle between opposite faces  $2a = 136^\circ$

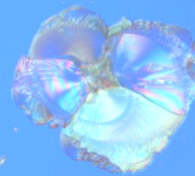


Vickers indents in ion-exchanged glass produce the median/radial and lateral crack systems seen in field damage

**Sharp Contact Field Damage**



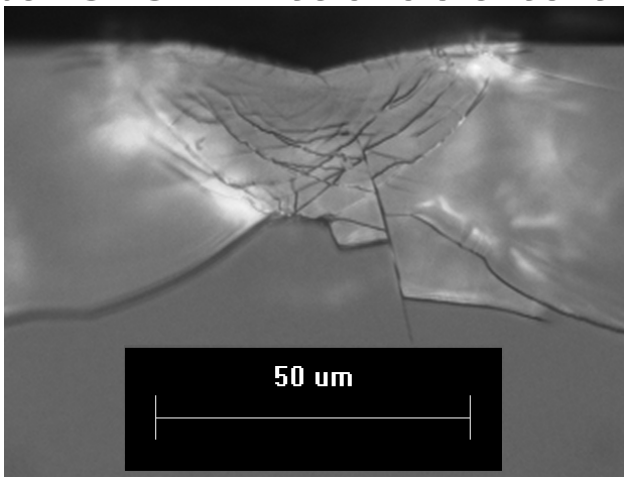
**Damage produced by Vickers indenter**



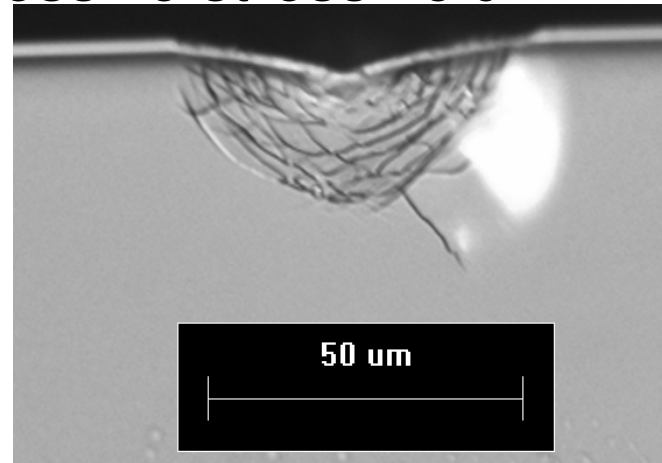
- Indentation is used to determine the resistance to the formation of strength limiting flaws, i.e. median/radial cracks, that are oriented perpendicular to the glass surface.
- The Vickers median/radial crack indentation threshold of alkali aluminosilicate glass increases from ~500 gf for non-strengthened glass to ~7000 gf for glass ion-exchanged to compressive stress (CS) ~700 MPa and depth of layer (DOL) ~50 microns.
- With DOL sufficient to contain the deformation region, the Vickers median/radial crack initiation load increases with CS for a given glass type. For example, if the depth of compressive layer is fixed at 50 microns, an alkali aluminosilicate sample with surface compressive stress of 500 MPa has a cracking threshold of 4 kgf, while a sample with surface compressive stress of 800 MPa has a cracking threshold of 7 kgf.

# Deformation of ion-exchanged glass

- Following ion-exchange, glasses still deform by the same mechanism as in the non-ion-exchanged glass.
- Normal glasses deform primarily by a shearing mechanism both pre- and post- ion-exchange.
- However, propagation of shear damage into median/radial cracks is limited due to the compressive stress field.



Cross-section of 1 kgf Vickers indentation in non-ion-exchanged normal glass



Cross-section of 1 kgf Vickers indentation in ion-exchanged normal glass

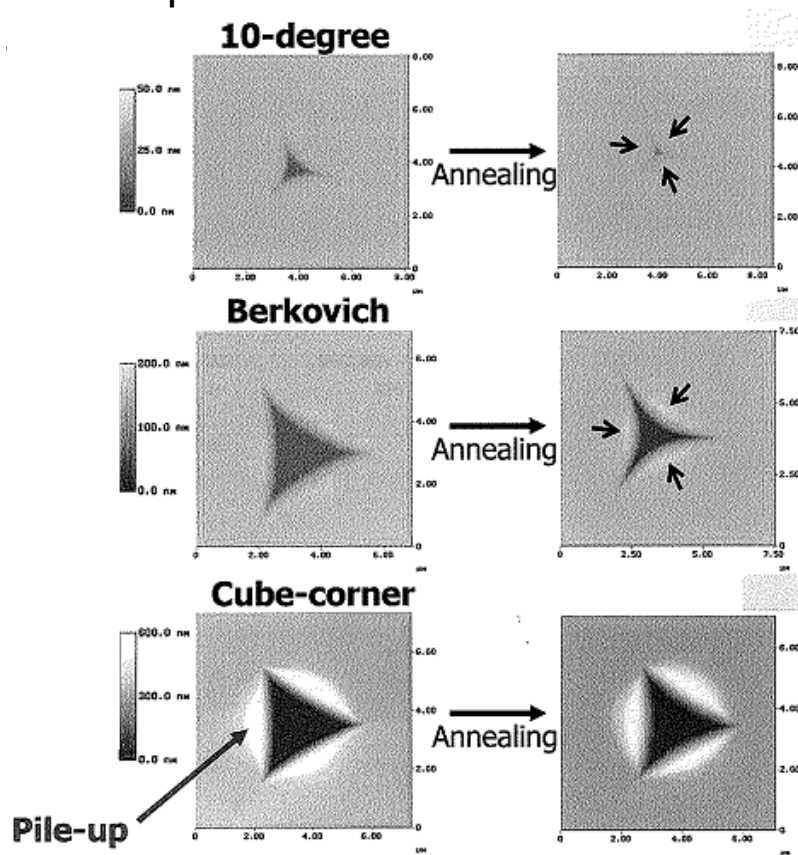
# Deformation mechanisms

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- The resulting permanent deformation by sharp contact is the result of two competing mechanisms: shear deformation and densification. Deformation mechanism depends on glass structure (i.e. network connectivity, free volume), contact geometry, and rate of contact.
- Shear deformation – Volume displacing mechanism leads to “pile-up” at the periphery of the indent. Indentation with sharper indenter tips favor shear deformation.
- Densification- Glass is compacted rather than displaced. Indentation with blunter indenter tips favor densification. High rate contact also appears to favor densification.

# Effect of indenter sharpness on amount of densification

Using densification recovery technique described by Mackenzie [JACS 46(1963) 461], Yoshida et al. demonstrated the reduction in densification with increasing indenter sharpness.

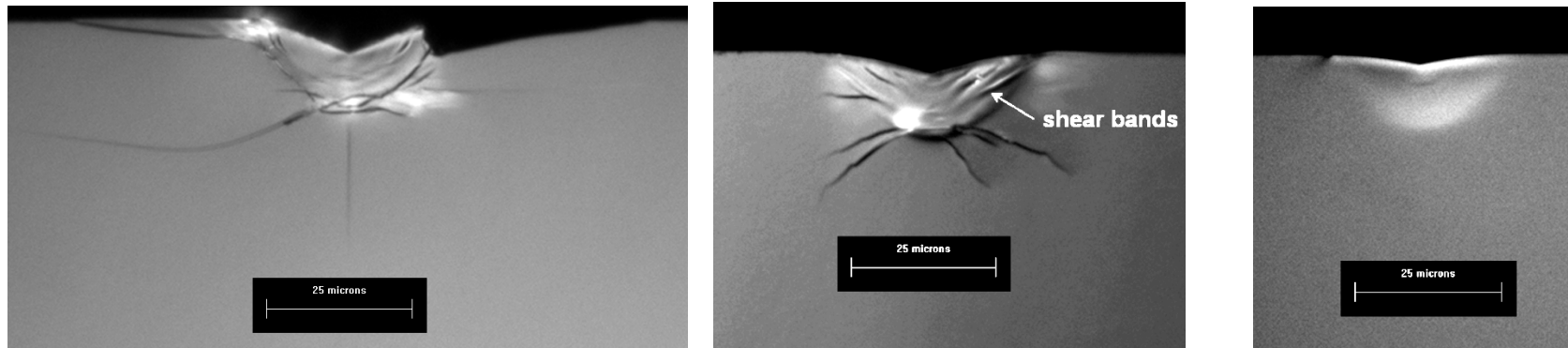


- Blunt tips produce more densification during indentation.
- During densification the glass is compacted rather than displaced.
- Subsequent sub  $T_g$  heat-treatment leads to nearly a full recovery of the deformed material.

- Sharp tips produce more shear deformation during indentation.
- This deformation leads to displaced material that piles up at the edges of the indent impression.
- Heat-treatment does not recover material that has deformed by plastic flow.

Yoshida et al., *J. Mater. Res.*, 25, 2203-2211 (2010).

# Cross-section of indents made in non-IXed alkali aluminosilicate at 500 gf with various indenter tips



120° tip

136° tip

150° tip

← Sharper, more shear

Blunter, more densification →

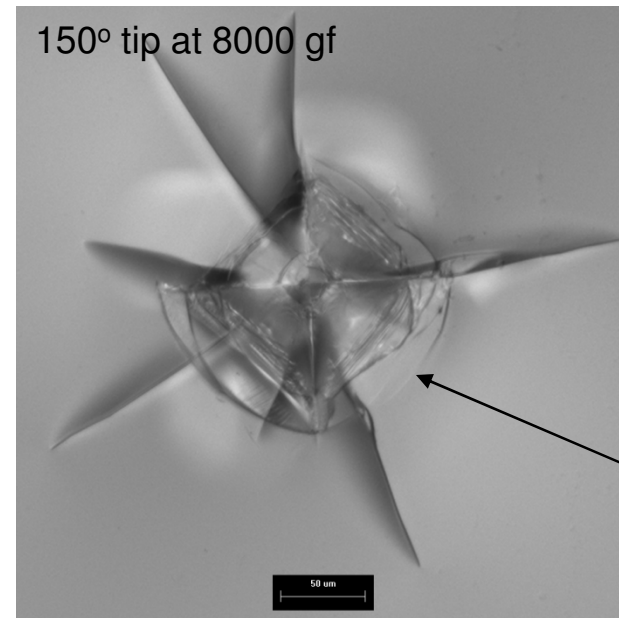
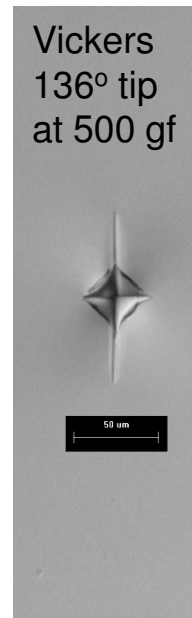
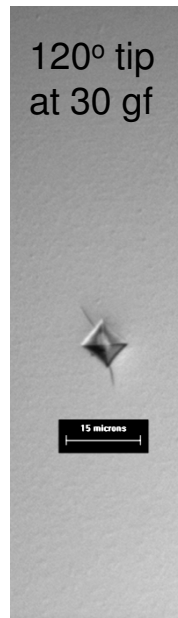
Deformation by a shearing mechanism creates subsurface cracking damage in the deformation region that initiates larger crack systems, i.e. median/radial and lateral cracks. The extension of crack systems is driven by the greater residual stress that results from volume-displacing shear deformation.

Deformation by densification produces less sub-surface damage and less residual stress, so that the threshold load required to initiate cracking systems increases.

Densification increases resistance to “normal” cracking. However, as the degree of densification increases, the propensity towards cone cracking also increases.



Indentation cracking behavior on the surface in non-IXed parts also indicates the change in deformation mechanism towards densification as the contact becomes blunter



Ring crack

Normal sharp cracking behavior indicates that deformation occurs with significant shear deformation

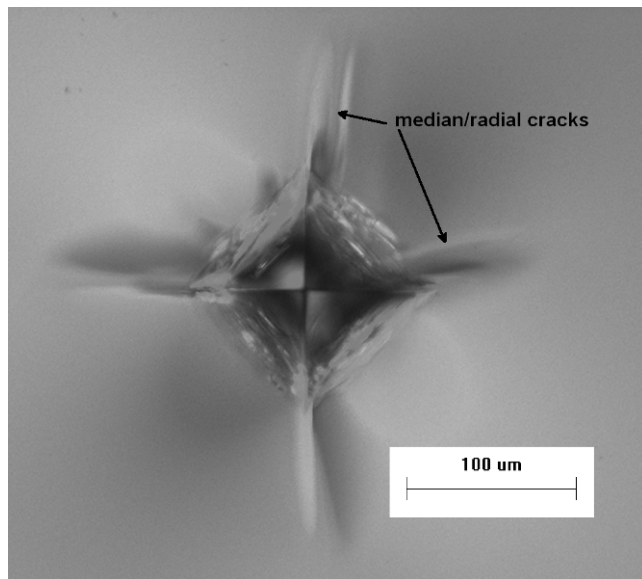
Mixed normal/anomalous cracking behavior indicates that deformation is occurring with greater densification

Resistance to the formation of cracks increases as the deformation mechanism tends towards densification

Indenter tip	Non-ion-exchanged median/radial cracking threshold (gf)	Ion-exchanged median/radial cracking threshold (gf)
120°	15-30	50-100
136°	300-500	5000 - 7000
150°	7000 - 8000	>10,000

