CORNING

Sharp Contact Damage in Ion-Exchanged Cover Glass

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Functional Glasses: Properties and Applications for Energy and Information
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Overview

- 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


Origin

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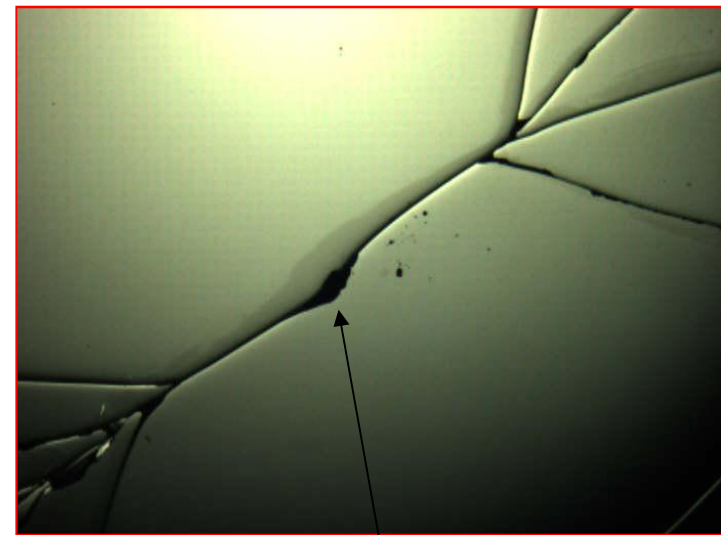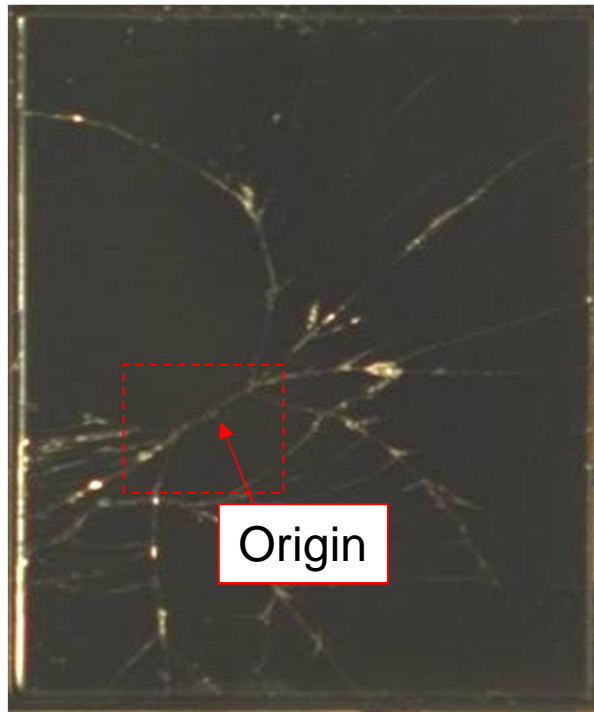
- Sharp contact deformation is defined by the glass response. It occurs when the contact load is distributed over small contact area and elastic limit is exceeded resulting in permanent deformation.
- Strength limiting flaw formation initiates within the permanent deformation region.
- Crack extension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

Using diamond indentation to mimic the response from sharp contact
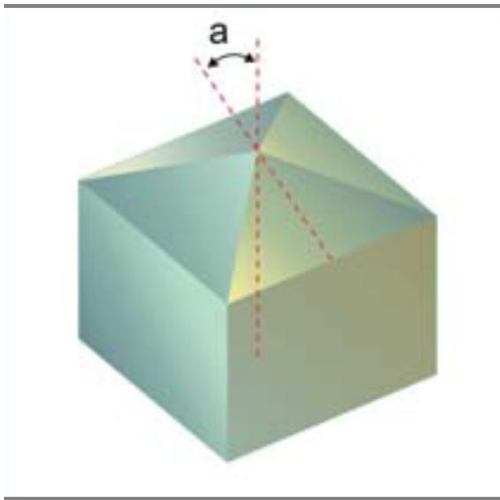
The Vickers diamond indenter is a 4-sided pyramid with angle between opposite faces 2a = 136°

Loading   Unloading

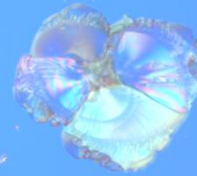Median crack   Lateral crack

(B) Radial
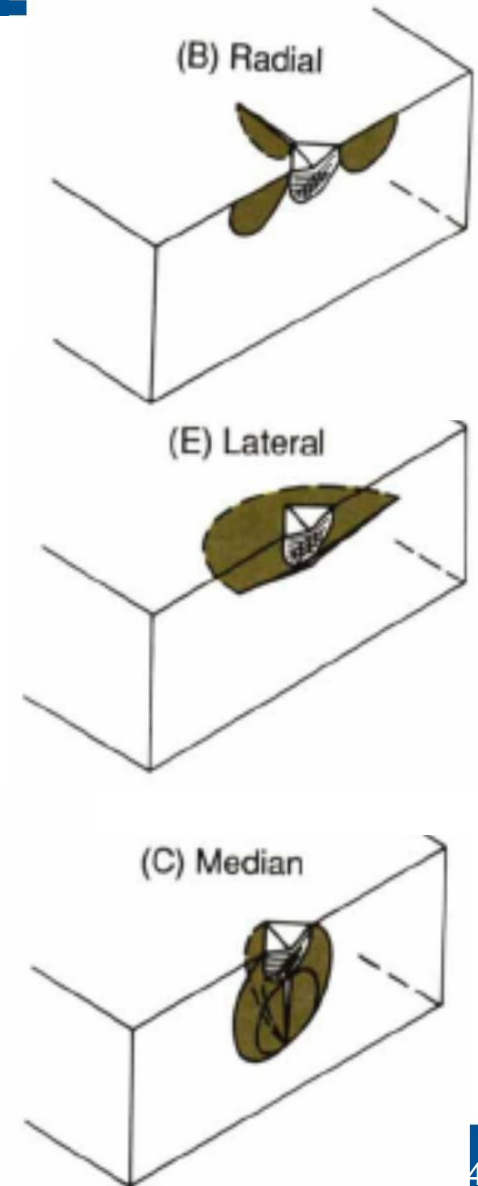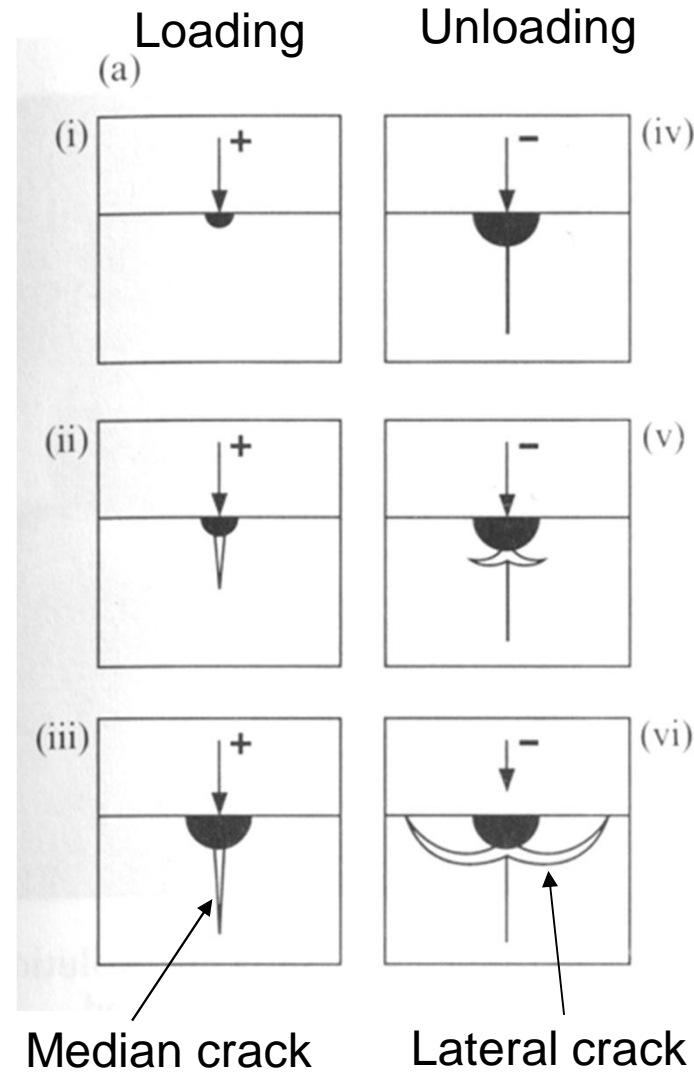
(E) Lateral

(C) Median

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


Sharp Contact Field Damage

200 µm
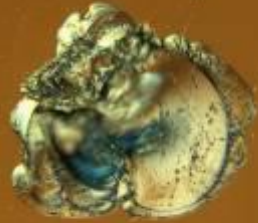
Damage produced by Vickers indenter

200 µm

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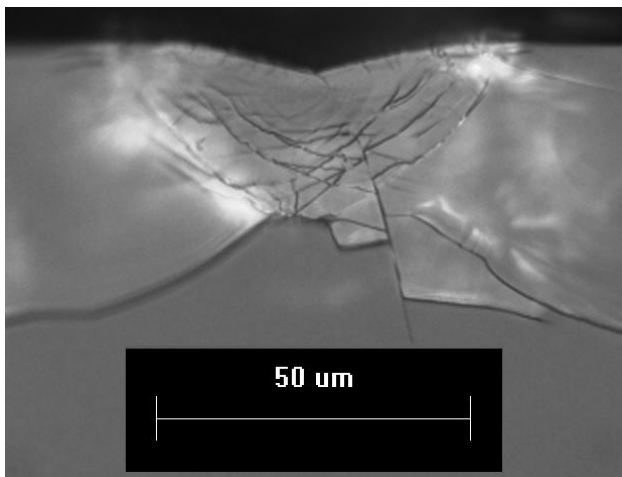
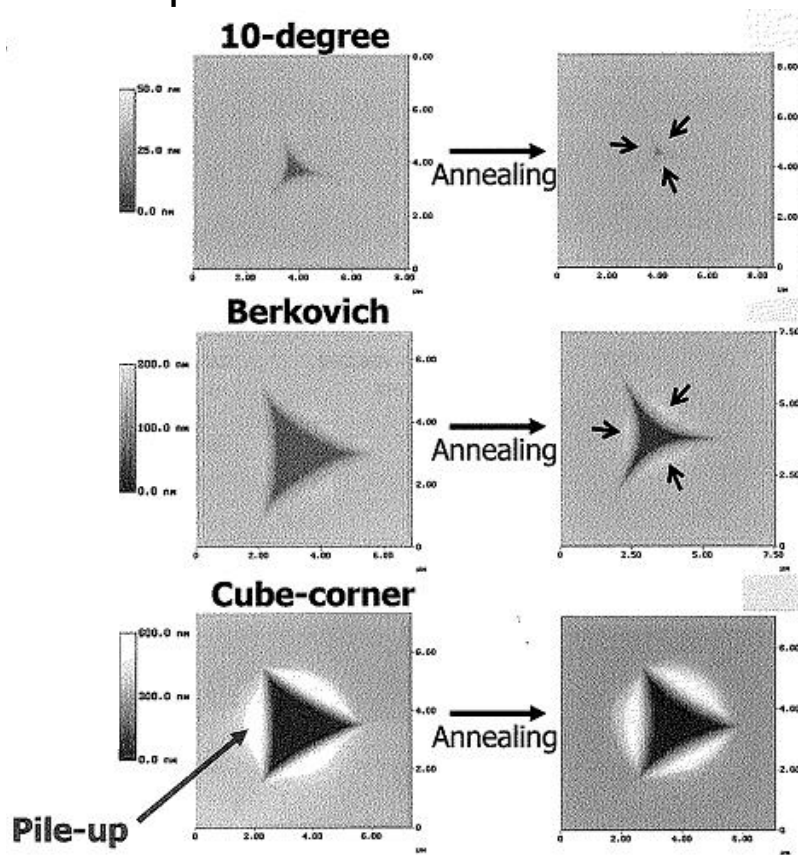Cross-section of 1 kgf Vickers indentation in non-ion-exchanged normal glass
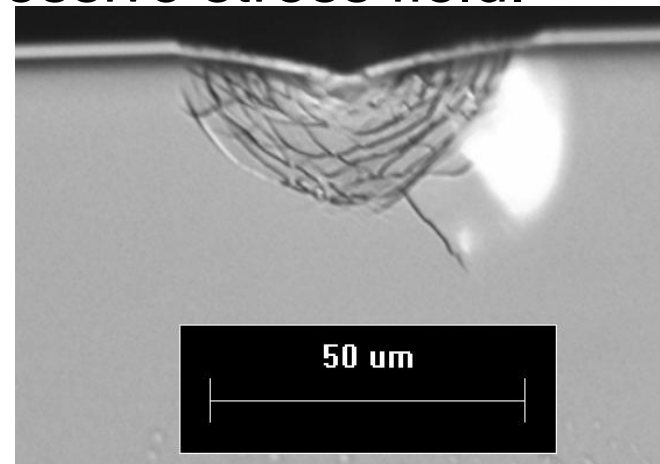
Cross-section of 1 kgf Vickers indentation in ion-exchanged normal glass

Deformation mechanisms

- 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

shear bands
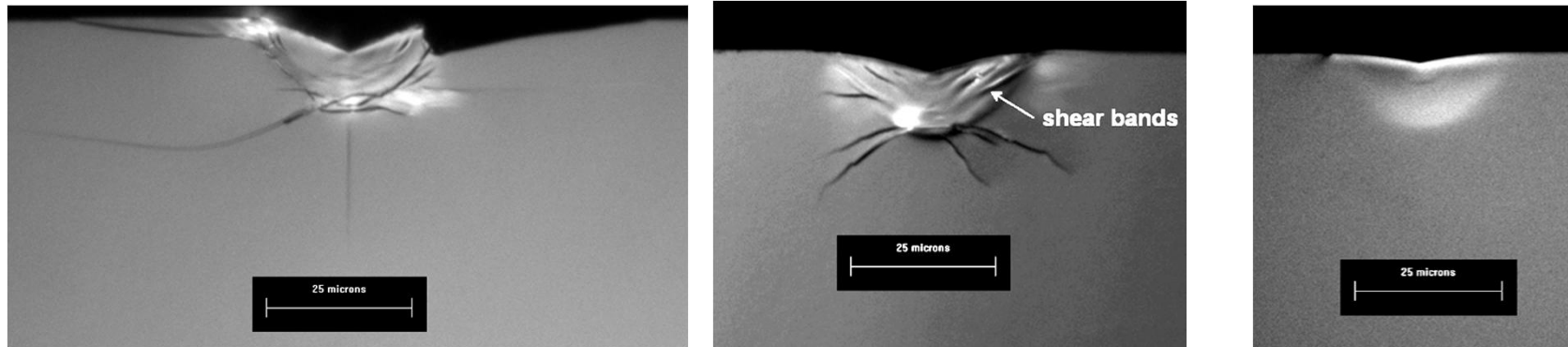
150° tip

Sharper, more shear ← → Blunter, more densification

T.M. Gross, ACerS Fractography VI Ceramic Transactions Vol. 320 (2012) 113-122.

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

120° tip at 30 gf

Vickers 136° tip at 500 gf

15 microns

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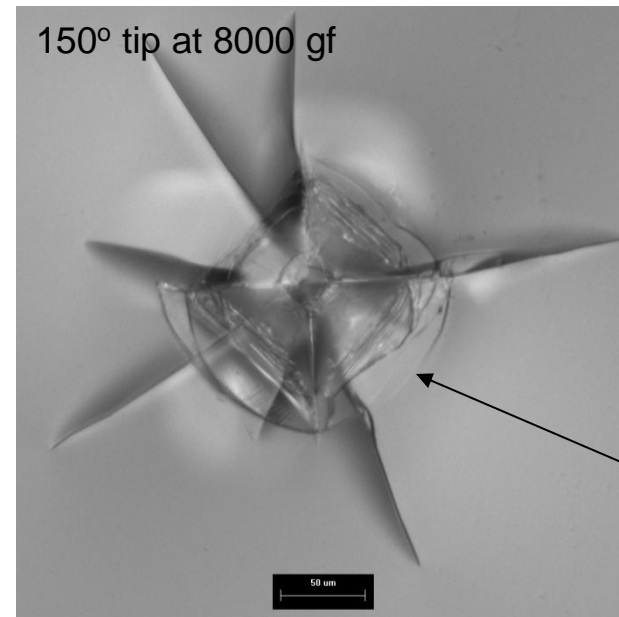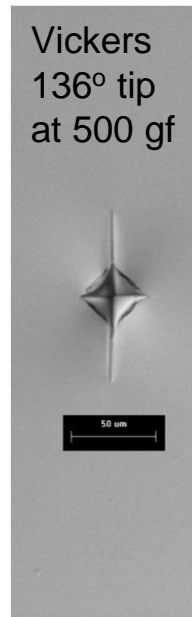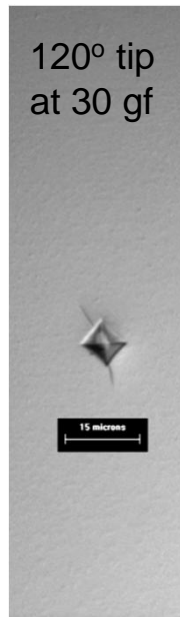
Normal sharp cracking behavior indicates that deformation occurs with significant shear deformation

150° tip at 8000 gf

Ring crack

50 um

Mixed normal/anomalous cracking behavior indicates that deformation is occurring with greater densification
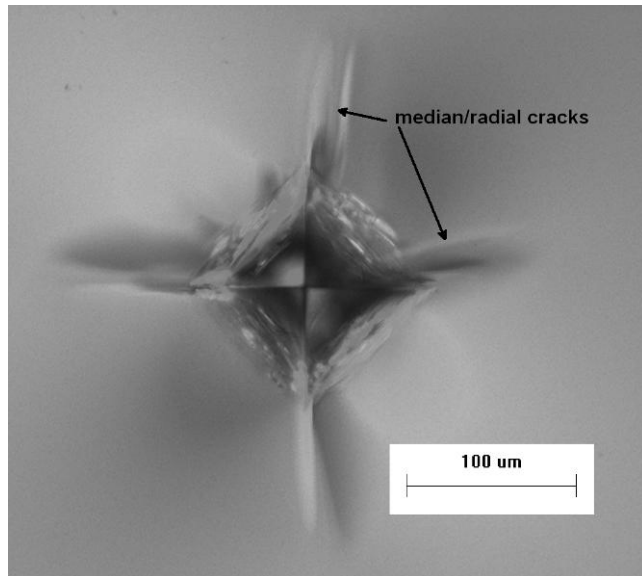
Resistance to the formation of cracks increases as the deformation mechanism tends towards densification

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

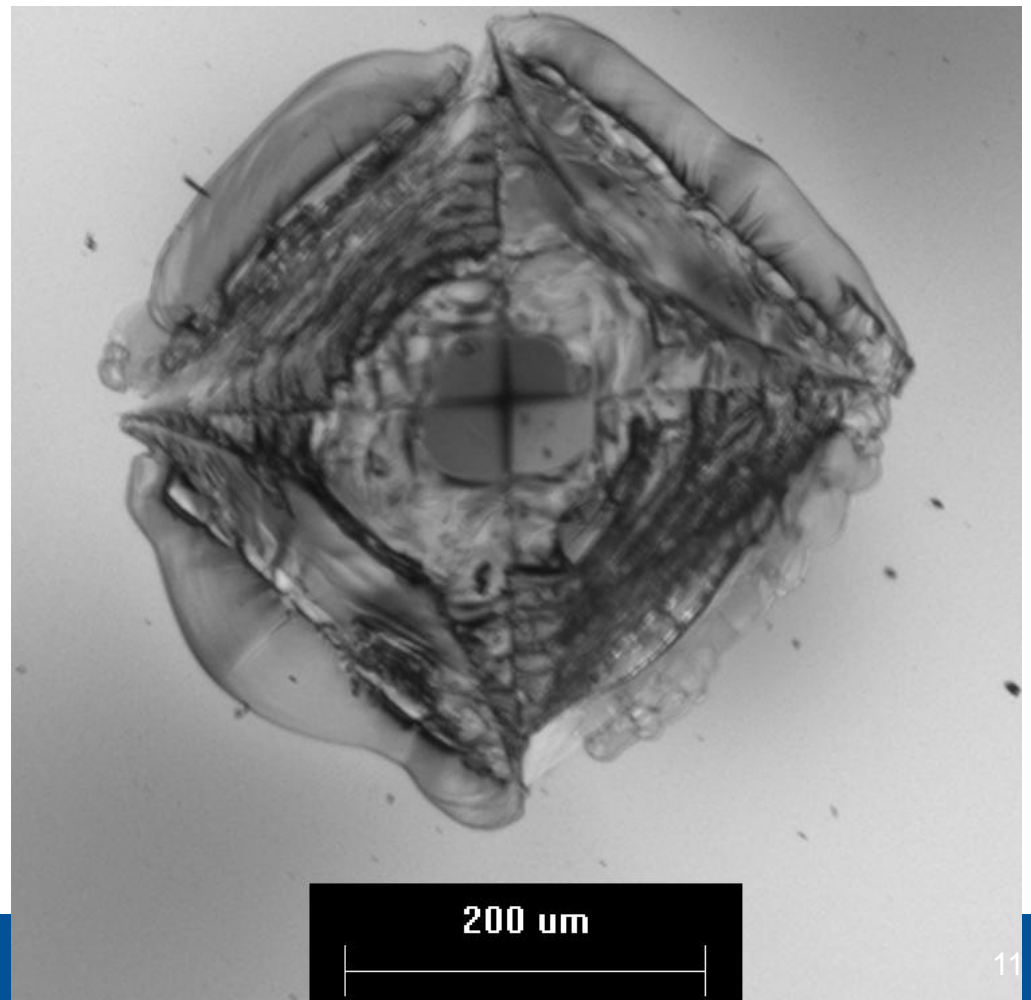T.M. Gross, ACerS Fractography VI Ceramic Transactions Vol. 320 (2012) 113-122.

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 at max load = 10 seconds.
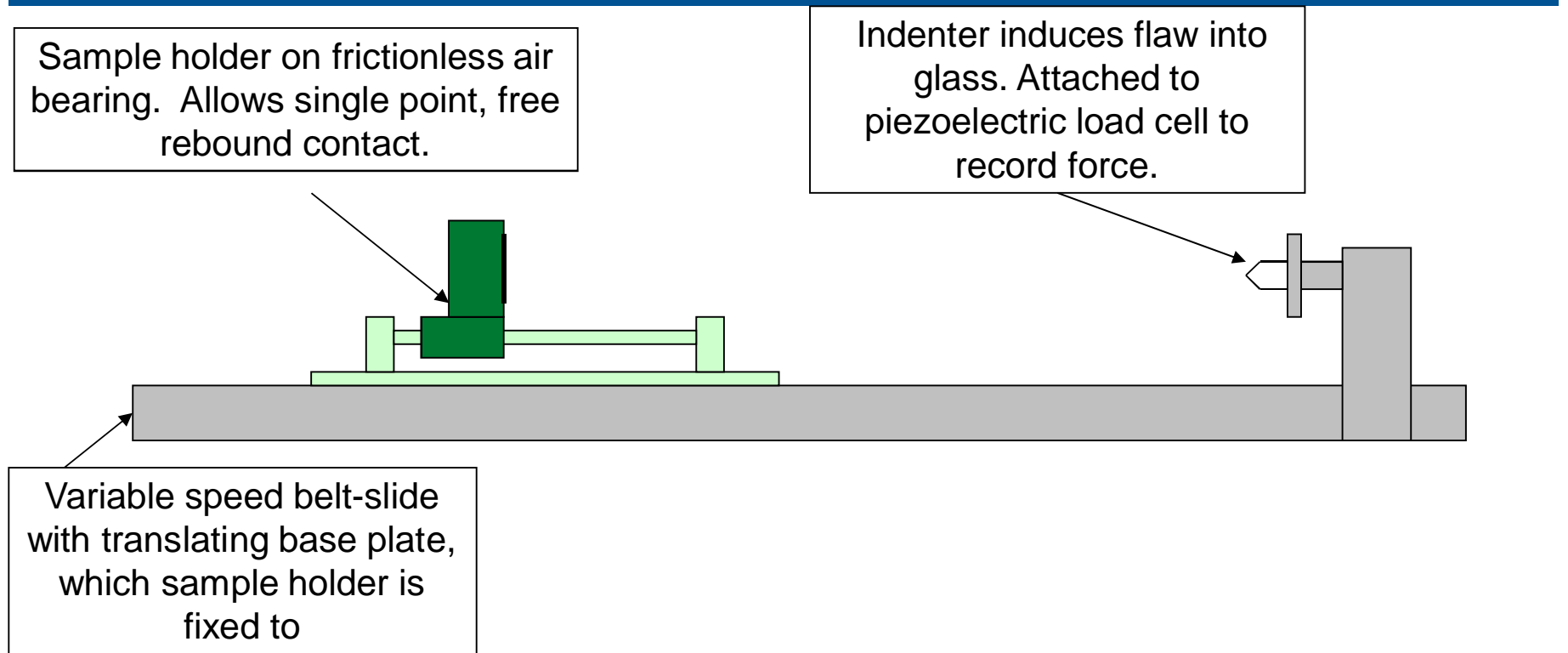
Ion-exchanged alkali aluminosilicate indented with Vickers at 57.3 kgf at 410 mm/s impact velocity. No median/radial cracks.

CS = 814 MPa DOL = 54 microns

Dynamic indentation

Sample holder on frictionless air bearing. Allows single point, free rebound contact.

Indenter induces flaw into glass. Attached to piezoelectric load cell to record force.