# Deformation mechanisms & rate of contact

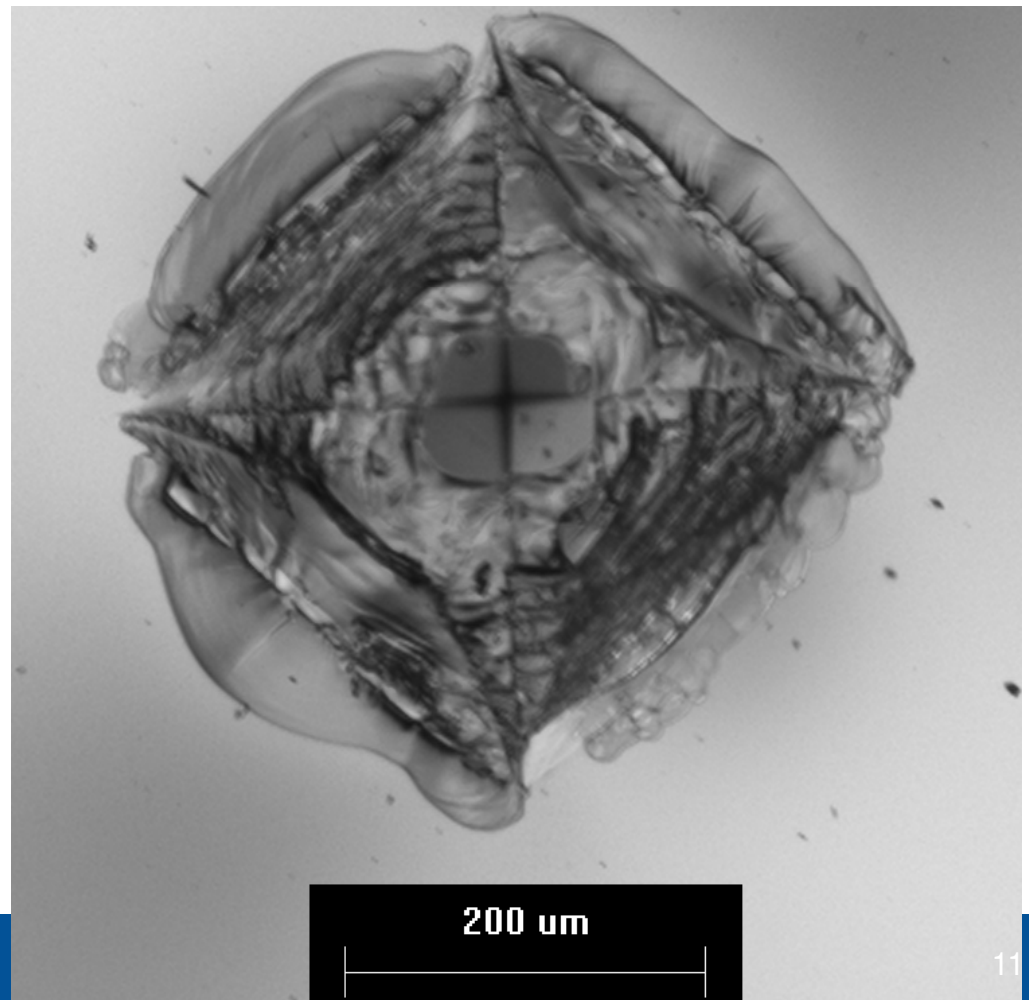
Ion-exchanged alkali aluminosilicate indented at 7 kgf. Quasi-static indentation with load/unload rate = 0.2 mm/min (0.00333 mm/s), dwell time = 10 seconds.



CS = 814 MPa DOL = 54 microns

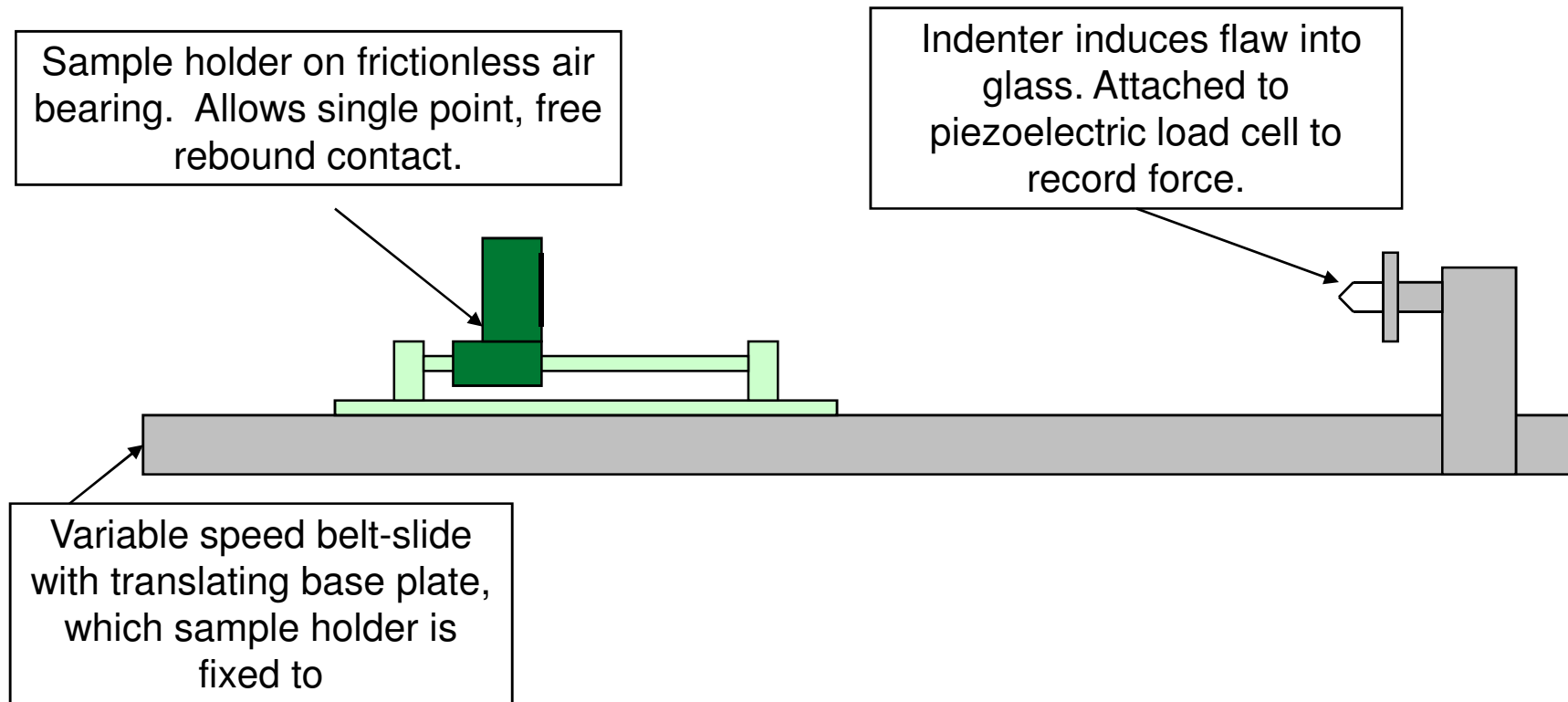
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Ion-exchanged alkali aluminosilicate indented with Vickers at 57.3 kgf at 410 mm/s impact velocity. No median/radial cracks

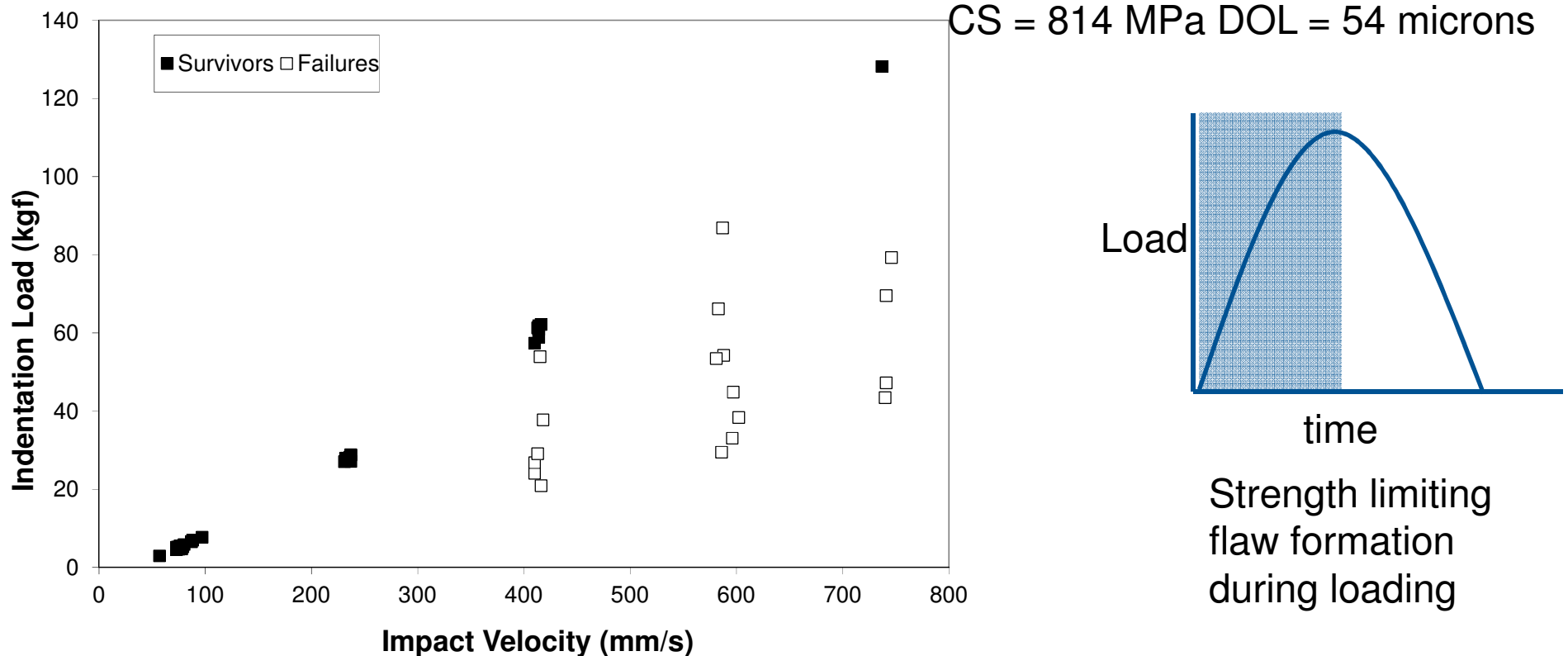




# Schematic of dynamic indenter

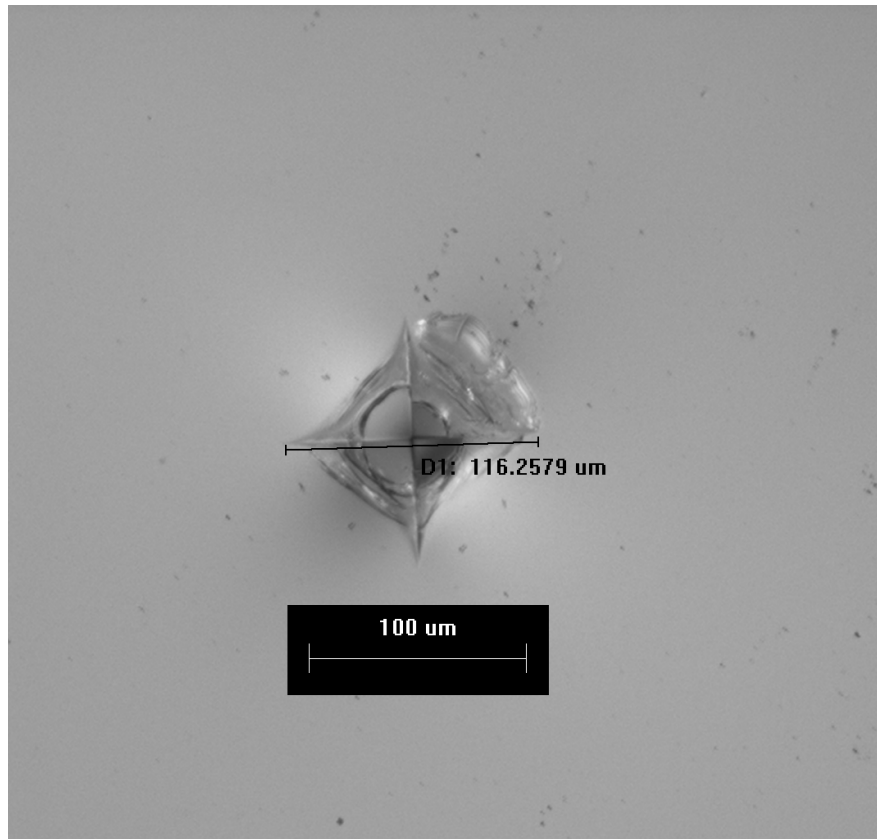


# Dynamic Vickers median/radial cracking threshold is substantially higher than for quasi-static indentation

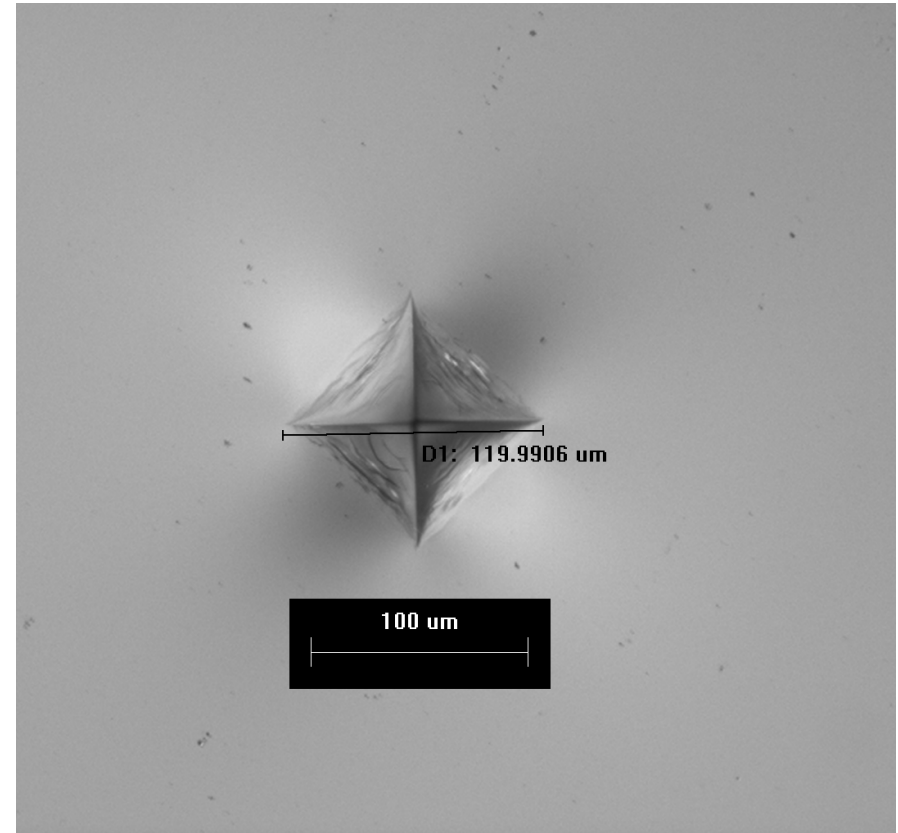


- For quasi-static Vickers indentation, median/radial cracks form during unloading.
- During dynamic Vickers indentation, median/radial cracks form during loading.
- Increased contact stress on median plane during loading is an indicator of densification according to Yoffe [Phil. Mag. A 46 (1982) 617]. Hint at a transition towards densification at high rate Vickers indentation?

# Indentations made at 4.5 kgf by dynamic and quasi-static indentation



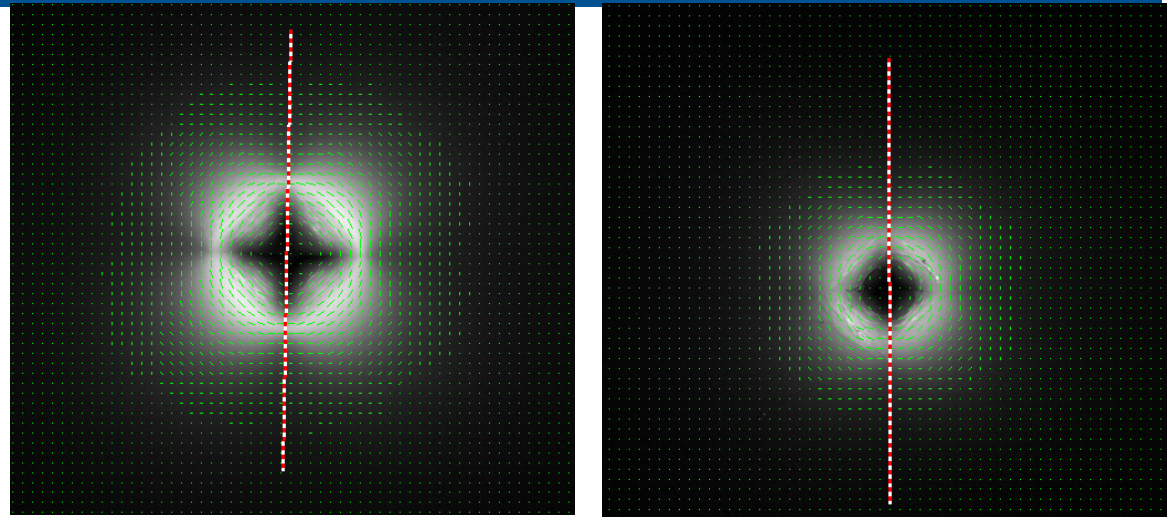
IXed alkali aluminosilicate indented at 4.5 kgf using dynamic indenter, contact event time 1266 microseconds, diagonal length ~ 116 microns.



IXed alkali aluminosilicate indented at 4.5 kgf using dynamic indenter, contact event time 55 seconds, diagonal length ~ 120 microns.

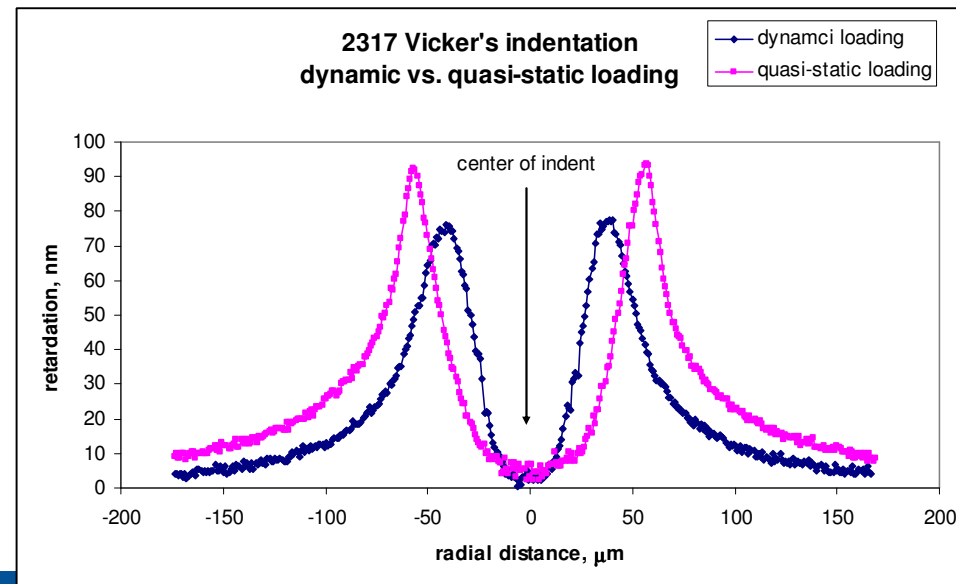
# Optical retardation indicates a drop in residual stress for a given load during dynamic contact

Quantitative 2D grayscale maps of stress-induced optical retardation of 4.5 kgf indents made in alkali aluminosilicate specimens using quasi-static and dynamic indentation. Magnitude of retardation is significantly larger for the quasi-static loading case.



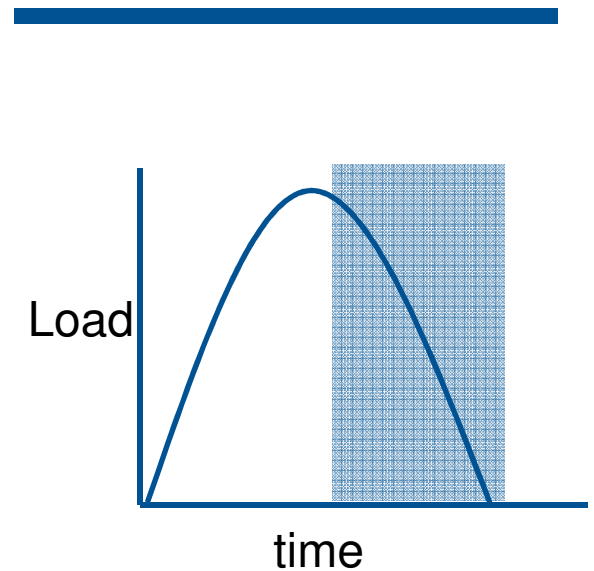
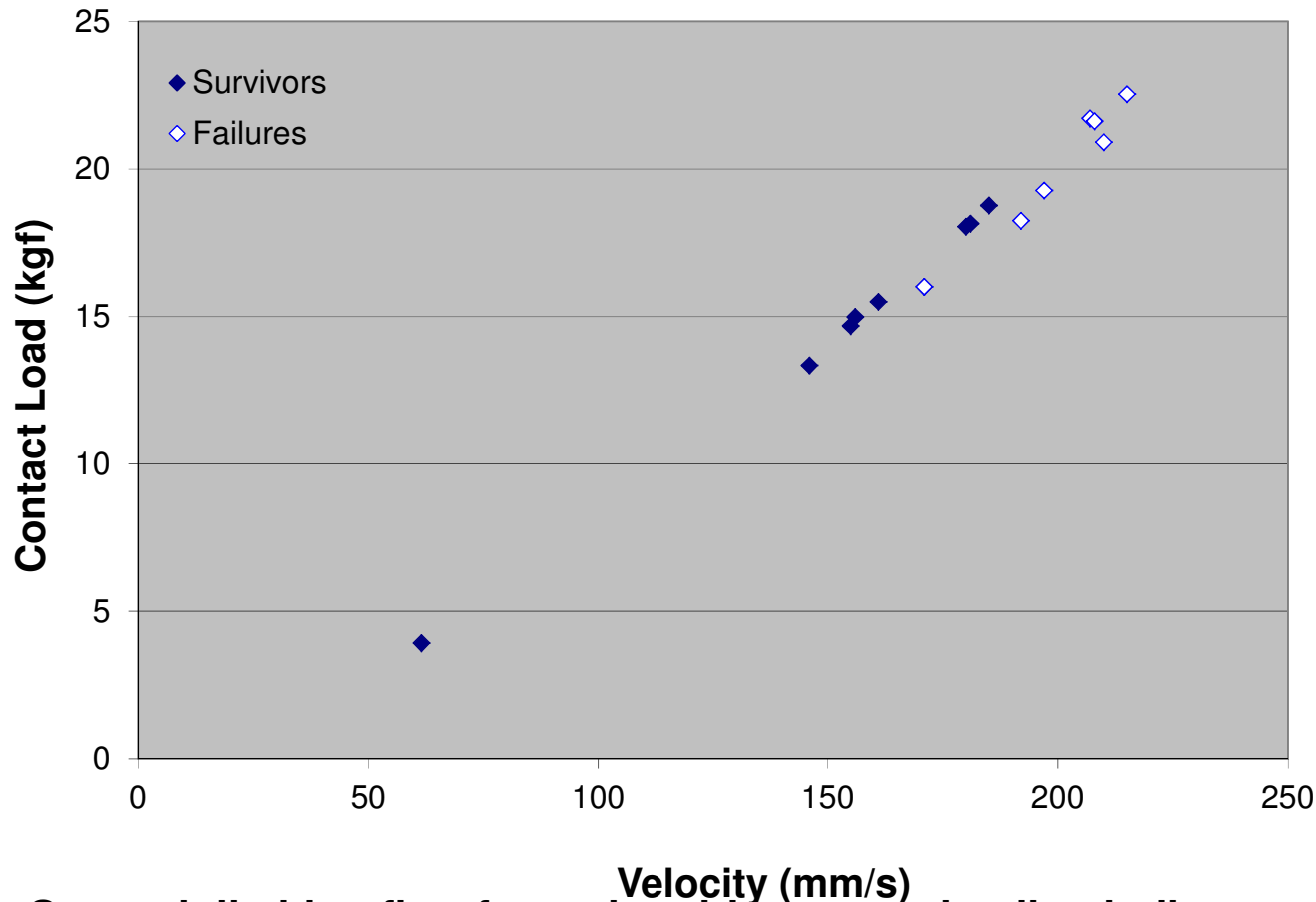
Stress-induced retardation profile comparison for alkali aluminosilicate indented using quasi-static and dynamic loading.

Less residual stress is an indicator of reduced amount shear deformation.



CS = 840 MPa DOL = 53 microns

## Dynamic Indentation with sharper 120° indenter tip

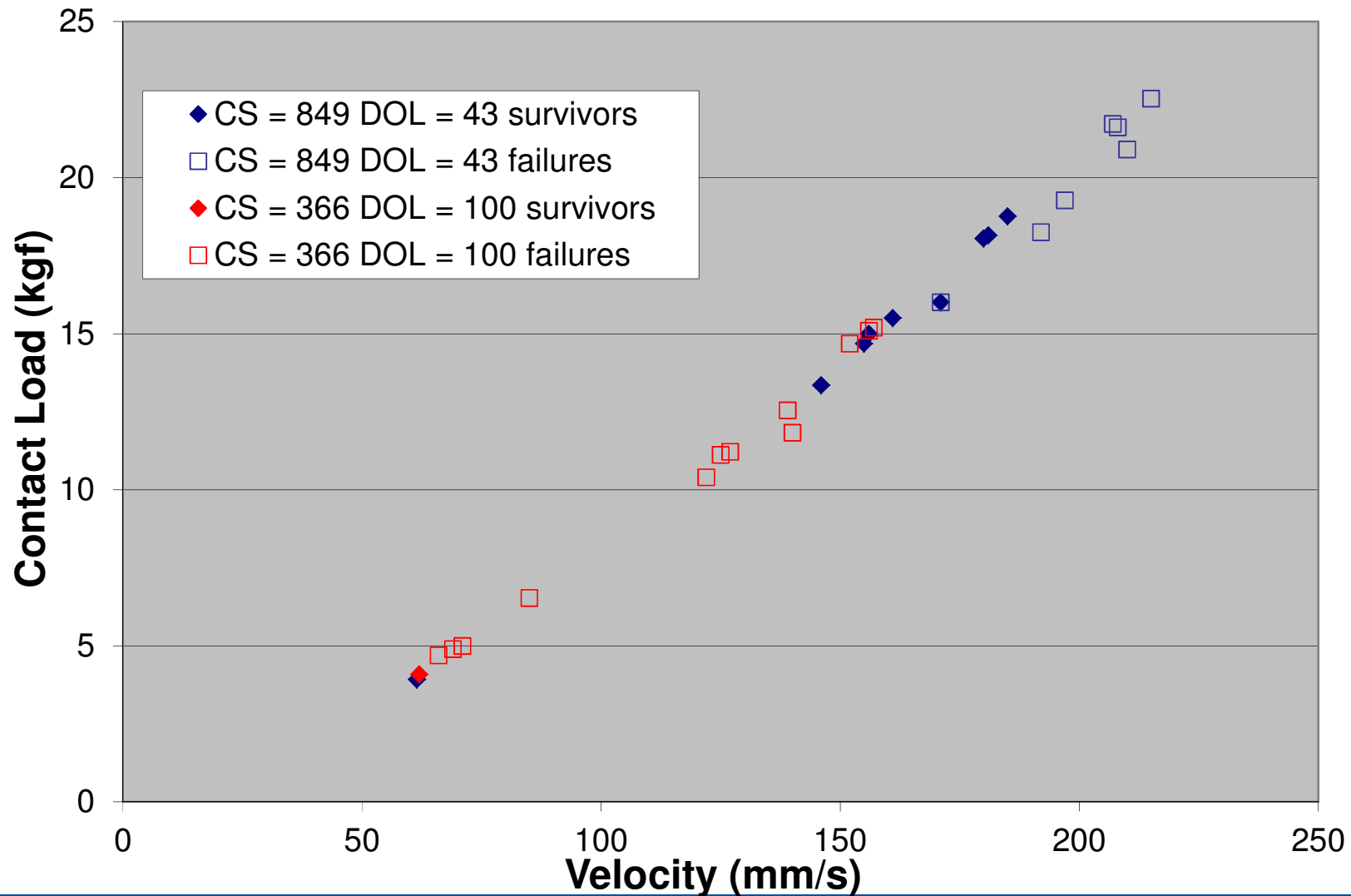


**Strength limiting flaw formation driven on unloading indicates subsurface damage coupled with higher residual stress in this load regime. Again, sharper contact promotes shear deformation.**