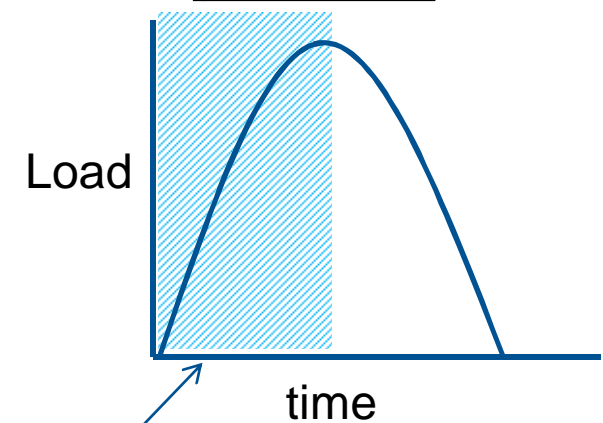Variable speed belt-slide with translating base plate, which sample holder is fixed to

- Each test specimen is indented a single time at a set velocity. The maximum possible load is constant for a set velocity.
- Failure criteria is the generation of a strength limiting crack. Specimen failure, i.e. complete separation of the specimen into two or more pieces, occurs as a result of formation of strength limiting flaws.

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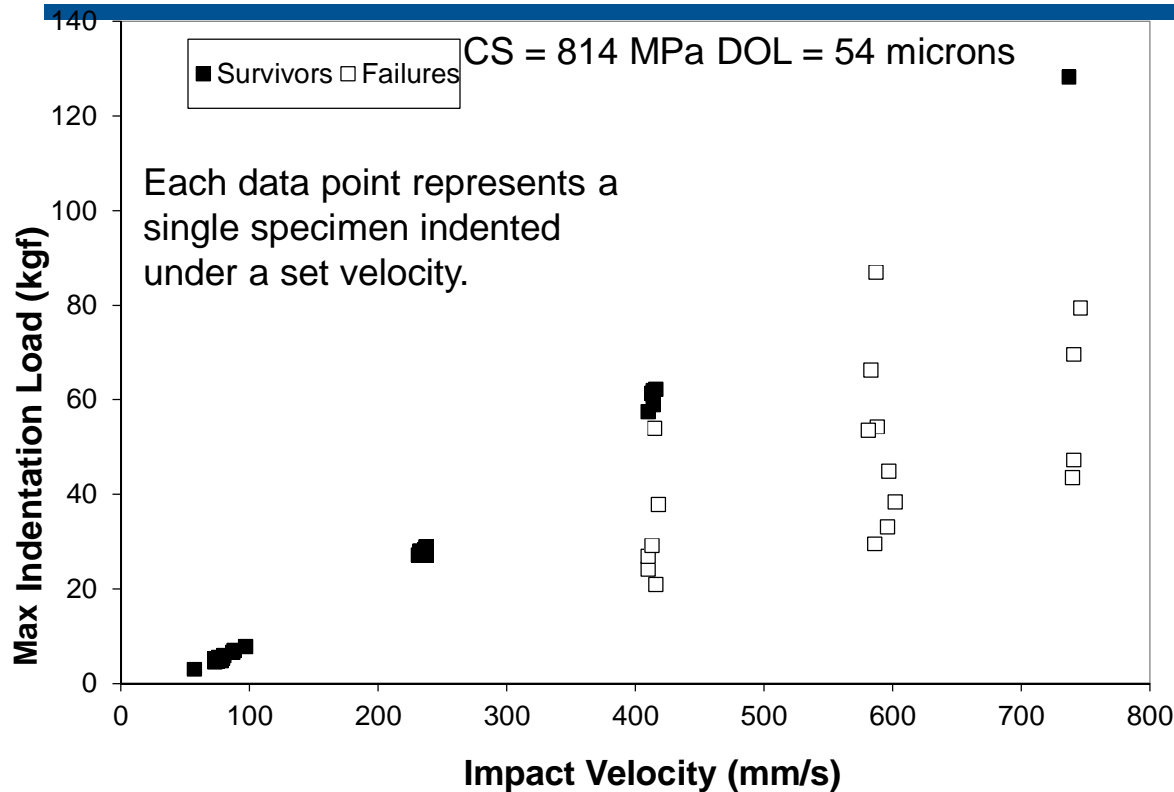
- For quasi-static Vickers indentation, median/radial cracks form during unloading.
- During dynamic Vickers indentation, median/radial cracks form during loading.
- Increased contact stress on median plane during loading is an indicator of densification according to Yoffe [Phil. Mag. A 46 (1982) 617].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

# Indentations made at 4.5 kgf by dynamic and quasi-static indentation
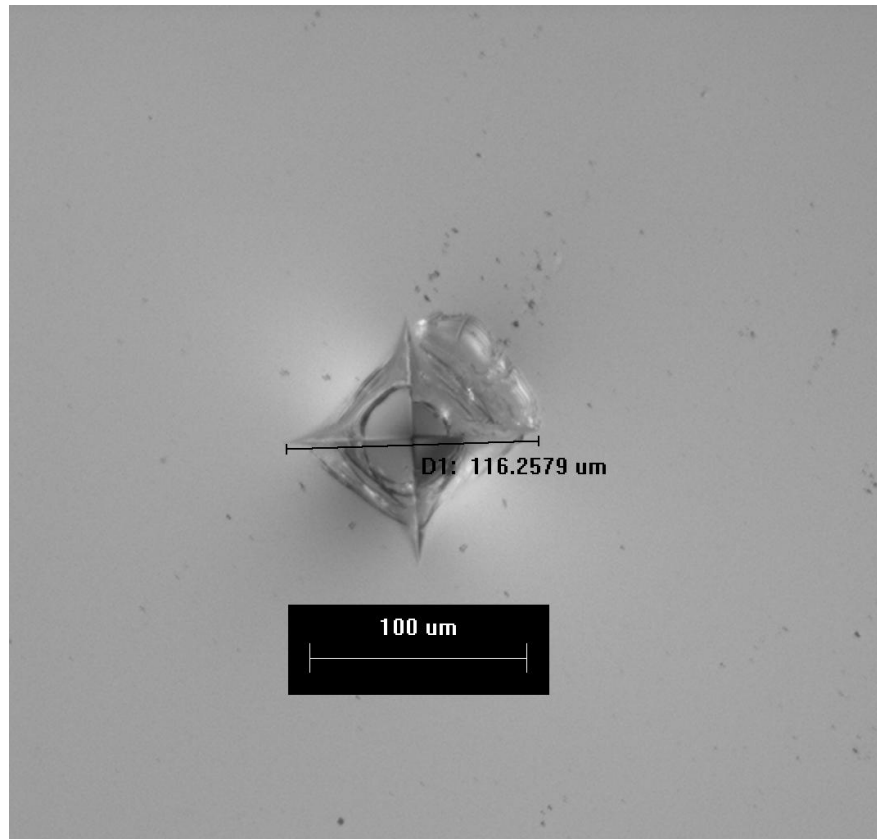
IXed alkali aluminosilicate indented at 4.5 kgf using dynamic indenter, contact event time (initial load to fully unloaded) was 1266 microseconds diagonal length ~ 116 microns.
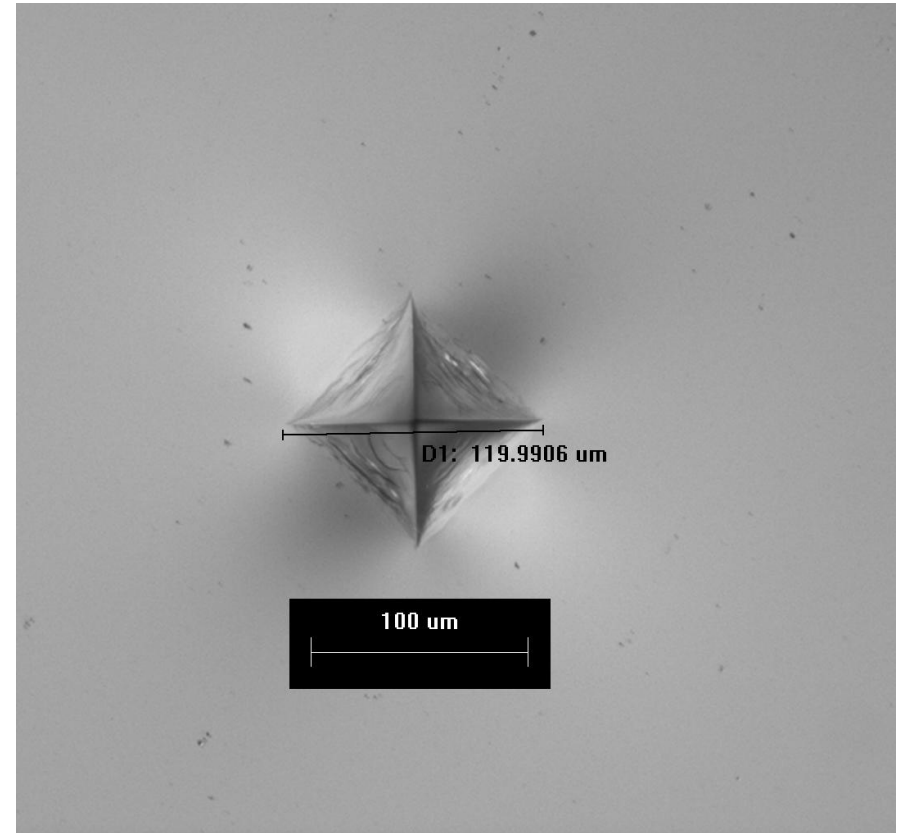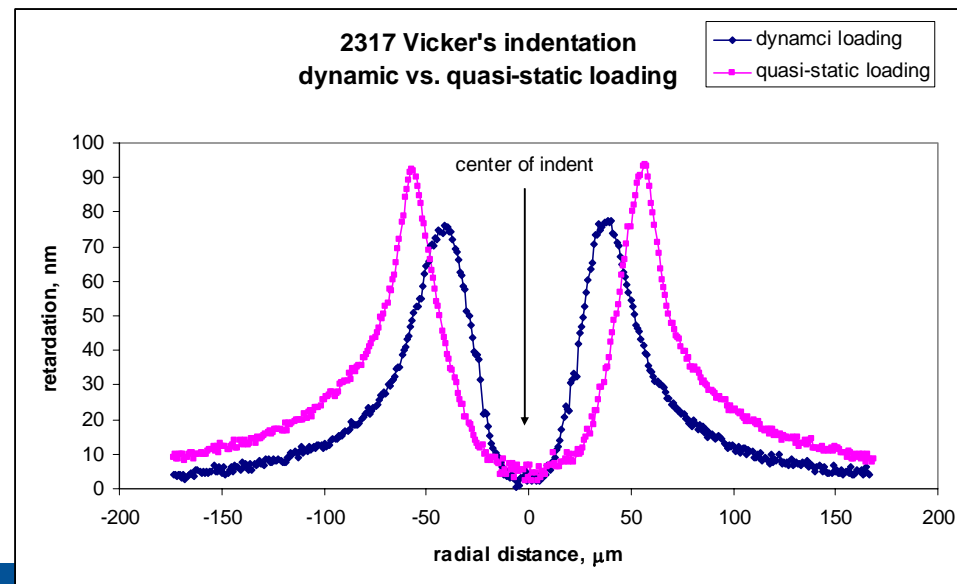
IXed alkali aluminosilicate indented at 4.5 kgf using dynamic indenter, contact event time was 55 seconds, diagonal length ~ 120 microns.