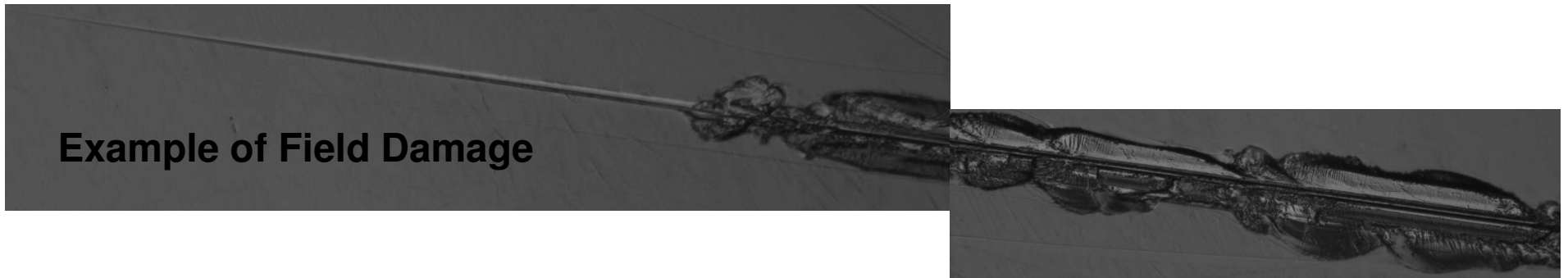
**Higher load rate still improves the median/radial cracking threshold drastically over quasi-static indentation.**

# Dynamic Indentation with sharper 120° indenter tip

## CS vs. DOL



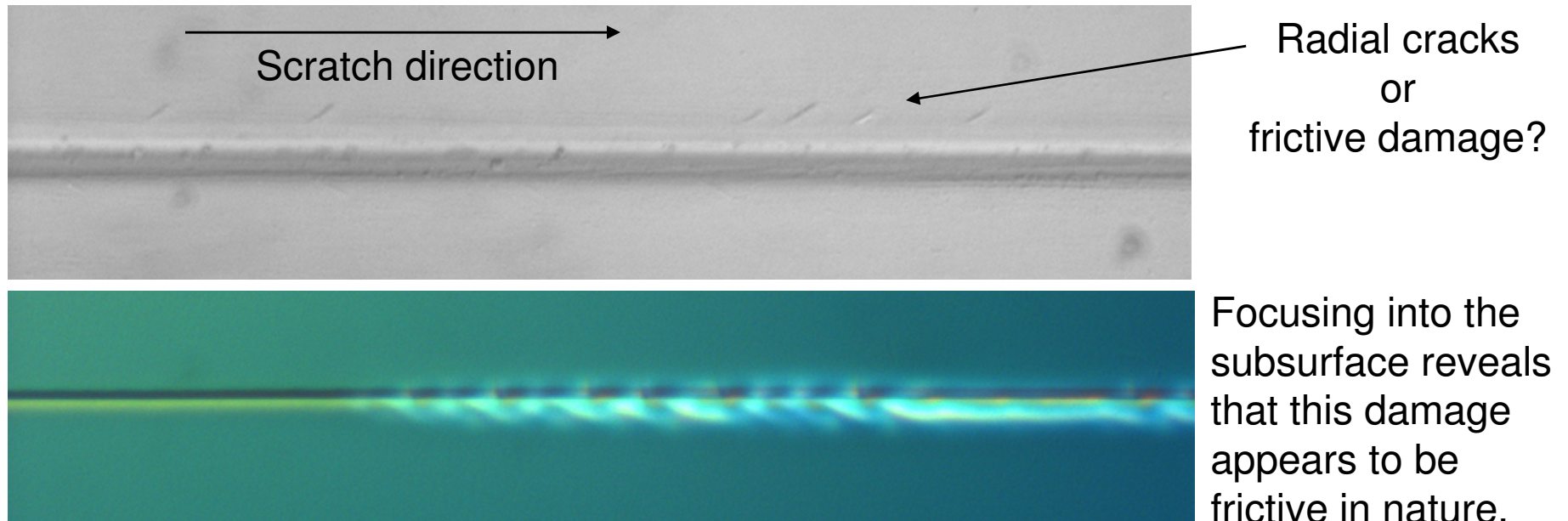
# Replicating sharp contact scratches in ion-exchanged glass



- I. Initially, plastic deformation occurs without the presence of cracks.
- II. Increased frictional forces cause minor frictive-type damage.
- III. Lateral cracking systems eventually cause highly visible chipping at the surface.



# Regime II damage in scratched glasses

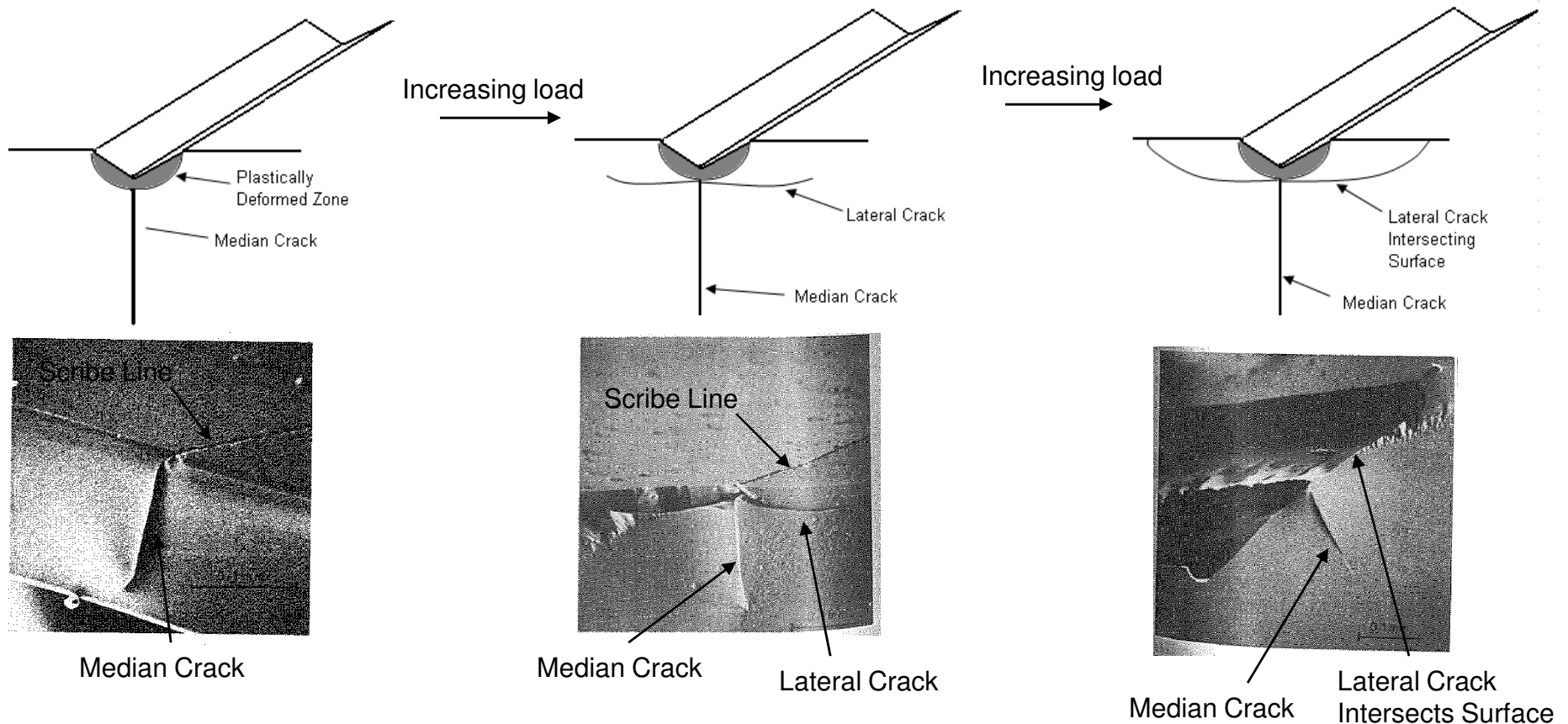


- Shallow cracks on either side of the scratch groove tend to form prior to the onset of larger median and lateral cracking systems.
- These have previously been described as radial cracks, but our measurements indicate that they are frictive since their presence depends on the surface quality of the glass being measured.
- Frictive cracks initiate at the surface from pre-existing flaws and extend into the subsurface to form crescent shaped cracks.
- At higher loads near-surface chipping connects these cracks.



# Subsurface look at scratch damage in non-ion-exchanged glass

Median crack formation occurs first and is followed by lateral cracking

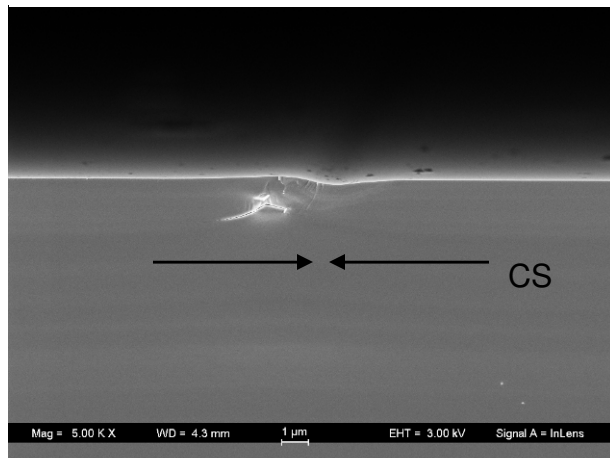


V.R. Howes and A. Szameitat, J. Mater. Sci. Letters 3 (1984) 872-874.

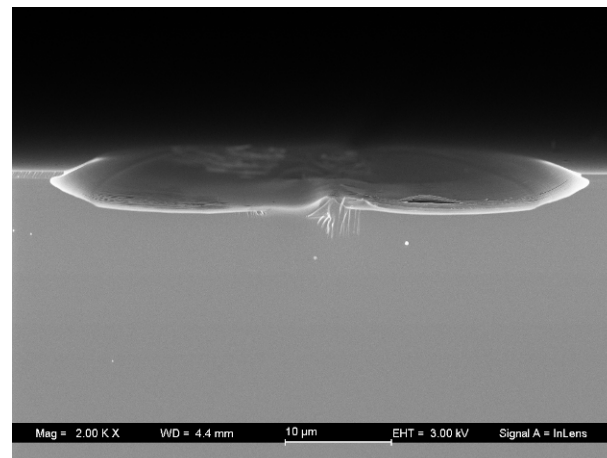
# Subsurface scratch damage in ion-exchanged glasses

In highly ion-exchanged glasses the lateral crack forms prior to the median crack

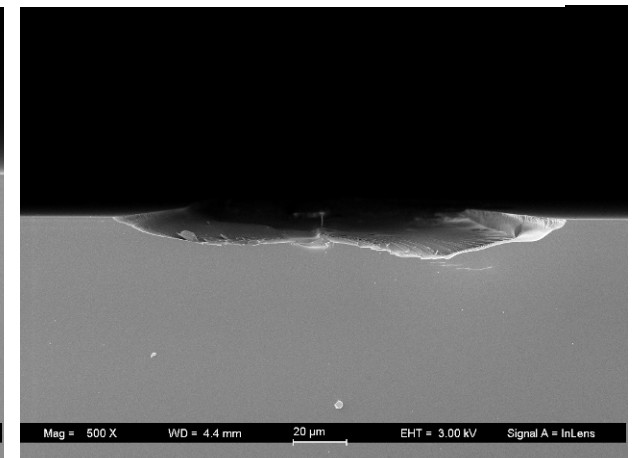
Ion-exchanged alkali aluminosilicate, Compressive stress = 770 MPa Depth of layer = 48 microns



Knoop Scratch at 0.5 N



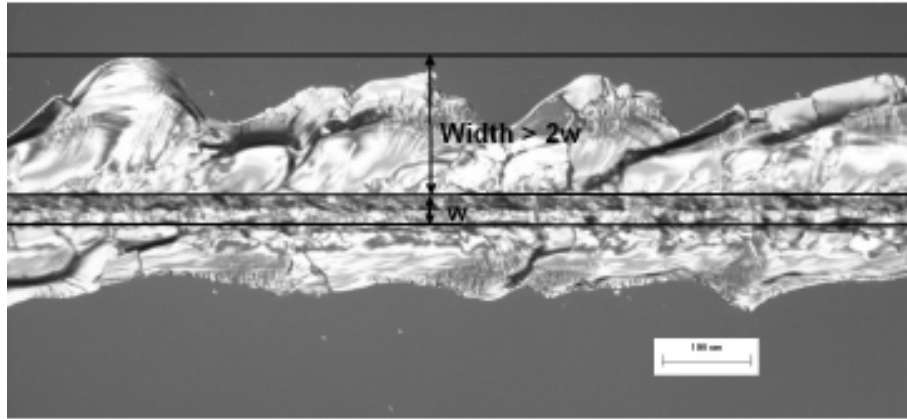
Knoop Scratch at 1 N



Knoop Scratch at 2 N

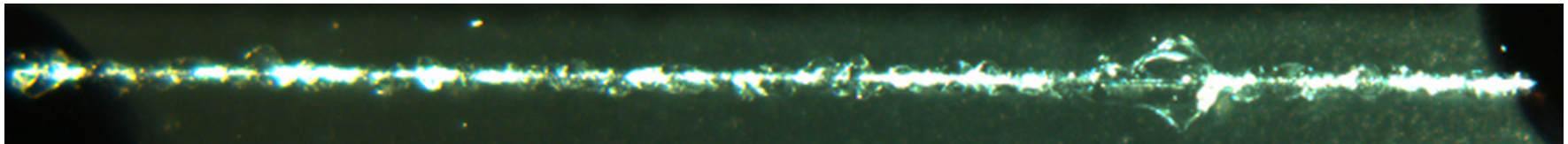
- The ion-exchange compressive stress prevents the formation of median cracks, but does not prevent lateral crack formation in the plane parallel to the glass surface.
- At substantially higher loads median cracks will form and the glass will separate through the thickness.
- The formation of lateral crack systems appears independent of compressive stress (CS) and depth of layer (DOL) as long as stress levels are high enough to suppress median crack as initial cracking system.
- The lateral cracks initiate from the subsurface damage in the deformation zone.