Optical retardation indicates a drop in residual stress for a given load during dynamic contact
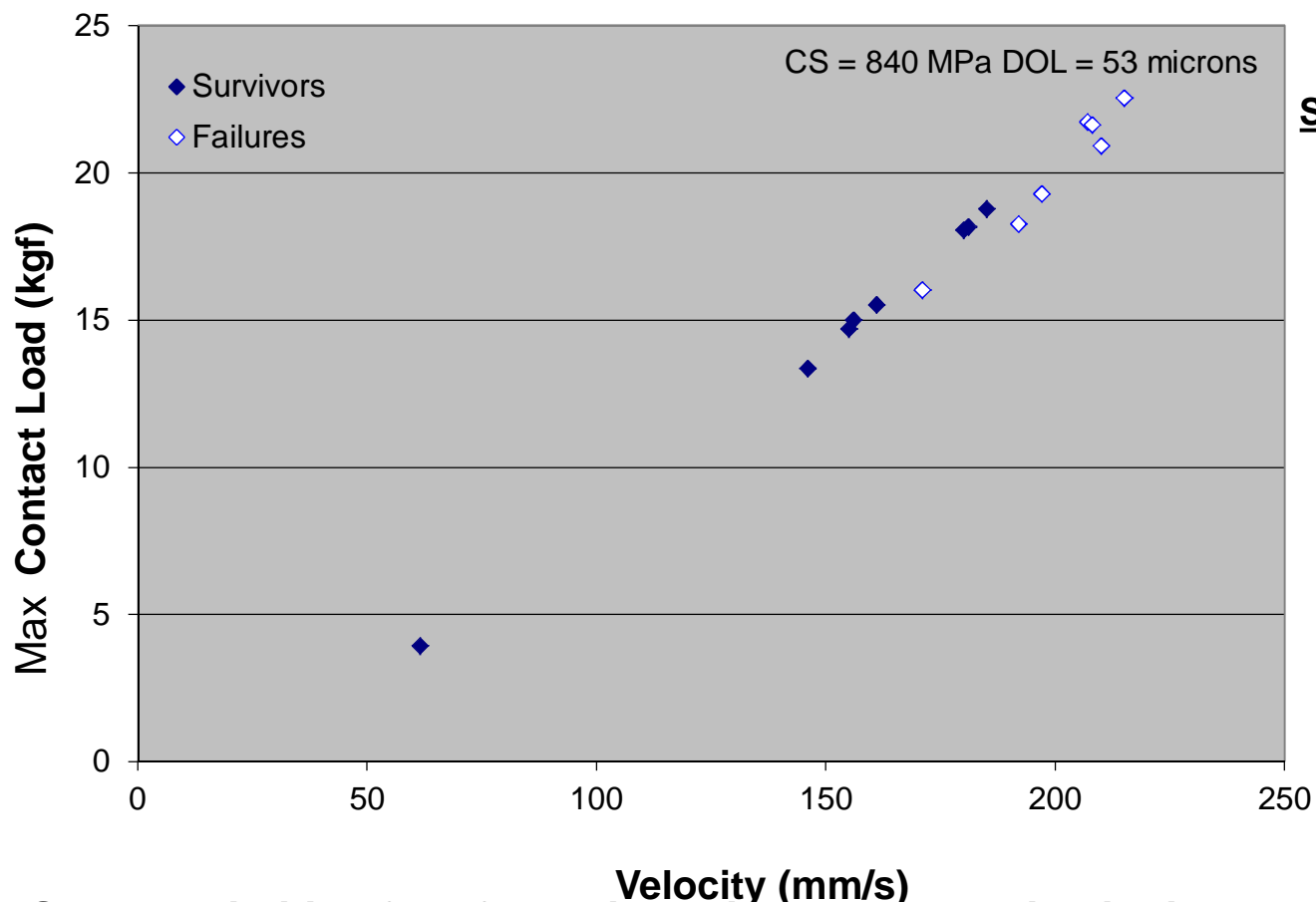
- Quantitative 2D grayscale maps of stress-induced optical retardation of 4.5 kgf indents made in alkali aluminosilicate specimens using quasi-static and dynamic indentation. Magnitude of retardation is significantly larger for the quasi-static loading case.
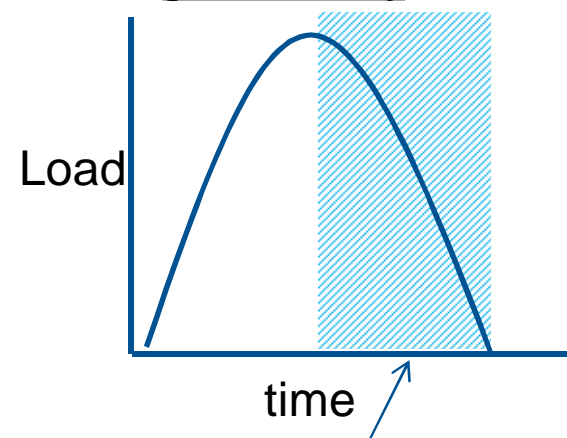

Quasi-static
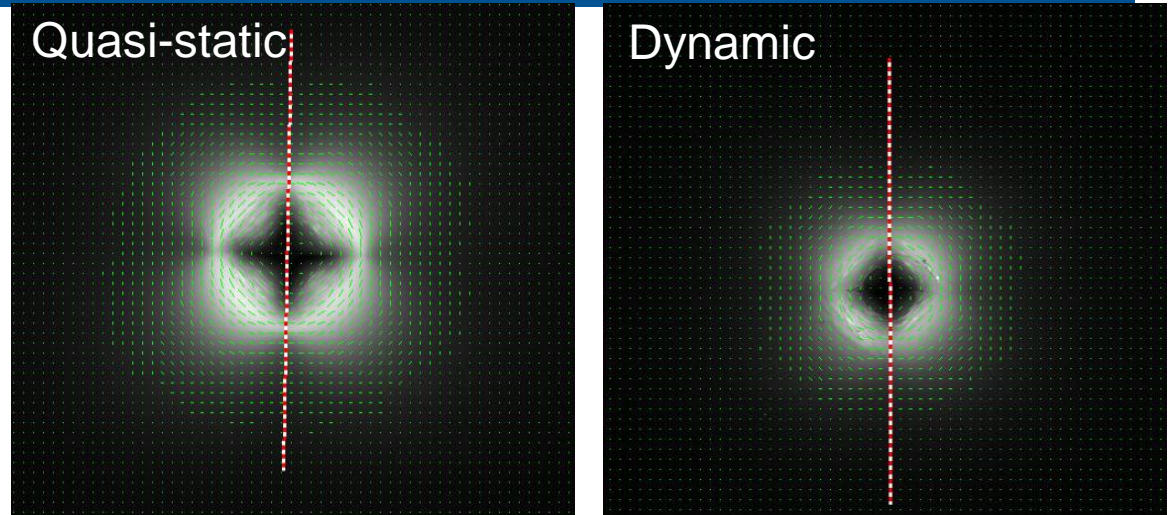
Dynamic

- Stress-induced retardation profile comparison for alkali aluminosilicate indented using quasi-static and dynamic loading indicates a reduced amount of residual stress (reduced shear deformation) during dynamic indentation.

2317 Vicker's indentation dynamic vs. quasi-static loading

— dynamci loading
— quasi-static loading

center of indent

retardation, nm

radial distance, µm

Dynamic Indentation with sharper 120° indenter tip

CS = 840 MPa DOL = 53 microns

◆ Survivors
◇ Failures

Max Contact Load (kgf) (y-axis: 0, 5, 10, 15, 20, 25)

Velocity (mm/s) (x-axis: 0, 50, 100, 150, 200, 250)

Schematic of load/time curve for a given velocity
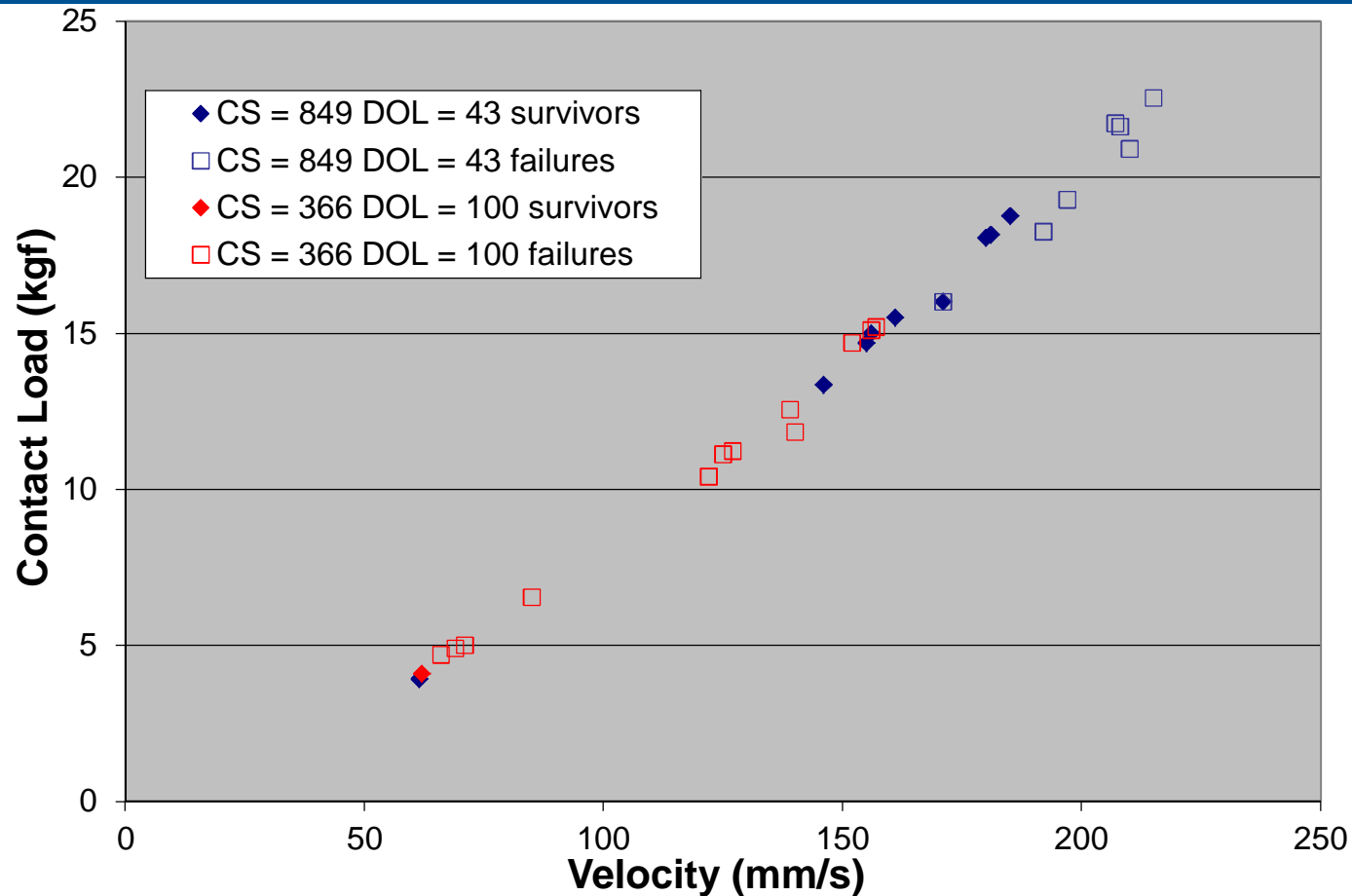
Load

time

Strength limiting flaw formation and failure occurs during unloading cycle for dynamic indentation with sharper 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.
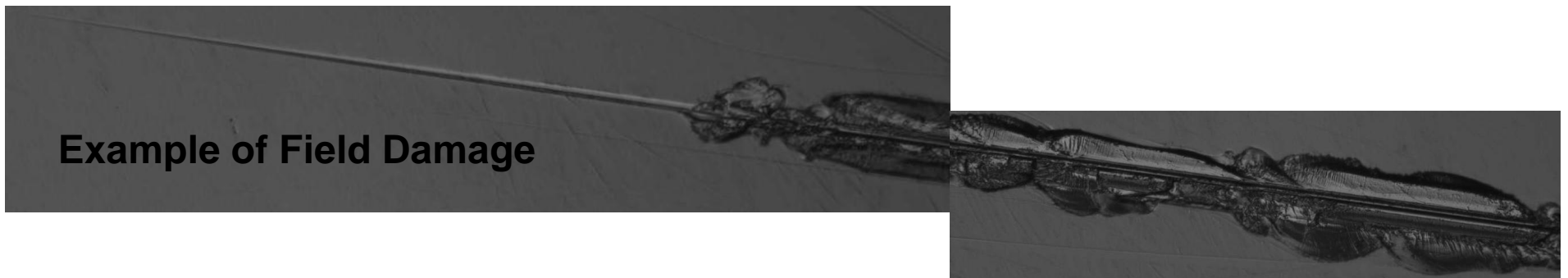
# Dynamic Indentation with sharper 120° indenter tip CS vs. DOL

Open symbols indicate failures, filled symbols indicate survivors. High compressive stress samples require much greater velocities/loads to cause failures under sharp dynamic indentation.