# Lateral cracking threshold criteria

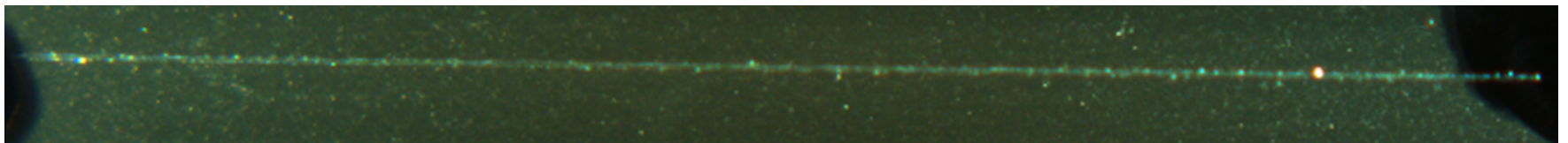


The lateral cracking threshold is defined in this work as the load which produces visual lateral cracks that extend a distance of twice the width of the scratch groove on either side.

10 mm long scratch with cracks exceeding lateral cracking threshold has high visual impact

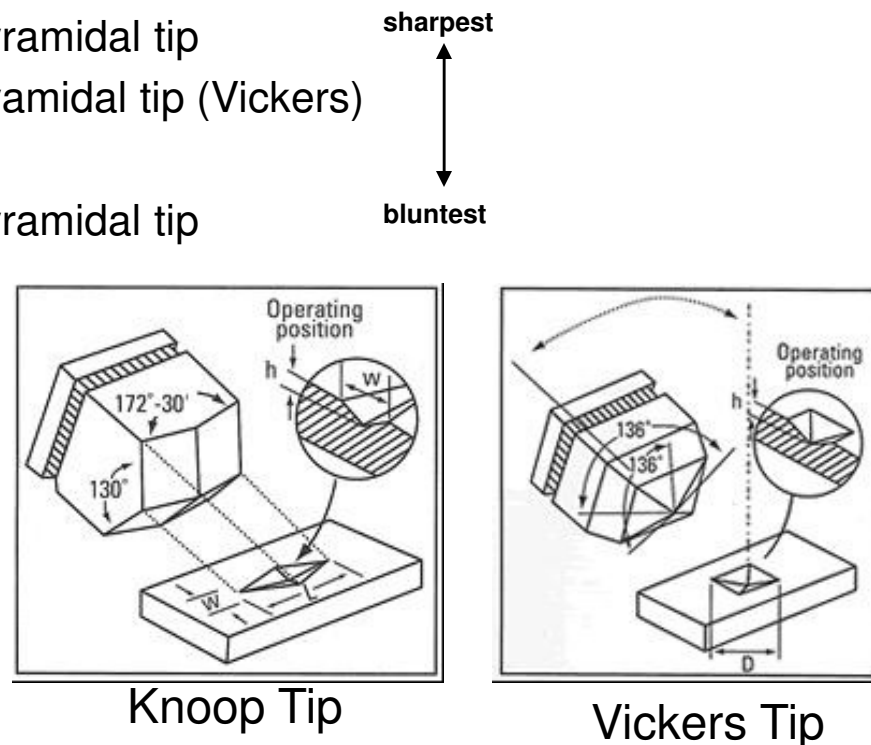


Pre-threshold scratches are difficult to see with the naked eye



# Scratch damage with various tips

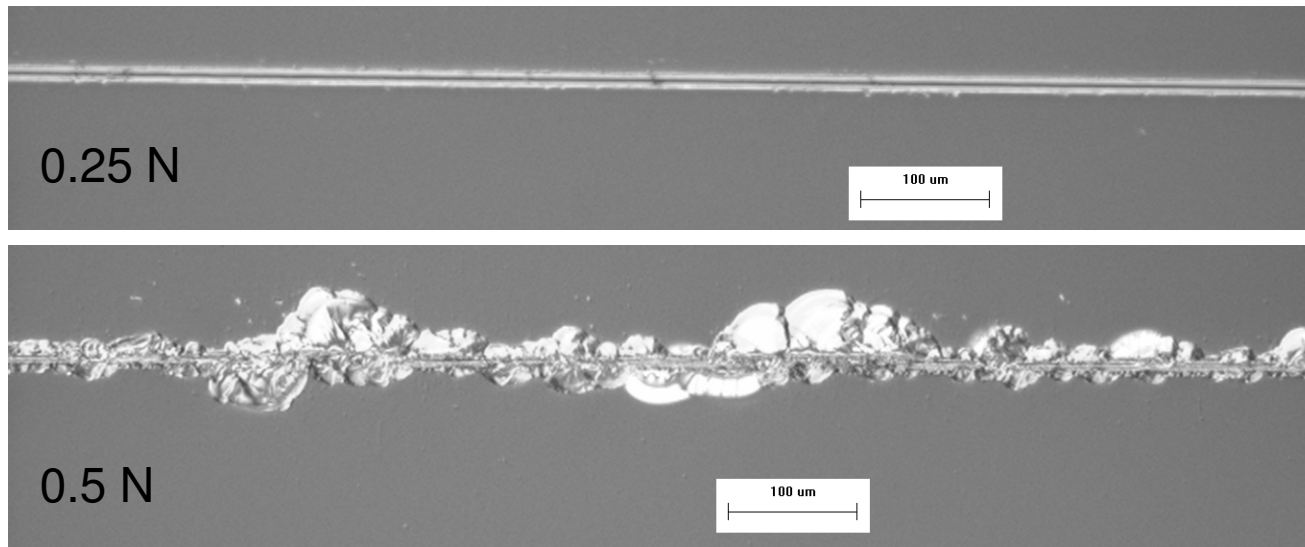
- The deformed region beneath an indent consists on material that has deformed by plastic flow (shear deformation) and by densification
- Again, as the indenter becomes sharper glass response tends more towards plastic flow and the crack initiation load decreases.
- To demonstrate the effect of tip geometry the scratch test is performed on ion-exchanged alkali aluminosilicate (CS = 770 MPa and DOL = 48 microns) with the following tips:
  - 120° 4-sided pyramidal tip
  - 136° 4-sided pyramidal tip (Vickers)
  - Knoop tip
  - 150° 4-sided pyramidal tip





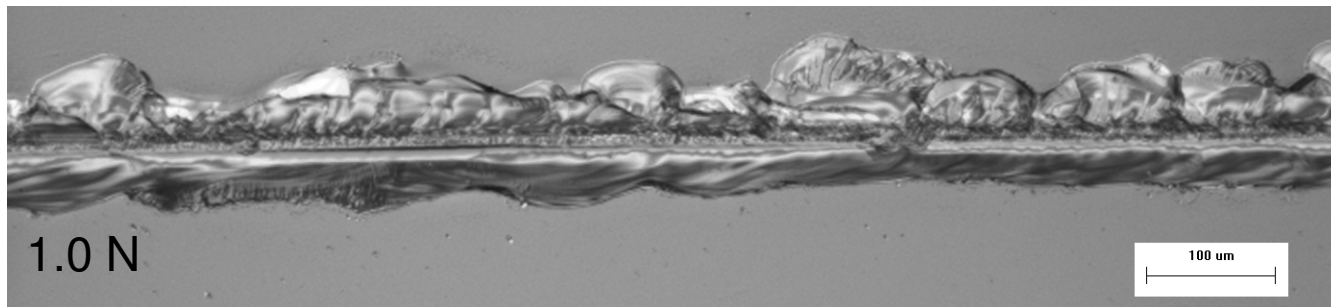
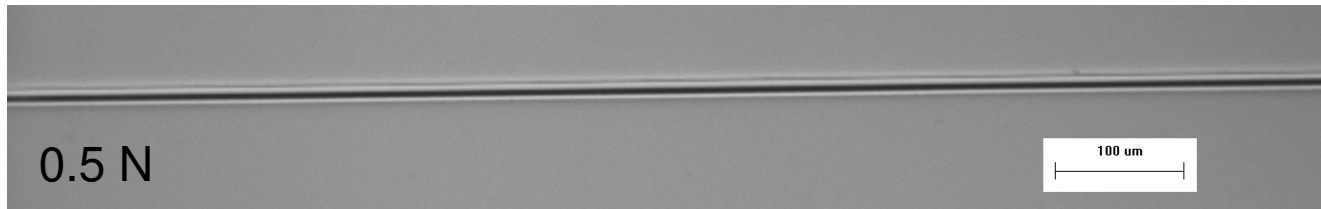
# Scratches in ion-exchanged alkali aluminosilicate with 120° 4-sided pyramidal tip (sharpest tip)

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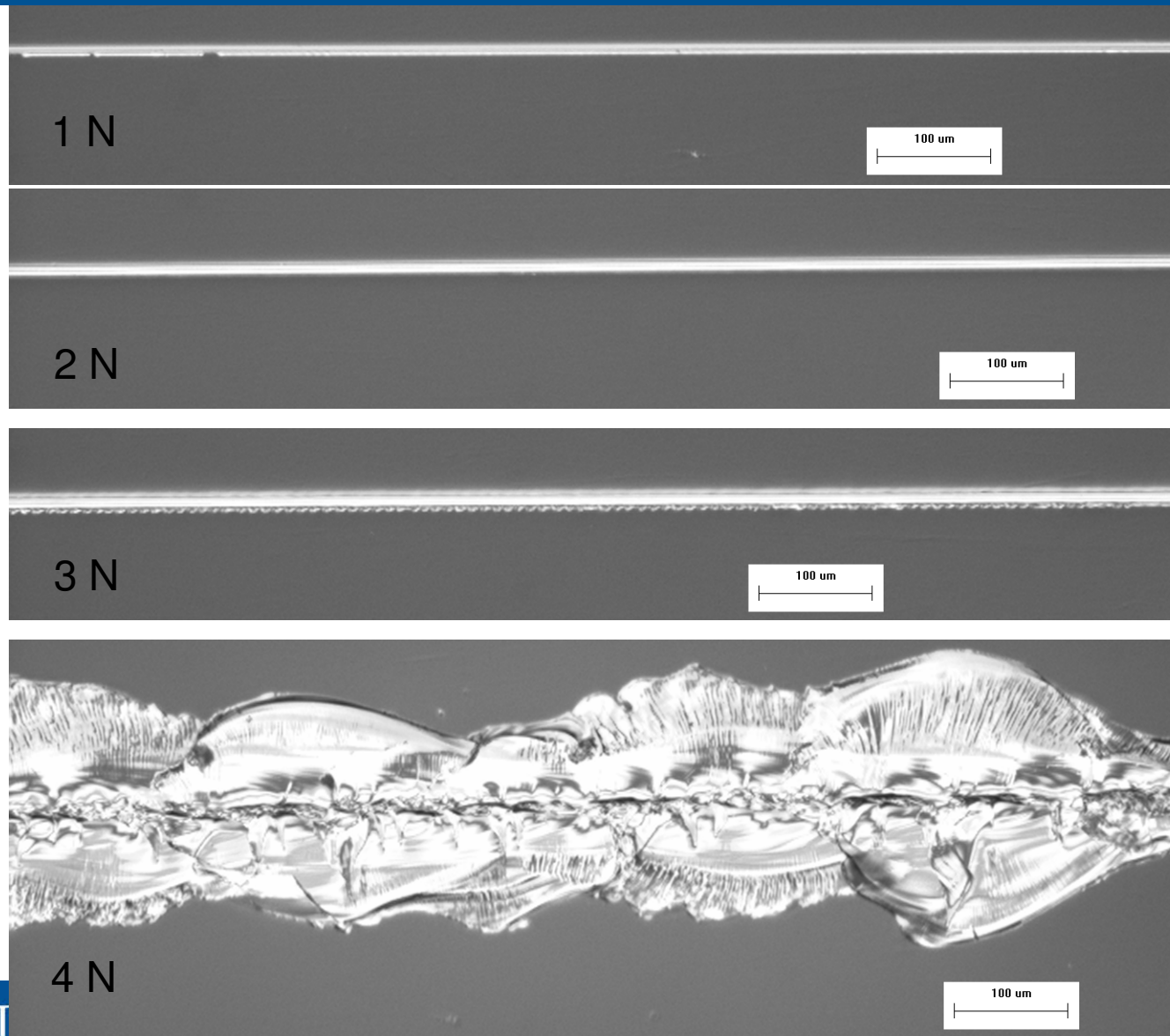


# Scratches in ion-exchanged alkali aluminosilicate with Vickers (136°) tip

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# Scratches in ion-exchanged alkali aluminosilicate with Knoop tip



# Scratches in ion-exchanged alkali aluminosilicate with 150° 4-sided pyramidal tip (least sharp tip)

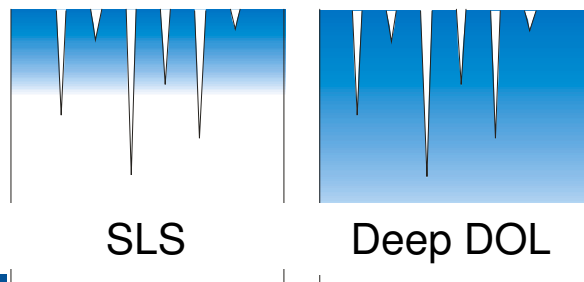




# Retained strength

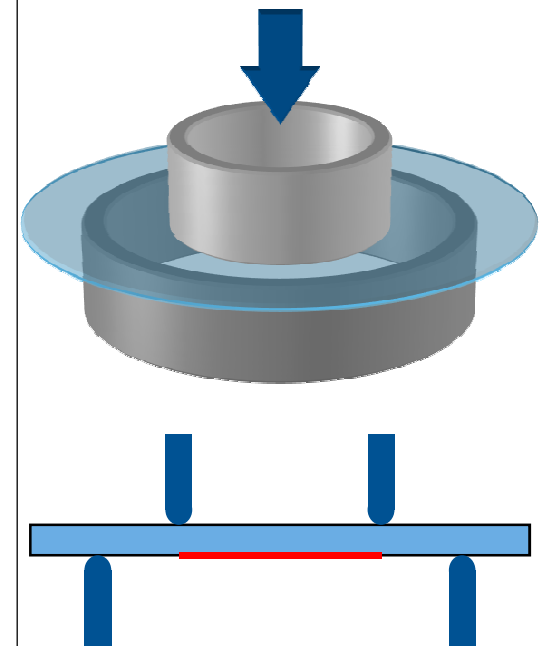
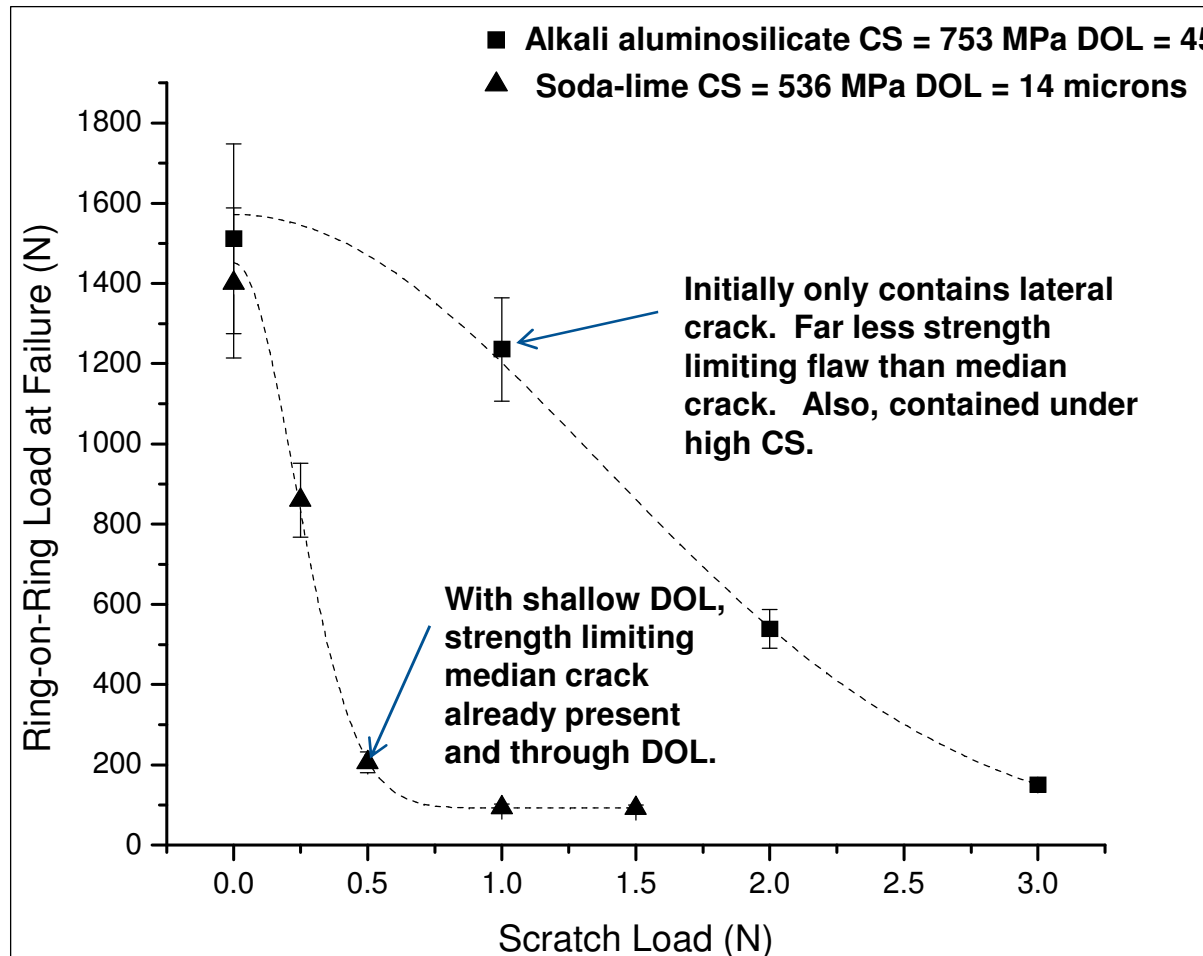
- The measurement of ring-on-ring strength before and after introduction of controlled damage can be a useful tool to quantify and understand flaw introduction and its impact on mechanical performance
- Controlled flaws can be introduced as scratches, indentation damage, or as grit blast abrasion
- This approach provides a convenient means to simulate field failures in a controlled manner.
- If flaws enveloped in compression, want high CS.

Flaw populations on the surface



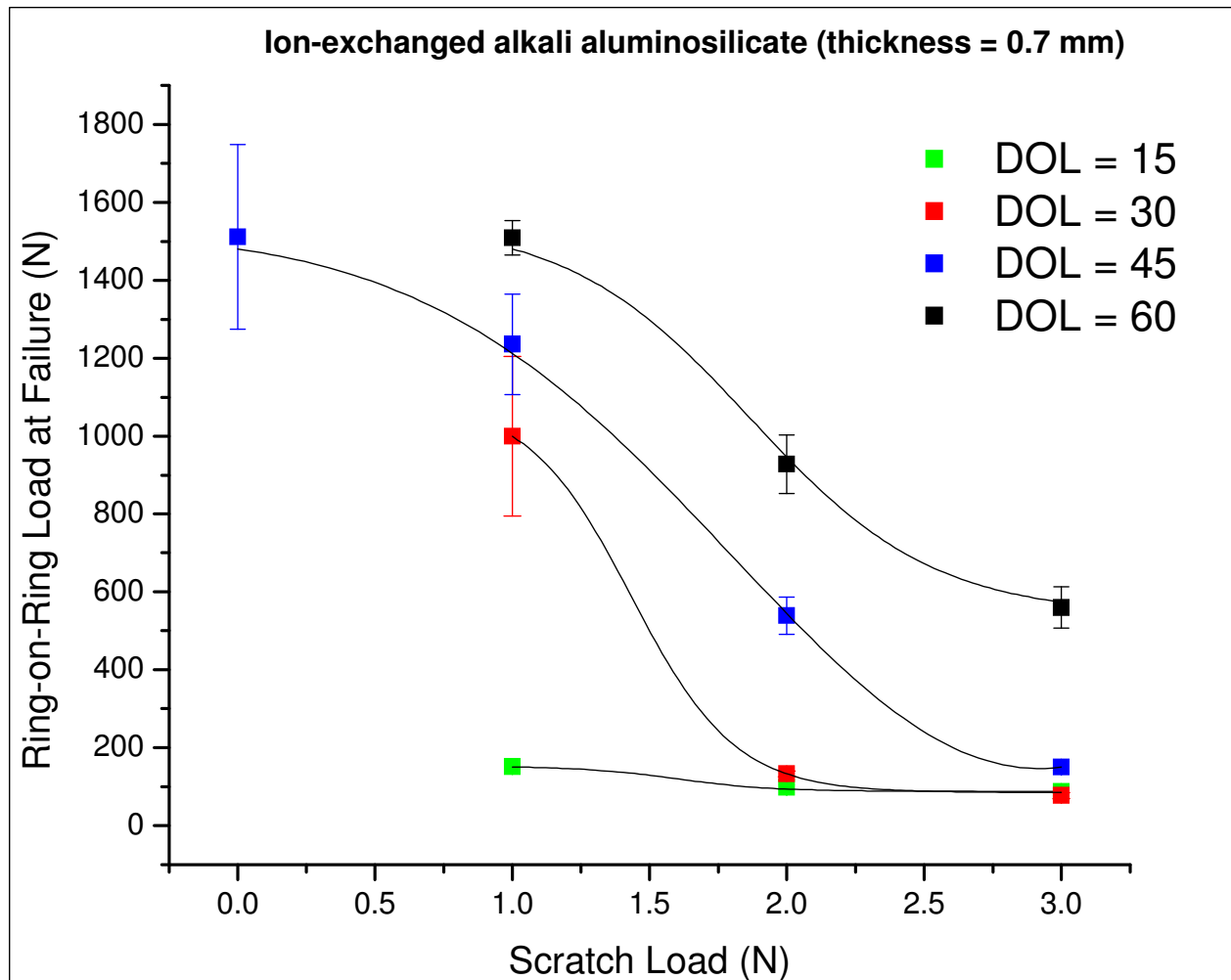
# Ring-on-ring testing of Vickers scratches

Comparison of Ion-Exchanged Glass with Thickness = 0.7 mm



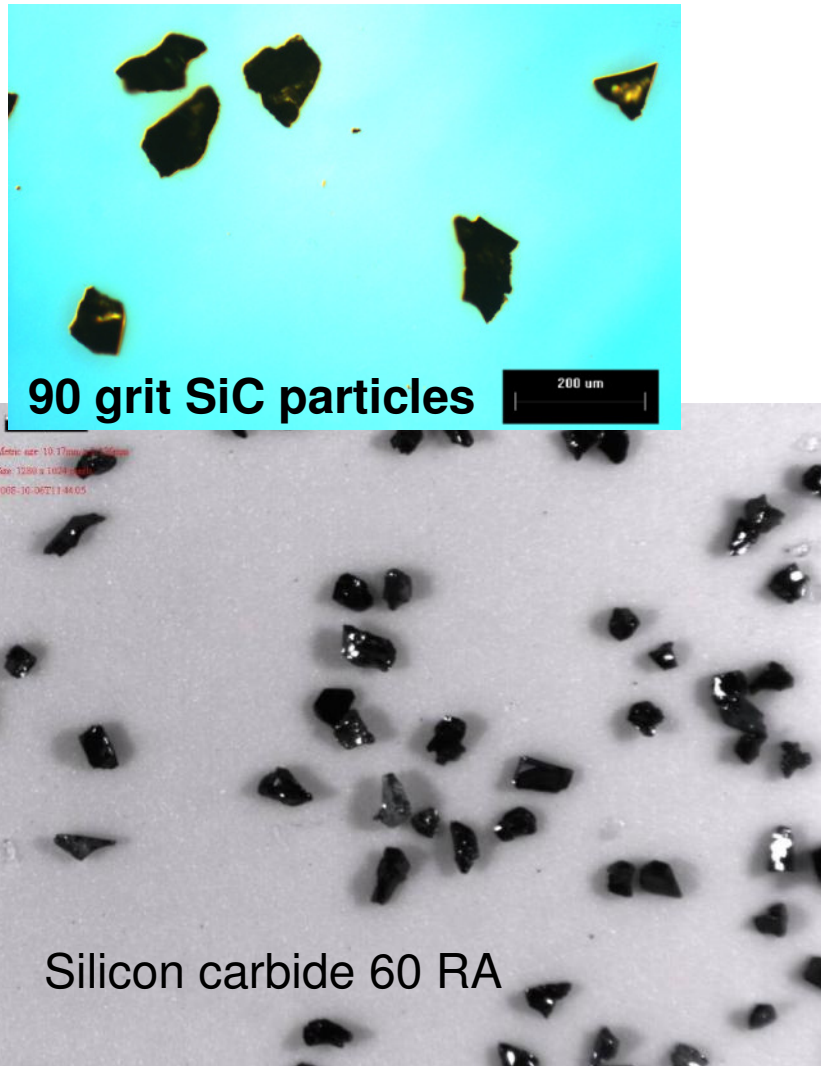
Scratch is fully within loading ring on bottom of ROR specimen. Load to strength conversion is non-linear.