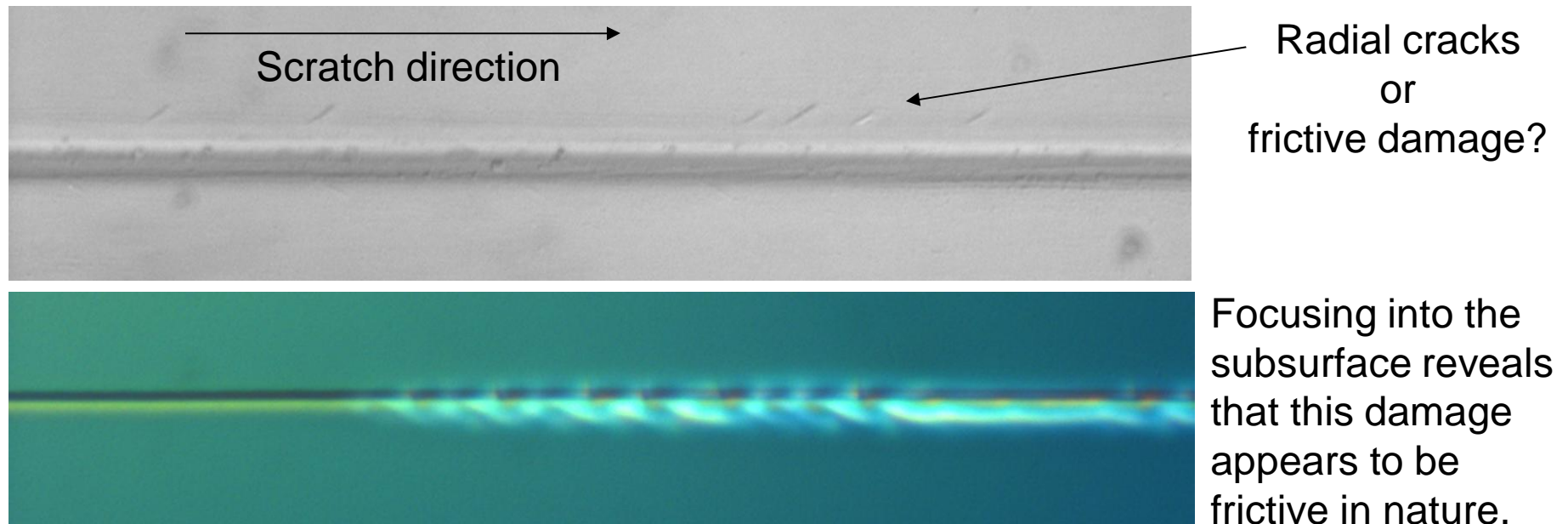# Replicating sharp contact scratches in ion-exchanged glass

Example of Field Damage

Ramped Scratch using Knoop diamond

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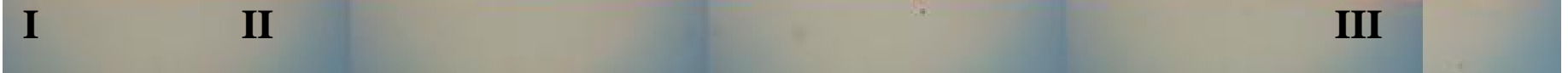
I.   Initially, plastic deformation occurs without the presence of cracks.
II. Increased frictional forces cause minor frictive-type damage.
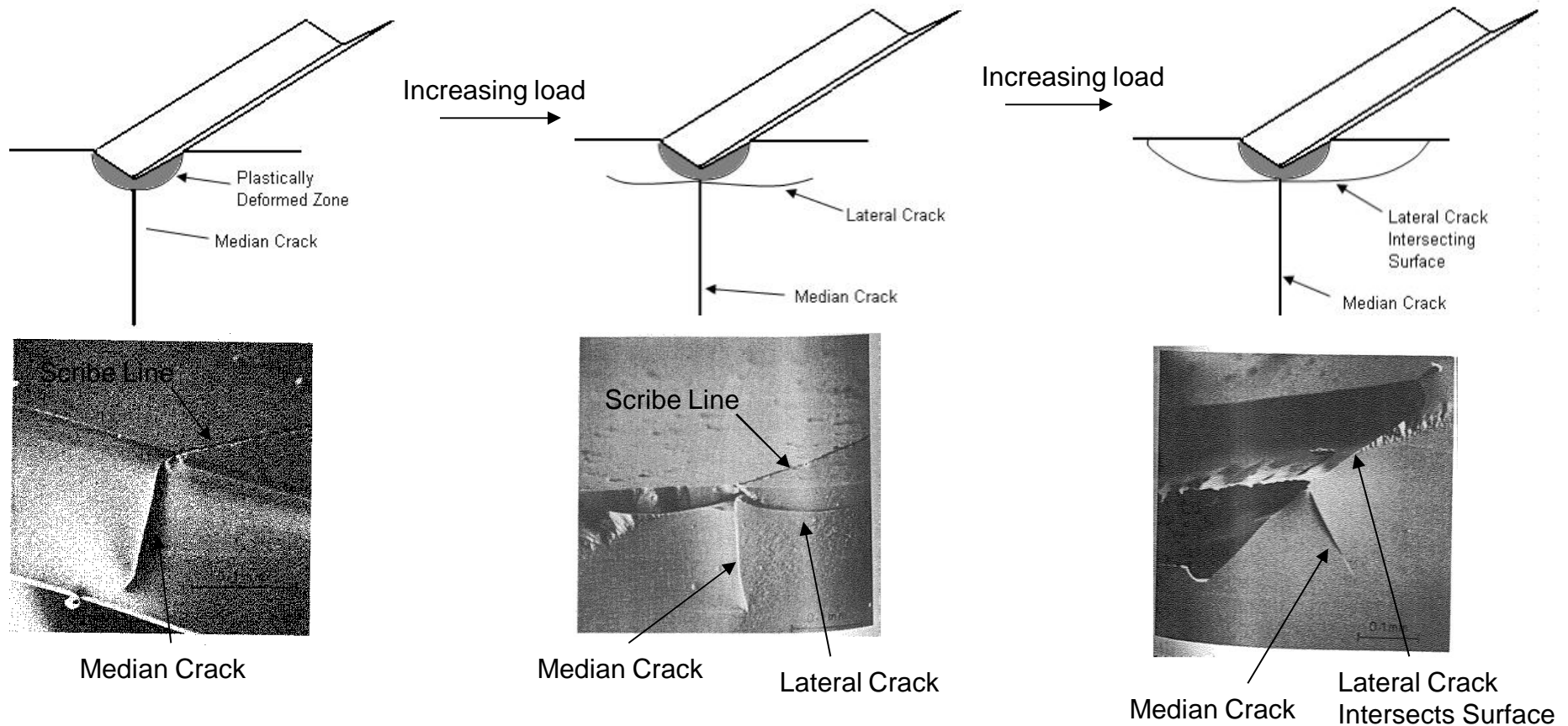III. Lateral cracking systems eventually cause highly visible chipping at the surface.

Regime II damage in scratched glasses

Scratch direction

Radial cracks
or
frictive damage?

Focusing into the subsurface reveals that this damage appears to be frictive in nature.

•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

Increasing load → Increasing load →

Plastically Deformed Zone
Median Crack

Scribe Line
Median Crack

Lateral Crack
Median Crack

Scribe Line
Median Crack
Lateral Crack

Lateral Crack Intersecting Surface
Median Crack

Median Crack
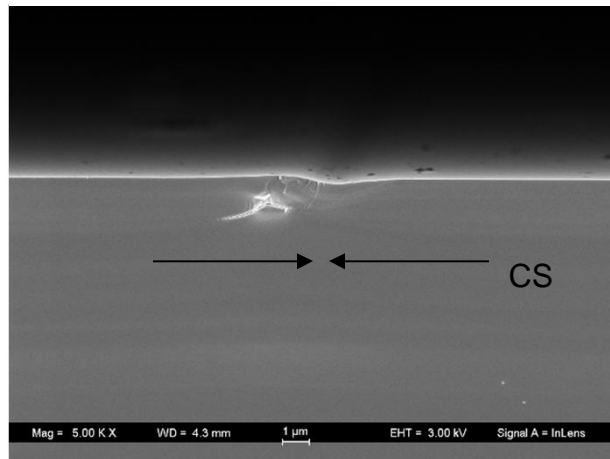Lateral Crack Intersects Surface

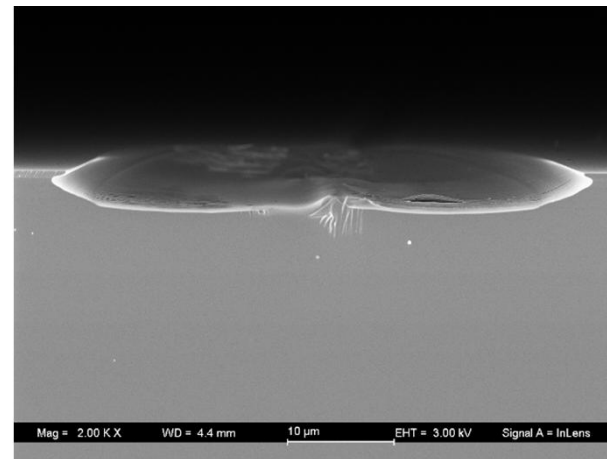V.R. Howes and A. Szameitat, J. Mater. Sci. Letters 3 (1984) 872-874.

Subsurface scratch damage in ion-exchanged glasses

In highly ion-exchanged glasses the lateral crack forms prior to the median crack
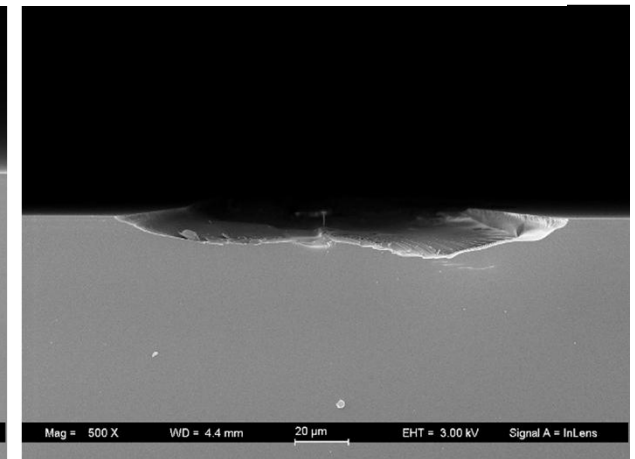
Ion-exchanged alkali aluminosilicate, Compressive stress = 770 MPa Depth of layer = 48 microns

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.

T.M. Gross, ACerS Fractography VI Ceramic Transactions Vol. 320 (2012) 113-122.

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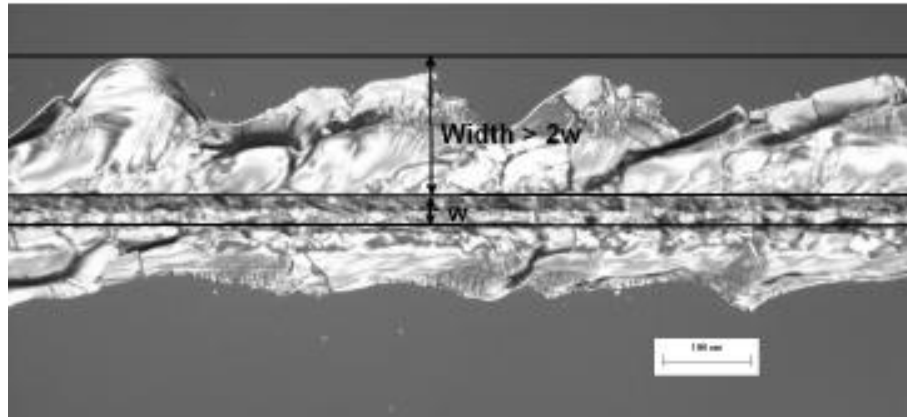
The lateral cracking threshold is defined in this work as the load which produces visual lateral cracks that extend a distance of twice the width of the scratch groove on either side.

10 mm long scratch with cracks exceeding lateral cracking threshold has high visual impact
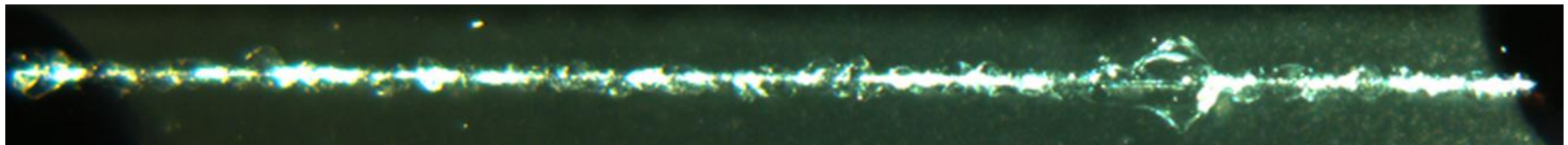
Pre-threshold scratches are difficult to see with the naked eye
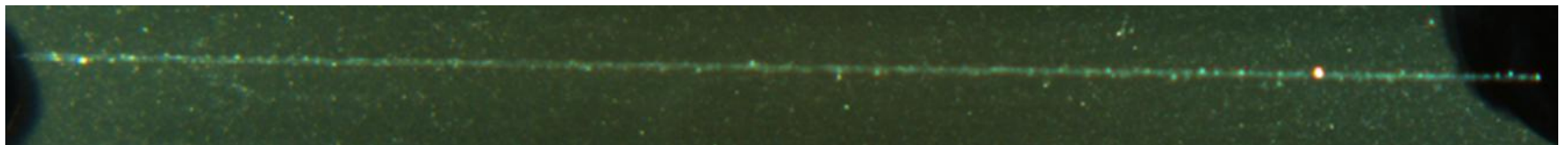
Scratch damage with various tips