# Scratch ring-on-ring of IXed alkali aluminosilicate at various DOL

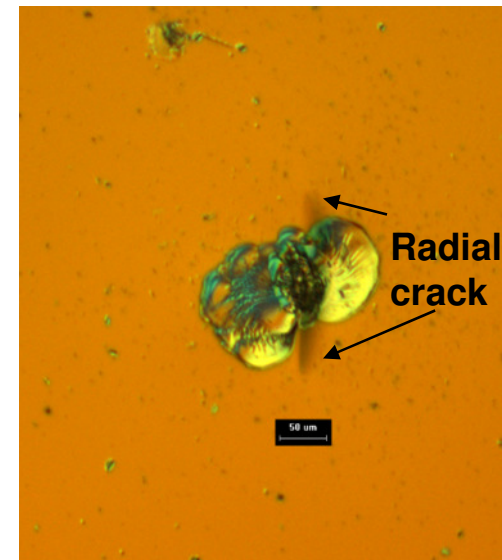
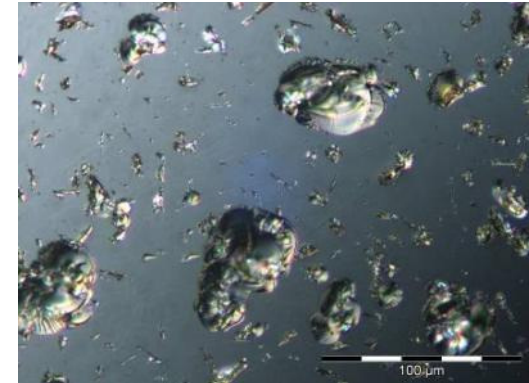


CS values held nearly constant

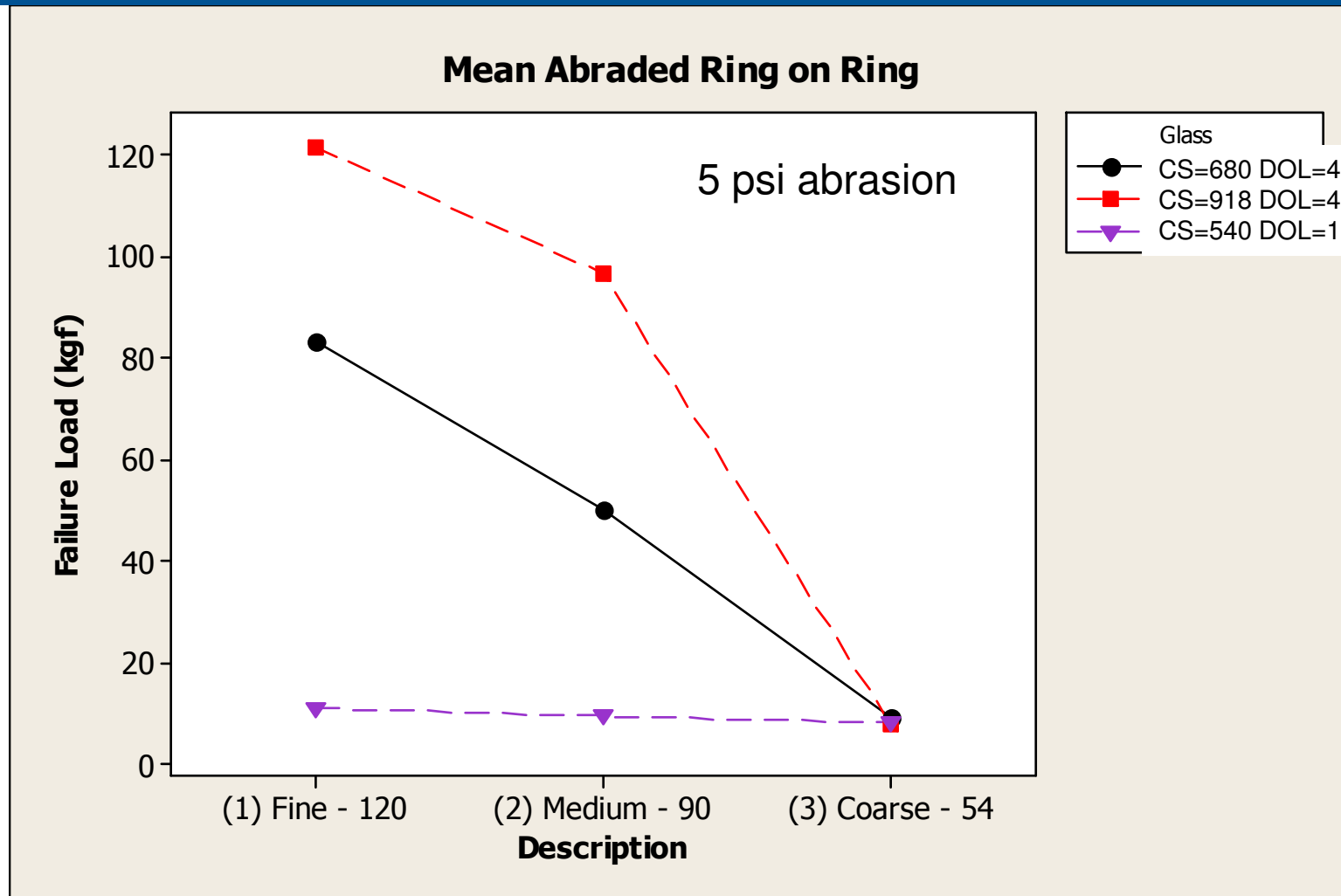
SiC abrasion causes radial/median cracks to form in both soda-lime and alkali aluminosilicate glasses. Irregular shapes of particles cause damage from a wide range of contact geometries.



Damage from SiC abrasion

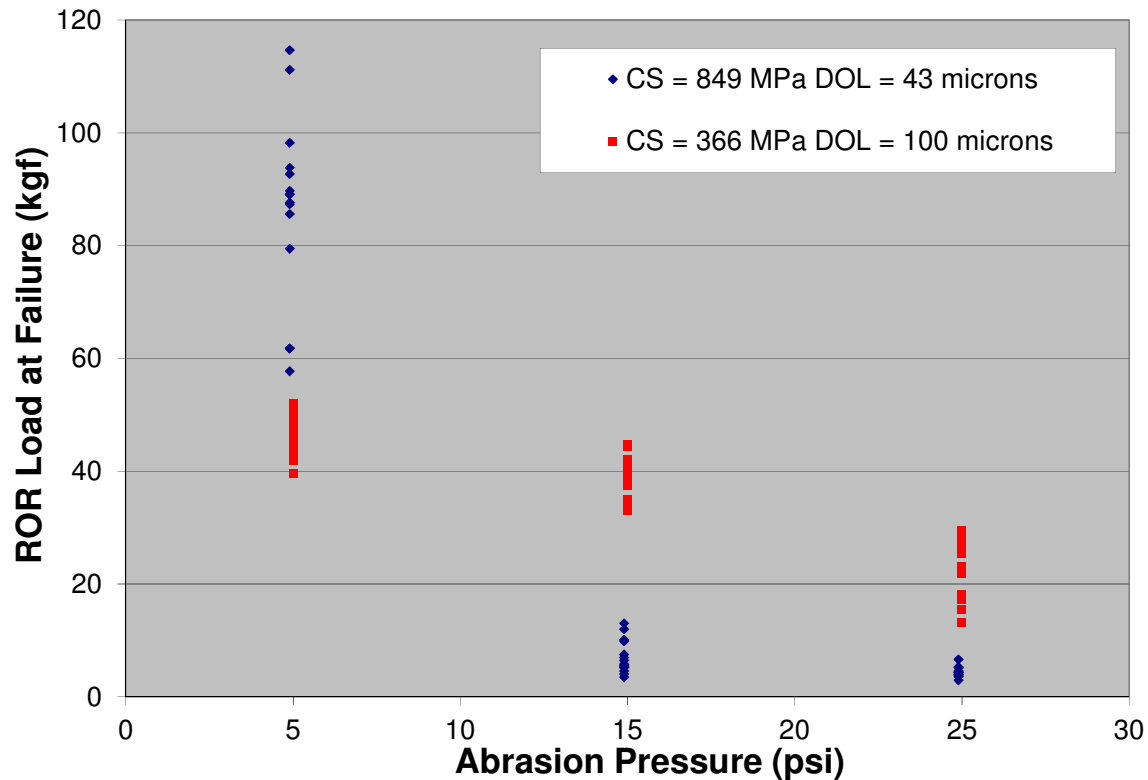


# Abraded ROR with fine, medium, and coarse SiC



If DOL contains flaws, then additional compressive stress improves failure load.

## 90 Grit SiC Abrasion at various pressures on 0.6 mm alkali aluminosilicate



- Strength limiting flaws are generated at each abrasion pressure used.
- If DOL contains the flaw, the retained load at failure will increase with compressive stress to the flaw depth.
- Low CS, deep DOL parts can contain very deep flaws, however cannot achieve high strength even at shallow flaw depths. Non-abraded, low CS samples are also considerably weaker than high CS samples when testing samples similarly handled.

# Summary

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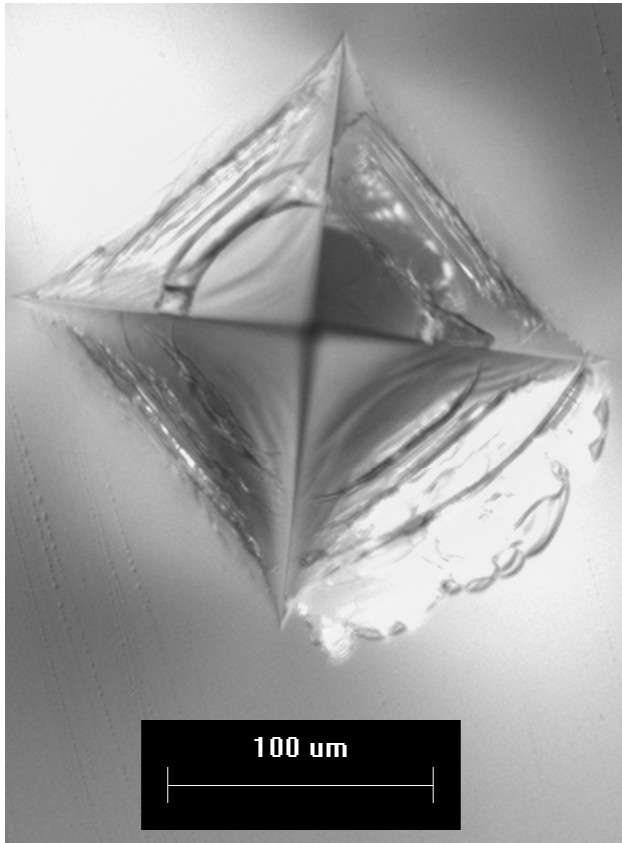
- The formation of strength limiting flaws by sharp contact depends on both the contact geometry and the rate of contact.
  - Sharper indenters promote shear deformation, blunter indenters promote densification
  - Quasi-static indentation promotes shear deformation, dynamic indentation promotes densification
- The resistance to the formation of strength limiting flaws is increased with higher compressive stress.
- Cosmetic damage in the form of highly visible lateral crack-containing scratches is also highly dependent on contact geometry.
- The retained strength following sharp contact can be measured using controlled damage introduction following by ROR. Key to retained strength is enveloping the flaw within the DOL and under as high stress as possible (within safe limits of frangibility).

# ADDITIONAL SLIDES

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# Indentation in N<sub>2</sub>

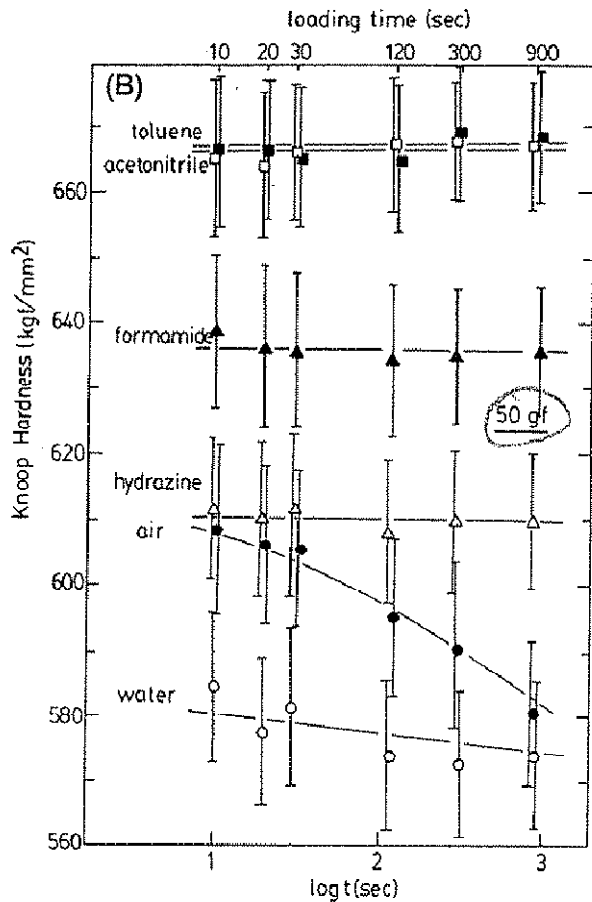


Indentation in IXed  
alkali aluminosilicate in  
dry N<sub>2</sub> atmosphere at  
15 kgf

- Indentation N<sub>2</sub> glovebag increases indentation threshold 2X.
- Water was removed from air, but adsorbed water on glass and diamond surfaces was not removed.
- High speed indentation prevents is expected to prevent sufficient water diffusion into glass.

# Water diffusion into glass during indentation?

Hardness vs. loading duration  
in various environments



Evidence of Water entry into glass  
during indentation

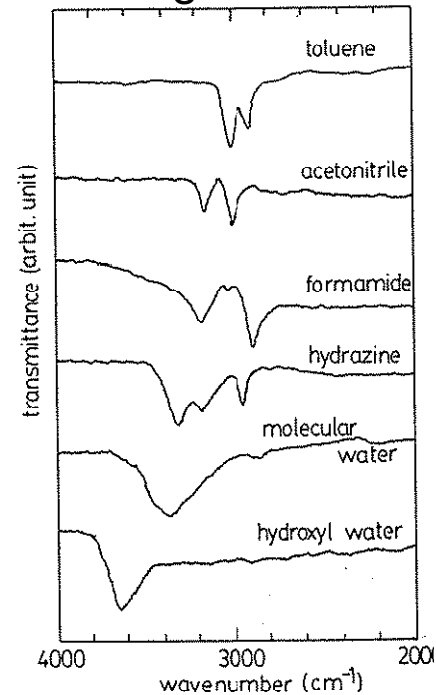


Fig. 4. Infrared transmittance spectra of various liquids.

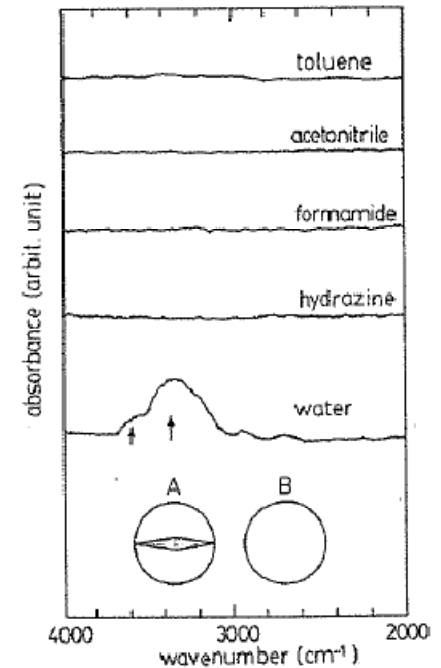


Fig. 5. Infrared absorbance spectra of indentation mark using a test load of 100 gf and loading duration of 30 s. Subtraction spectra (indented mark (A) minus nonindented mark (B)) are shown. Arrows indicate the positions of hydroxyl and molecular water absorption bands.

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# Water diffusion into glass has also been shown to reduce indentation crack initiation load

Crack initiation load vs. loading duration in different environments

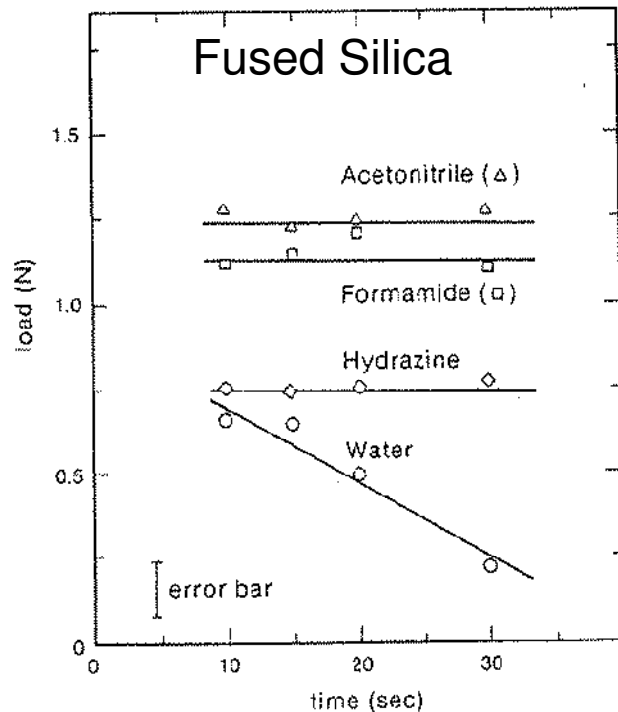


Fig. 1. Crack initiation load of silica glass in various liquids under a Vickers indenter as a function of loading duration. The crack initiation load is defined as the load which produced radial cracks, on an average, 50% of the time (i.e., at two corners out of four).

- Water diffuses into glass occurs in both air and water environments.
- The water uptake is dependent on contact time.
- Contact time for dynamic indentation ranges from 200 to 600 microseconds.
- Contact time for quasi-static indentation is ~30s. Around 100,000 times longer than dynamic indentation.

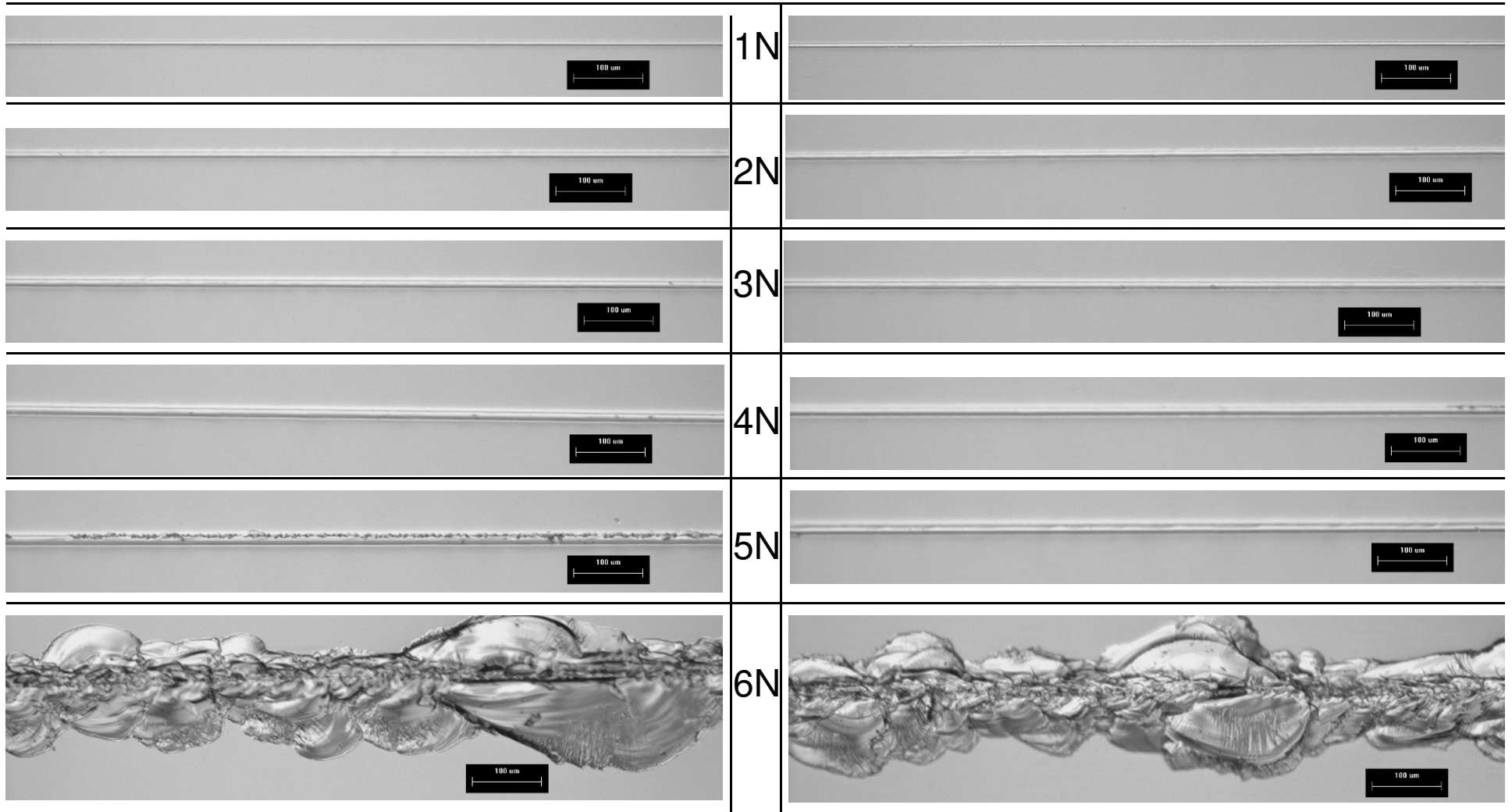
K. Hirao and M. Tomozawa J. Am. Ceram. Soc. **70** 497-502 (1987)

Some examples showing that lateral cracking threshold is independent of Ion-Exchange Levels

Constant Load Knoop Scratches in Glass A

CS = 861 MPa DOL = 29 microns

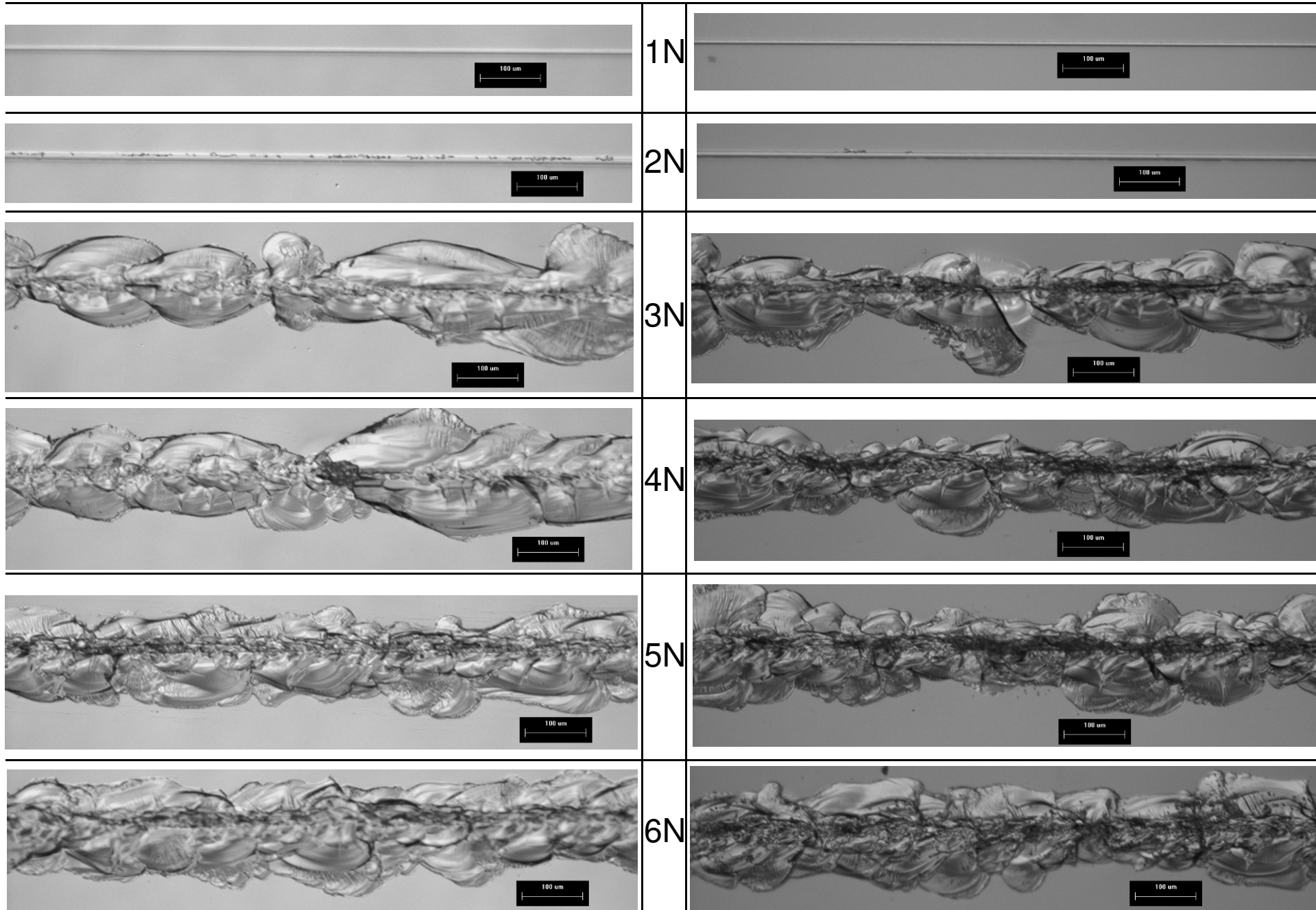
CS = 772 MPa DOL = 41 microns



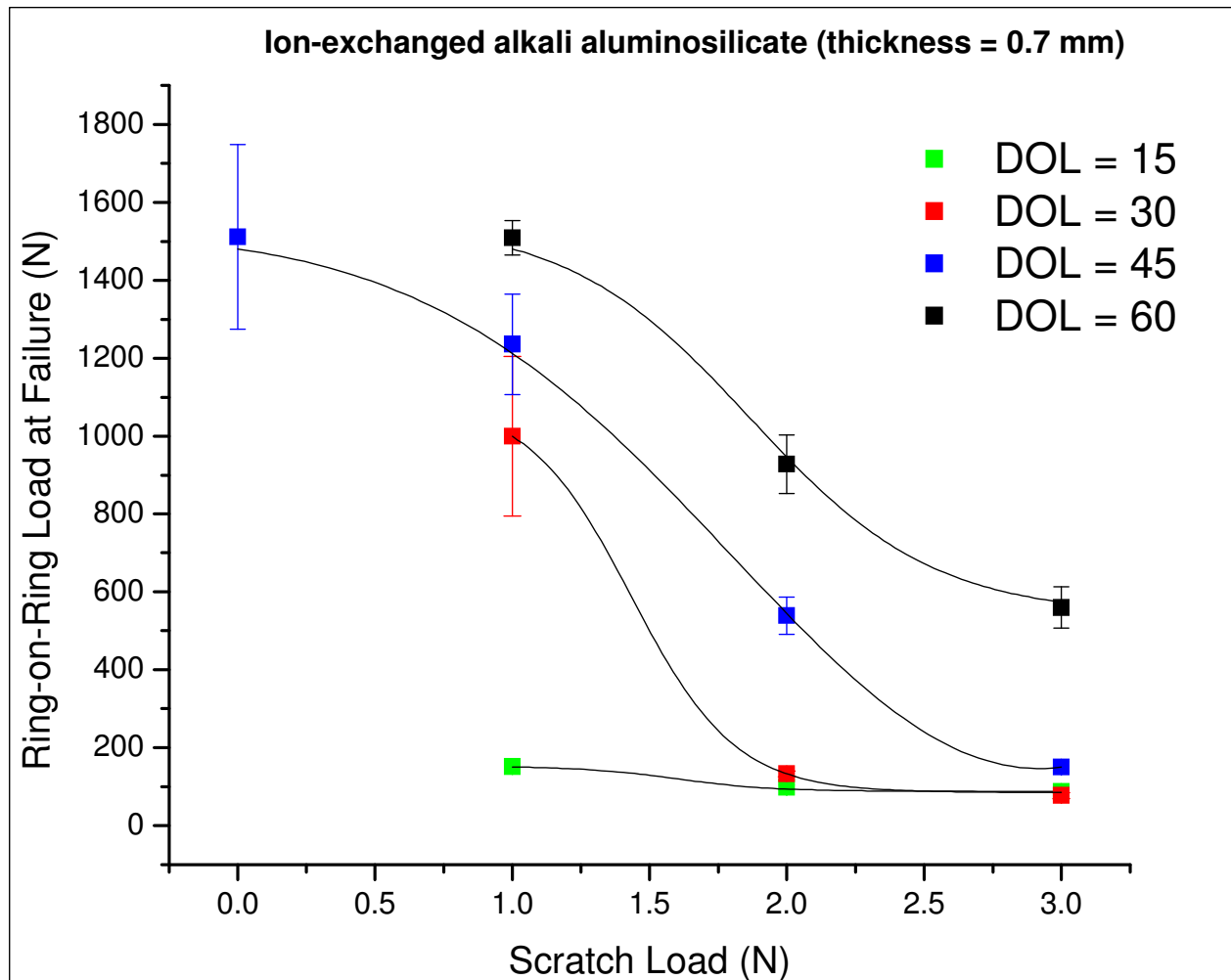
# Constant Load Knoop Scratches in Glass B

CS = 750 MPa DOL = 33 microns

CS = 630 MPa DOL = 52 microns



# Scratch ring-on-ring of IXed alkali aluminosilicate at various DOL



CS = 792 MPa  
CS = 785 MPa  
CS = 753 MPa  
CS = 722 MPa