- 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 pyamidal tip (Vickers)
    - Knoop tip
    - 150° 4-sided pyramidal tip

sharpest

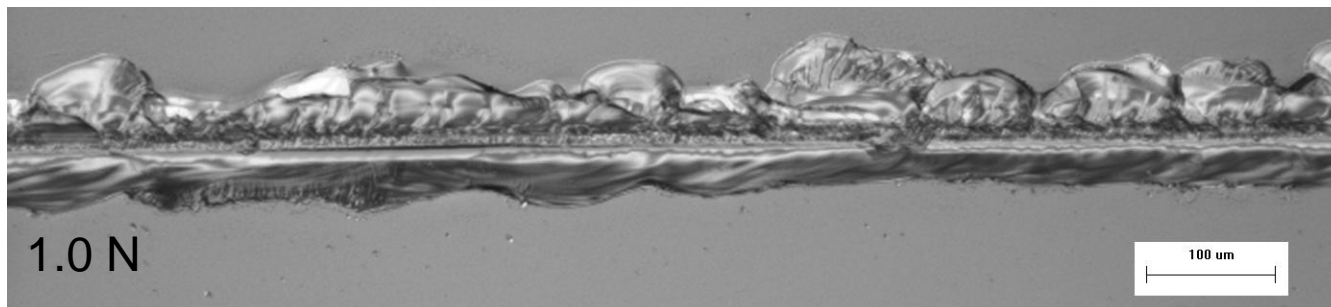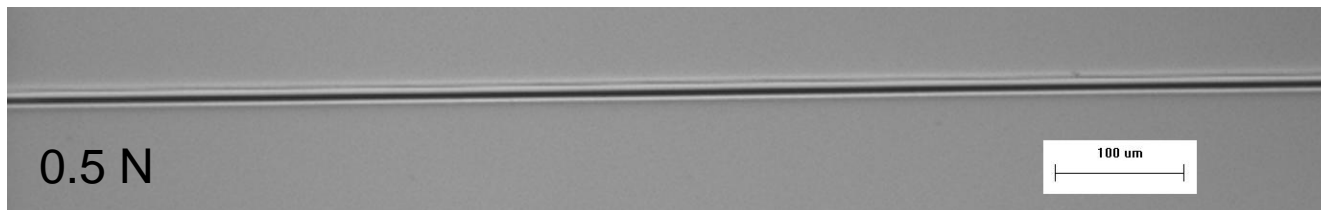**bluntest**

Knoop Tip

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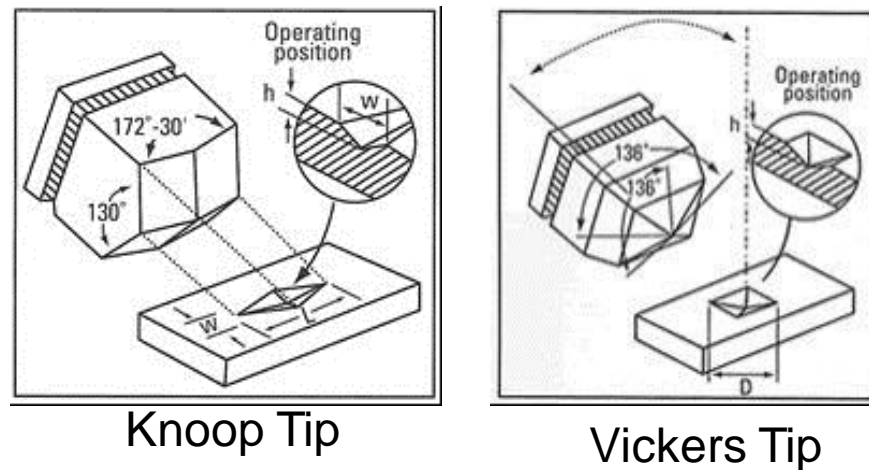
Scratches in ion-exchanged alkali aluminosilicate with 120° 4-sided pyramidal tip (sharpest tip)
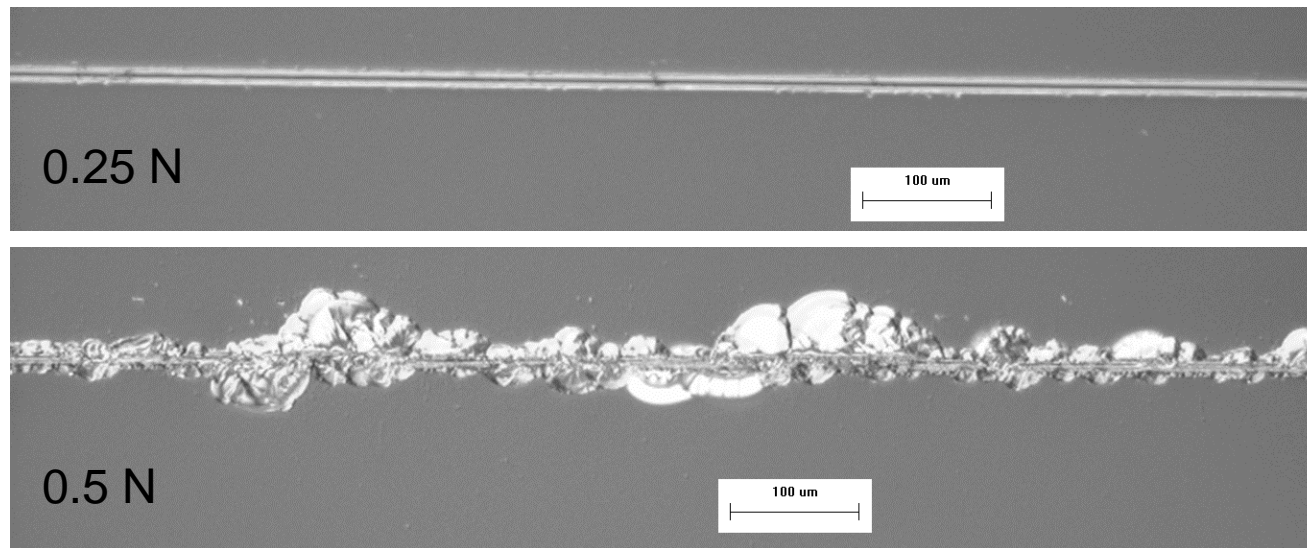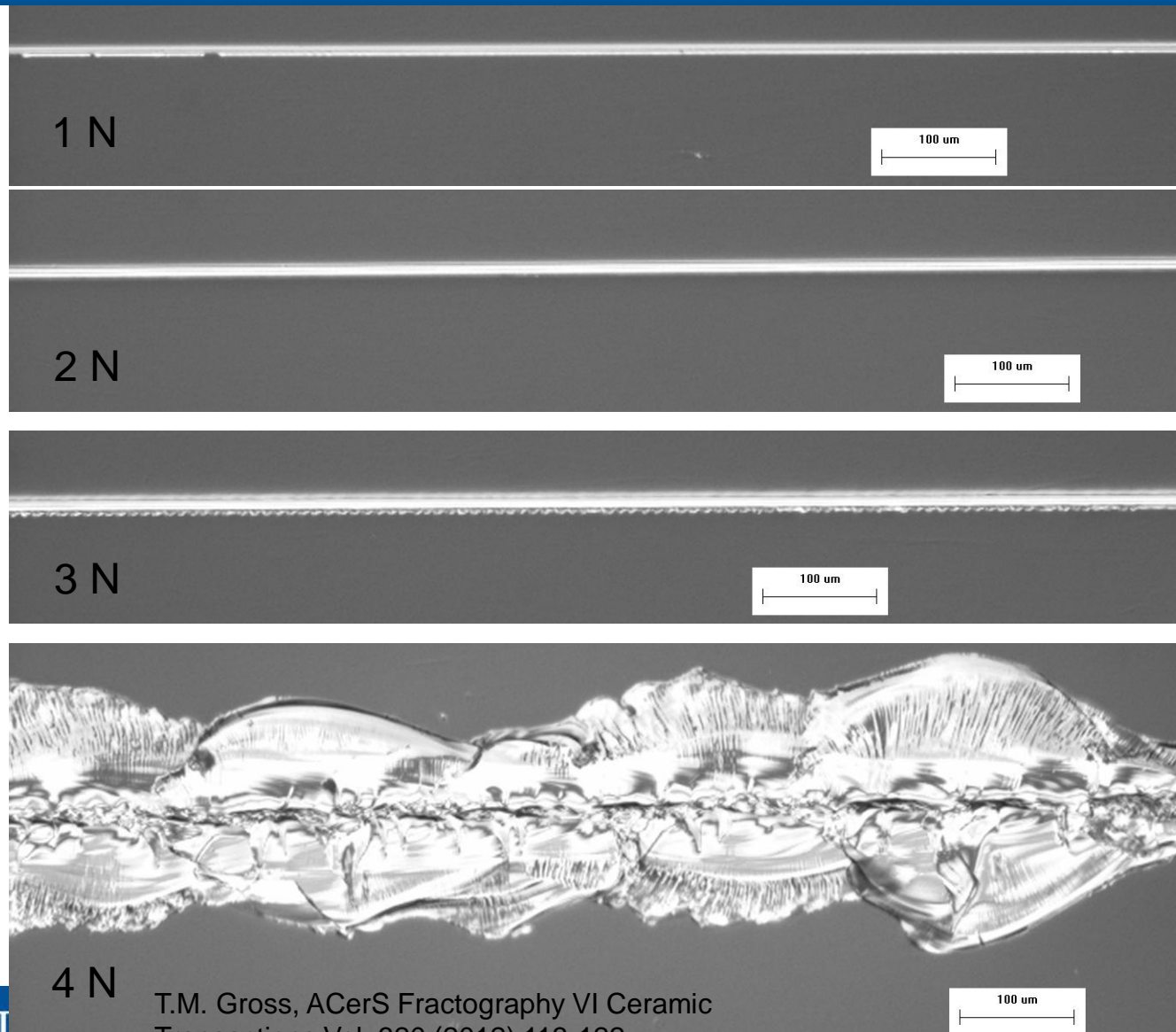

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Scratches in ion-exchanged alkali aluminosilicate with Vickers (136°) tip

T.M. Gross, ACerS Fractography VI Ceramic Transactions Vol. 320 (2012) 113-122.

Scratches in ion-exchanged alkali aluminosilicate with Knoop tip

1 N

2 N

3 N

4 N

T.M. Gross, ACerS Fractography VI Ceramic Transactions Vol. 320 (2012) 113-122.

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

5 N

100 um

10 N

100 um

15 N

100 um

Maximum
of load cell

20 N

100 um

T.M. Gross, ACerS Fractography VI Ceramic Transactions Vol. 320 (2012) 113-122.
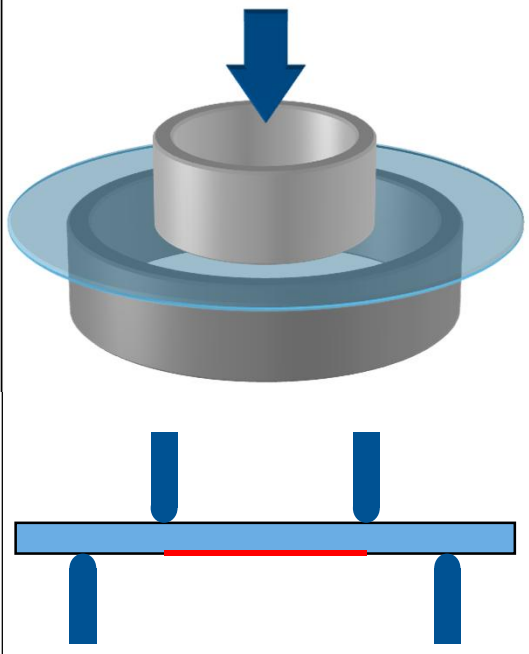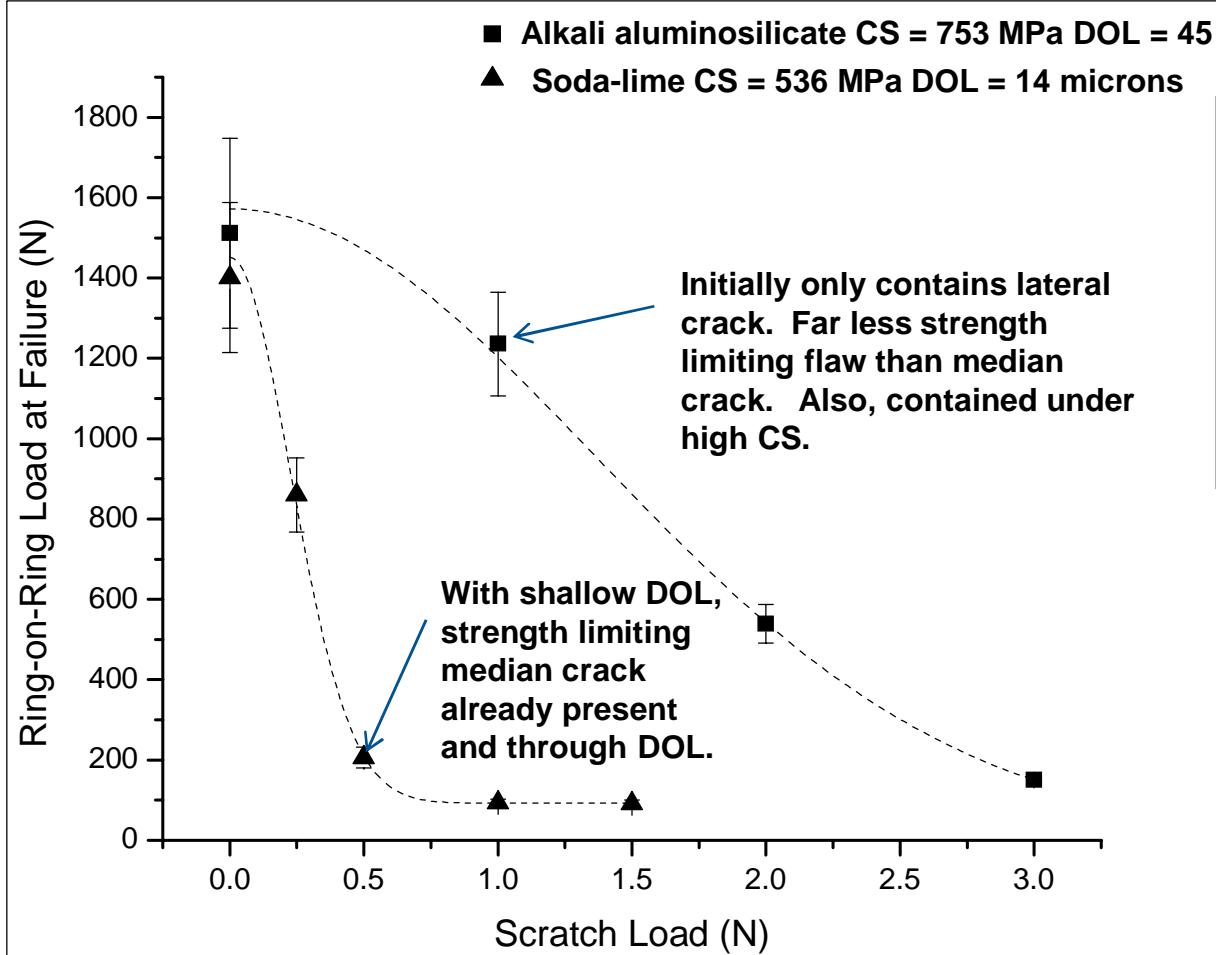
Retained strength

- 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

SLS   Deep DOL
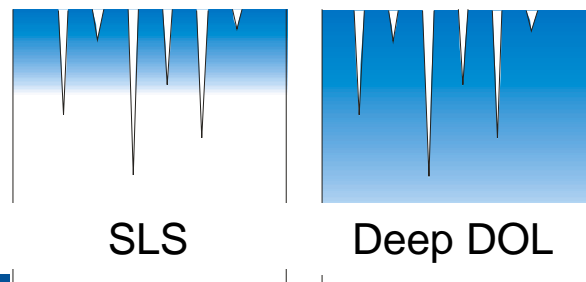
Ring-on-ring testing of Vickers scratches

Comparison of Ion-Exchanged Glass with Thickness = 0.7 mm

- ■ Alkali aluminosilicate CS = 753 MPa DOL = 45 microns
- ▲ Soda-lime CS = 536 MPa DOL = 14 microns

Initially only contains lateral crack. Far less strength limiting flaw than median crack. Also, contained under high CS.

With shallow DOL, strength limiting median crack already present and through DOL.
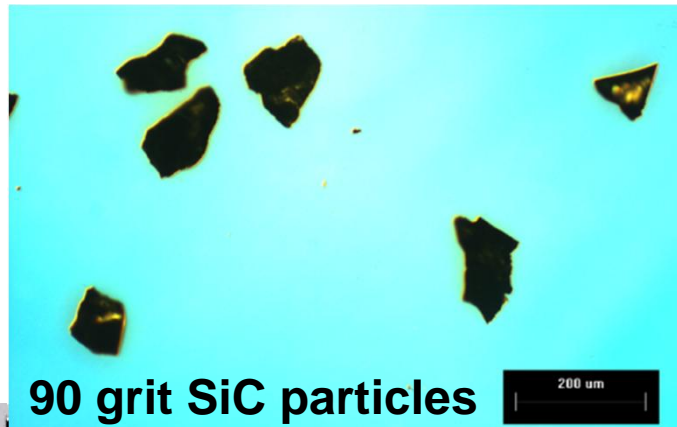
Scratch is fully within loading ring on bottom of ROR specimen. Load to strength conversion is non-linear.

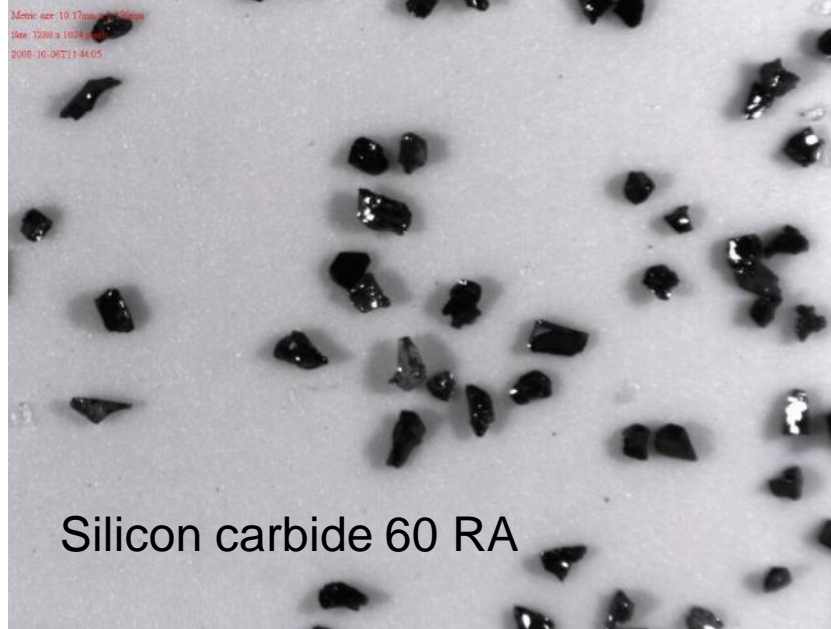# Scratch ring-on-ring of IXed alkali aluminosilicate at various DOL
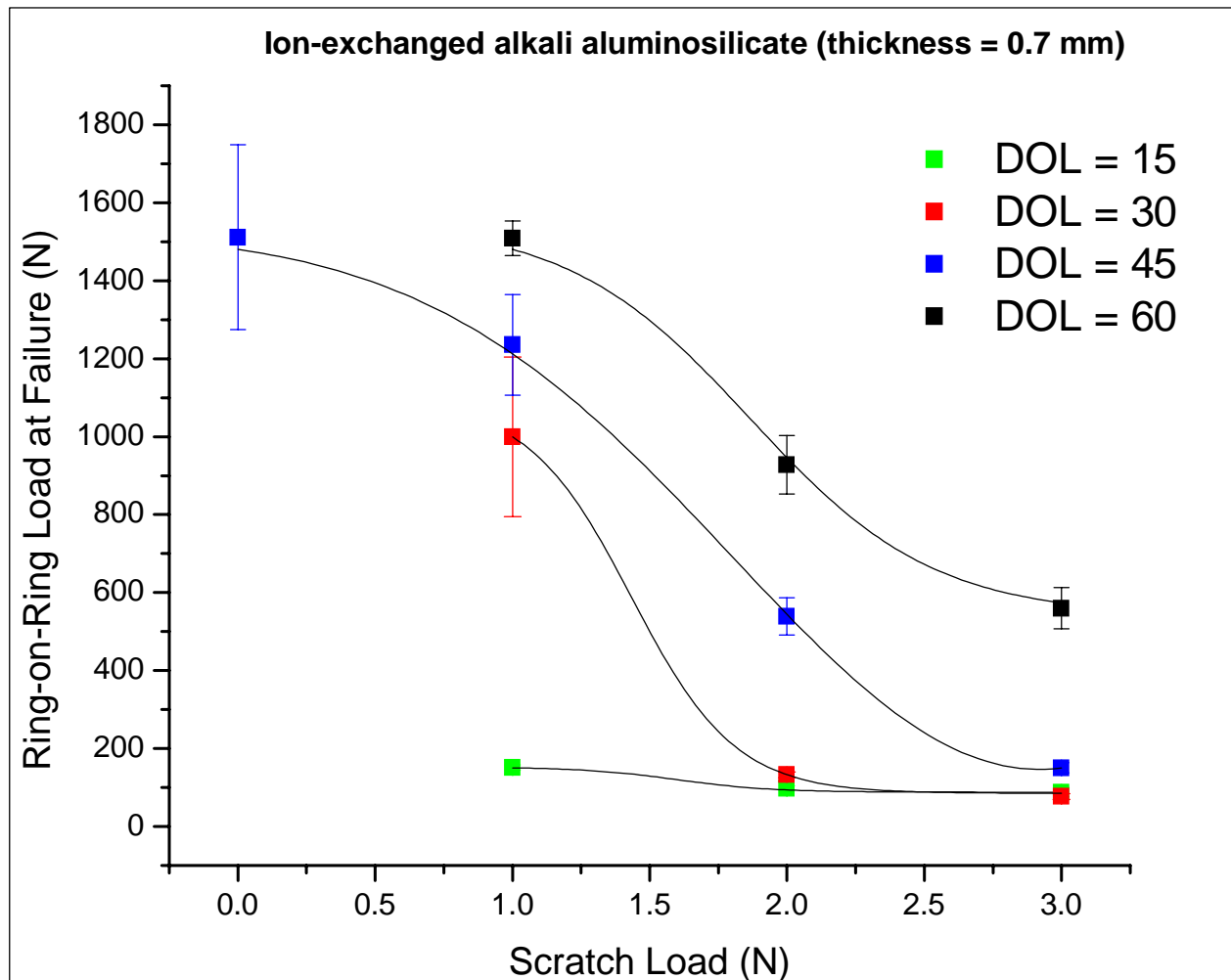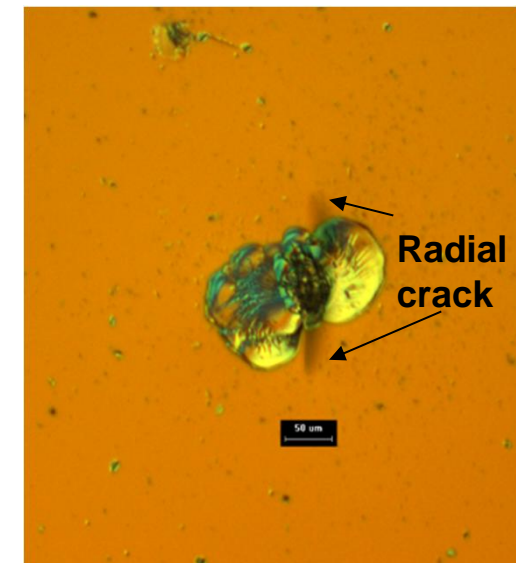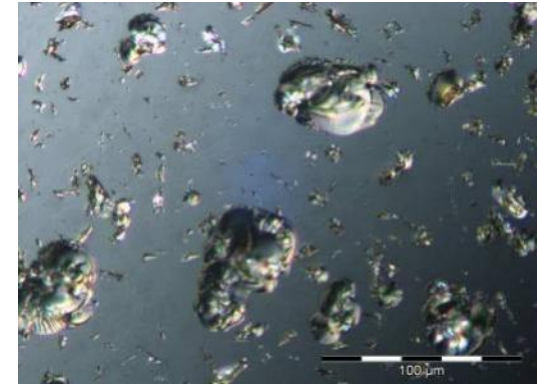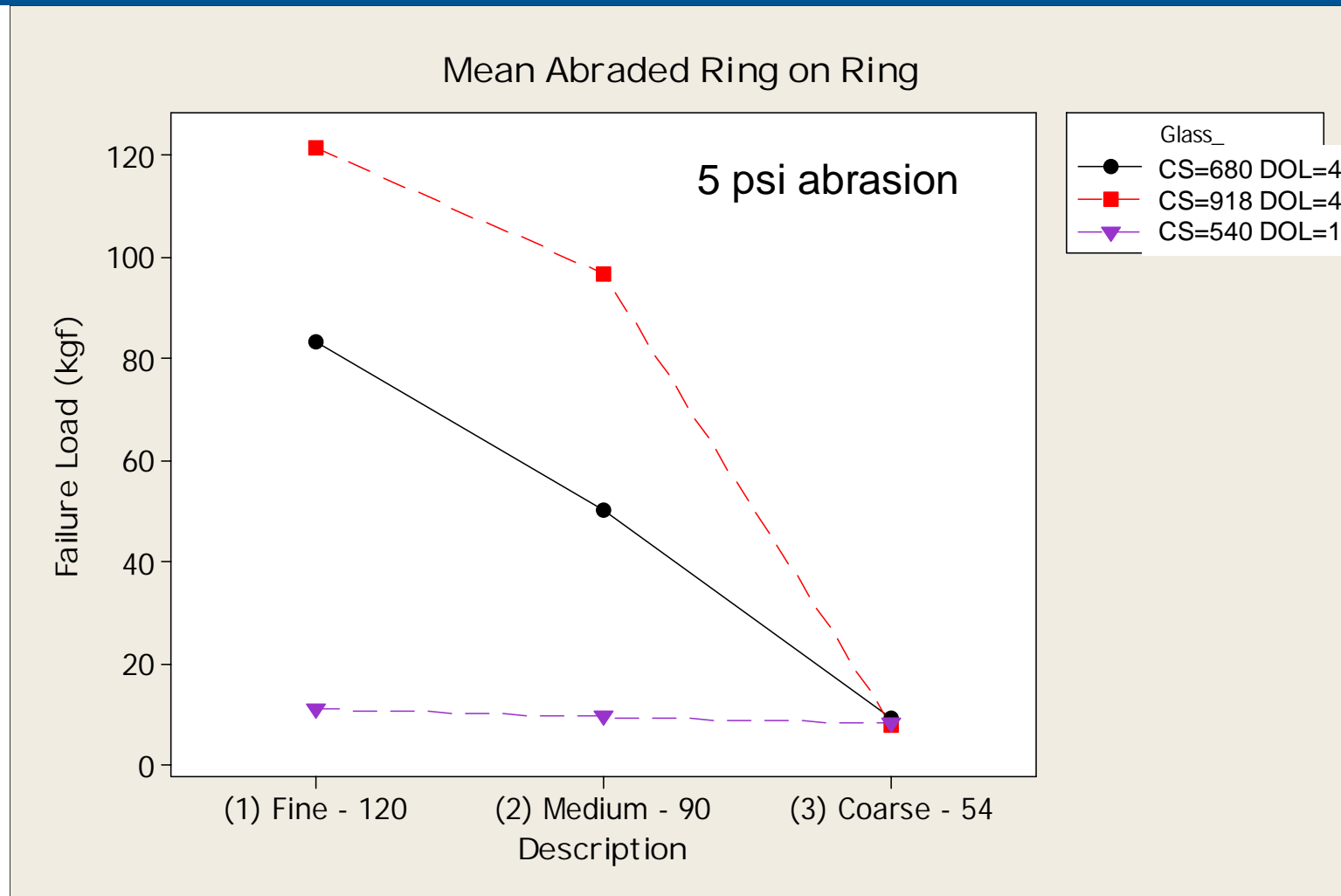

CS values held nearly constant

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

90 grit SiC particles

200 um

Silicon carbide 60 RA

100 µm

Radial crack

50 um

CORNING

Abraded ROR with fine, medium, and coarse SiC

Mean Abraded Ring on Ring

5 psi abrasion

Glass_
- CS=680 DOL=41
- CS=918 DOL=41
- CS=540 DOL=12

Failure Load (kgf)

(1) Fine - 120   (2) Medium - 90   (3) Coarse - 54

Description

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

# 90 Grit SiC Abrasion at various pressures on 0.6 mm alkali aluminosilicate
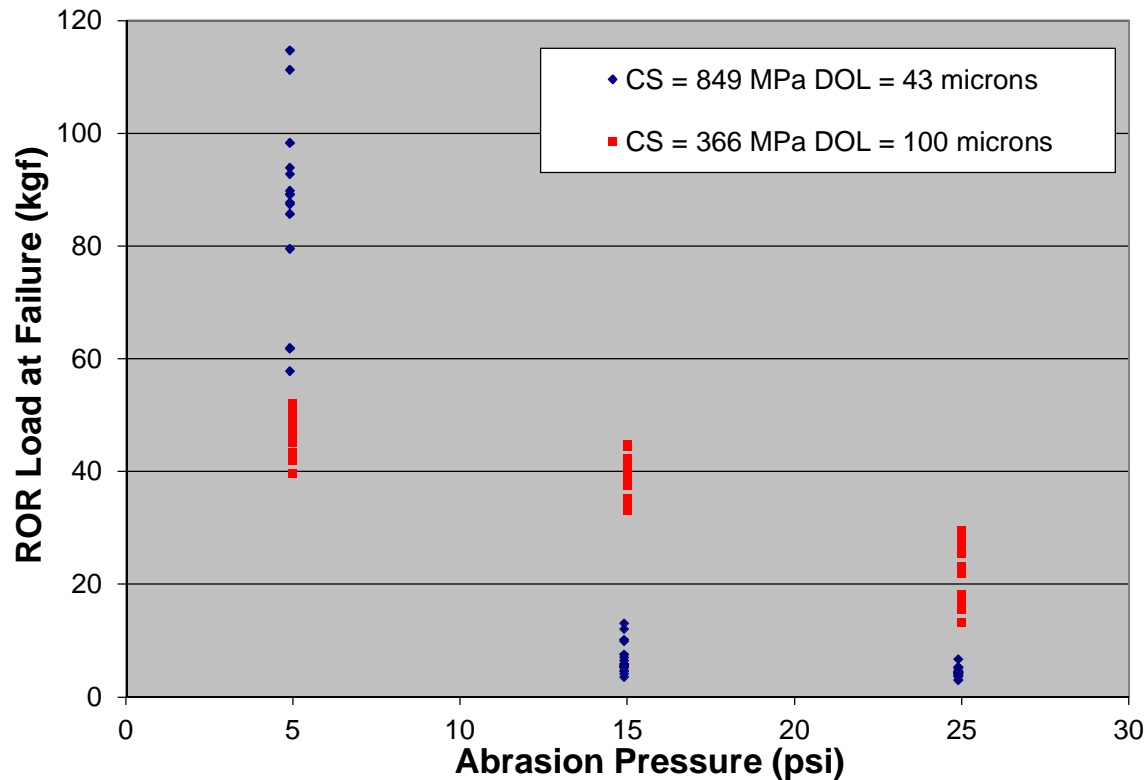
- Strength limiting flaws are generated at each abrasion pressure used.
- If DOL contains the flaw, the retained load at failure will increase with compressive stress to the flaw depth.
- Low CS, deep DOL parts can contain very deep flaws, however cannot achieve high strength even at shallow flaw depths. Non-abraded, low CS samples are also considerably weaker than high CS samples when testing samples similarly handled.

Summary

- 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).
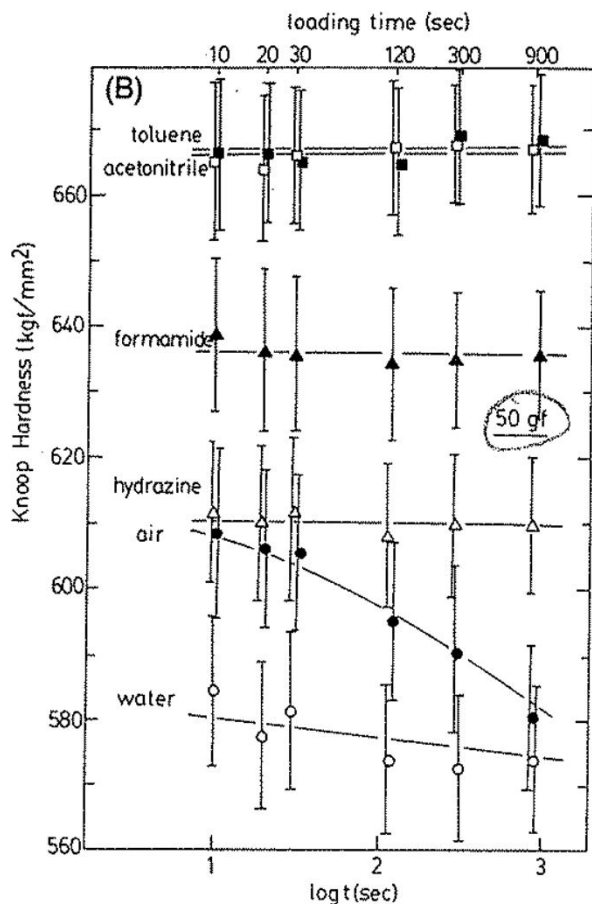
Indentation in N_2

Indentation in IXed alkali aluminosilicate in dry N_2 atmosphere at 15 kgf
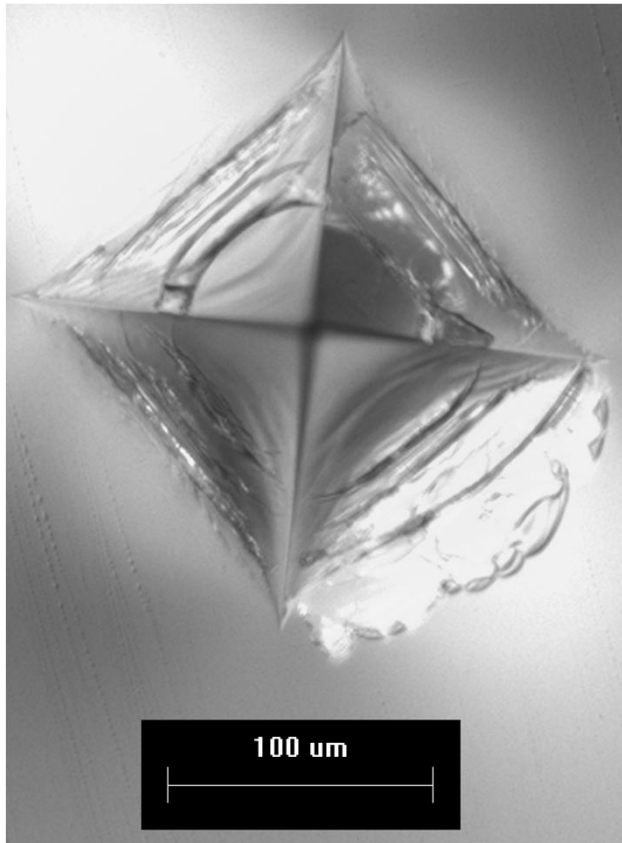
- Indentation N_2 glovebag increases indentation threshold 2X.
- Water was removed from air, but adsorbed water on glass and diamond surfaces was not removed.
- High speed indentation is expected to prevent sufficient water diffusion into glass.

Water diffusion into glass during indentation?

Hardness vs. loading duration in various environments

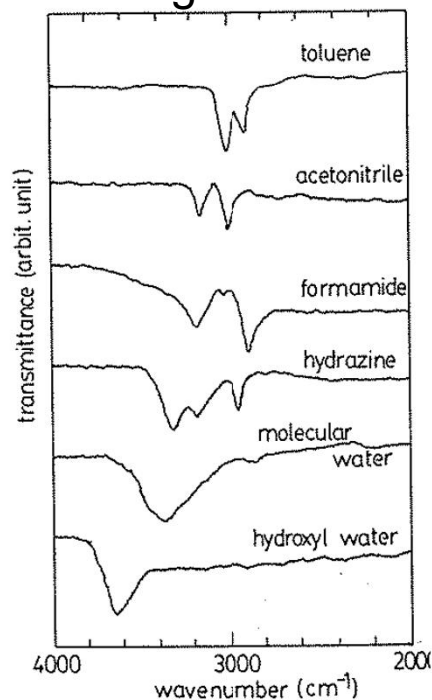Evidence of Water entry into glass during indentation

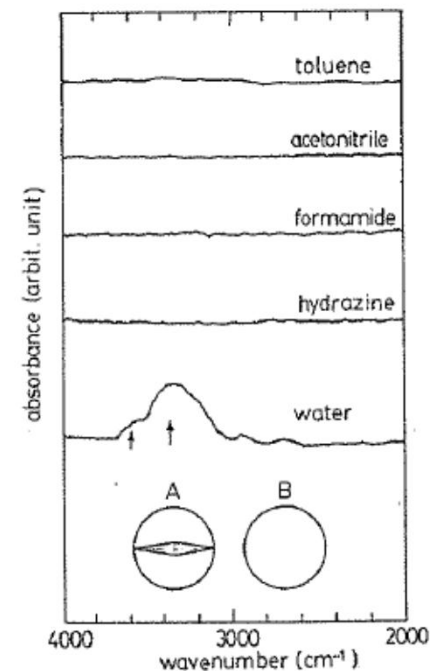Fig. 4. Infrared transmittance spectra of various liquids.

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
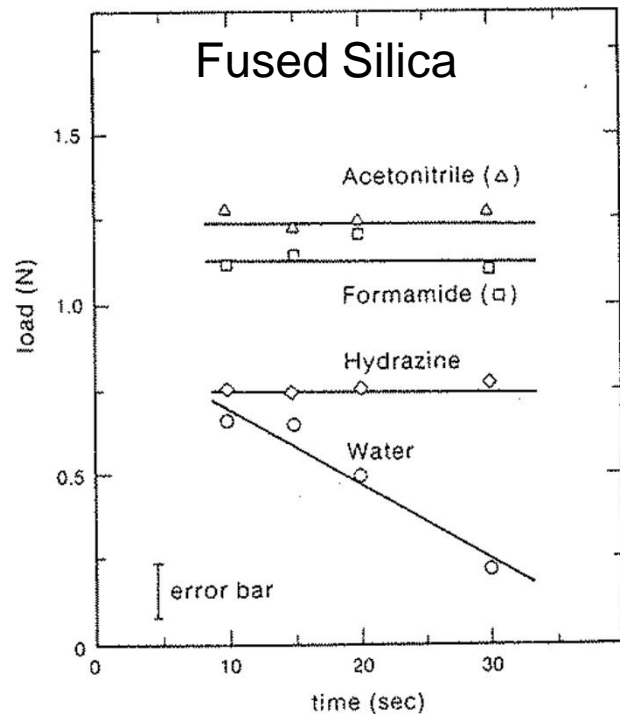duration in different environments

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.

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