Chemically Strengthened Glass: Science, Technology and its Future

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Acknowledgments

Morey Award Selection Committee

Parents

My guru:
Prof. Alfred R. Cooper
1924 –1996

Former students from Alfred Univ
Staff at Saxon Glass Technologies

2018 GOMD Morey Award Lecture
Visitors to his home admired his kitchen garden displaying Na$_2$O-CaO-SiO$_2$ phase diagram with different colored flower arrangements to identify different phase fields.

George Washington Morey (1888-1965)
B. S. Chemistry, University of Minnesota, 1909
Geophysical Laboratory, staff member (1912 - 1952); acting director (1953)

Research on phase equilibria relations in glass forming silicate systems

Properties of Glass 1938 book one of the most thorough compilations of data and discussions on glass properties, phase diagrams, including many sets of additivity relations.

D.Sc., Alfred University, 1939
(Honorary doctorate honoris causa)

AU president Norwood citation, “Noted son of the University of Minnesota, outstanding scientist in the field of chemistry and glass technology, public servant, manufacturer, business executive, lecturer, member, officer and past officer of the American and foreign scientific societies…”

2018 GOMD Morey Award Lecture
Objectives of this presentation

• Fundamentals of glass chemical strengthening
• Science of ion exchange and stress development
• Testing of strengthened glass
• Some important applications
• Recent developments in science and technology
Glass chemical strengthening

(1) “Chemical Strengthening of Glass: Lessons Learned and yet to be Learned”

(2) “Buildup and relaxation of stress in chemically strengthened glass”


3rd edition!

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Terminology

Strengthening

*Stress to fracture is higher*

Hardening

*Harder material abrades a less hard material
Has greater fraction of elastic recovery upon unloading the indent*

Toughening

*Area under the stress-strain curve*

There is no such thing as an unbreakable glass

*just like hearts, glass will break when sufficient tensile stress is applied*
Chemical Strengthening of glass

Kistler JACerS 1962

- **How it works:**

Submerge in a bath of molten KNO₃

Original surface

Surface in compression

Ion Size

- Na⁺ (0.95 Å)
- K⁺ (1.33 Å)

*Not drawn to scale, demonstration only*
Examples of Ion Exchange

Surface compressive stress generation
Na⁺ (glass) ↔ K⁺ (salt bath)
Li⁺ (glass) ↔ Na⁺ (salt bath)

Surface tensile stress generation
K⁺ (glass) ↔ Na⁺ (salt bath)
Na⁺ (glass) ↔ Li⁺ (salt bath)

Surface color generation without stress development
Na⁺ (glass) ↔ Cu⁺ (salt bath)

Surface refractive index change without stress development
Na⁺ (glass) ↔ Ag⁺ (salt bath)
Characterization of chemically strengthened glass

Concentration of invading ions and stress vs depth

• IMPORTANT PARAMETERS:

(1) MAGNITUDE OF SURFACE COMPRESSION “CS”
(Typically 400 – 1,000 MPa)

(1) CASE-DEPTH
Or Depth of Layer “DOL”
(typically 15 to 300µm)

- Basis of ASTM C-1422
Chemical strengthening principles

- Alkali (e.g. Na)-containing glass is immersed in a molten salt e.g. KNO₃ bath.
- Temperature lower than $T_g$ of glass.
- Sodium ions from glass surface diffuse out into the bath.
- Potassium ions occupy those sites.
- Stuffing leads to glass surface compression.
- Compression leads to glass strengthening.
- Some balancing interior tension can lead to fragmentation in thin glasses after the compression case is penetrated.

S. S. Kistler, JACerS, 45,59(1962)
(1) Glass composition: Only alkali-containing glasses. Silica impossible to strengthen; low-expansion borosilicate and PbO-containing glasses are difficult.

(2) Exchange temperature (typically 350-475°C)

(3) Exchange time (typically 2h – 24h)

(4) Salt bath composition (pure salt vs mixed alkali salt)

(5) Bath impurities build up with time: replaced alkali ions, silica, CaO, iron, chrome, nickel and carbon.

(6) pH of the bath
Effect of bath impurities

Fig. 3. Effect of different concentrations of Ca$^{2+}$, Sr$^{2+}$ and Ba$^{2+}$ in a KNO$_3$ bath on the strength of Na$_2$O–CaO–SiO$_2$ glass. Ion exchange at 470°C for 3 h. ○ Ca$^{2+}$, △ Sr$^{2+}$, □ Ba$^{2+}$. X. Zhang, O. He, C. Xu, and Y. Zheng, J. Non-Cryst. Sol. 80(1986) 313-318
Effect of Ca\textsuperscript{++} impurity
V. Sglavo; GOMD Madison WI 2016

Buildup of Ca\textsuperscript{++} and Na\textsuperscript{+} impurities in the bath over time is a strong poison for ion exchange strengthening.

Saxon Glass Technologies approach:
Add certain compounds that scavenge Ca\textsuperscript{++} and Na\textsuperscript{+}.
No need to turnover the salt bath…

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Pros and cons relative to thermal tempering

**Pros:**
- Very high surface compression
- Almost no geometric distortion
- Small internal tension (almost no dicing unless much thinner glass used)
- Complex geometries, e.g. tubes, can be strengthened

**Cons:** Cost, cost, cost, cost
- Several hours of immersion process
- Disposal costs of hazardous waste
- Low case depth 20 to 50 microns (less forgiveness to handling flaws)
- Limited to alkali-containing glasses
Concentration dependent interdiffusion coefficient

\[ \bar{D} = \frac{D_{Na}D_K}{D_{Na}N_{Na} + D_KN_K} \]

where \( D_i \) = self diffusion coefficient of \( i \)

- \( N_i \) = fractional molar conc of \( i \).
- In the surface \( N_{Na} = 0 \), \( \bar{D} = D_{Na} \)
- In the deep \( N_K = 0 \), \( \bar{D} = D_K \)
Mathematics of stress development

Semi-infinite plate; y and z dimensions are much larger than the thickness

Diffusion in ± x direction

Biaxial stress system

Free elongation of each x-layer

\[(e_y)_x = (BC)_x\]

C = concentration of the invading ion
B = linear network dilation coefficient

x- layers are constrained from free expansion.

Cooper: Use analogy to thermal stress; C ≡ T; B ≡ α
Cooper’s extension of Richmond, Leslie, Wriedt 1964

In-plane stresses

\[
(\sigma_{yy})_x = (\sigma_{zz})_x = -\frac{(BC)_x E}{1-\nu} + \frac{E}{2(1-\nu)L} \int_{-L}^{+L} (BC)_x dx
\]

Perpendicular stress

\[
(\sigma_{xx})_x = 0
\]

C = concentration of the invading ion
2L = plate width ; d = case-depth = DOL
E = Young’s modulus; \( \nu \) = Poisson’s ratio
B = linear network dilation coefficient ( = “Cooper Coefficient”)

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Figure 14.12. Dimension changes after ion exchange.
(A) No ion exchange. (B) Ion exchanged and unrestrained.
(C) Ion exchanged and restrained.
Any efforts to get profiles deeper than ~25 µm in SLS glasses end in relaxed stress profiles.

A. Y. Sane & A. R. Cooper, JACerS 70(2) 86-89 (1987)

R. Gy, Mat Sci & Eng B, 149, 159-165 (2008)
• Optimization of time-temperature in bath and bath salt composition are most important.
General observations with variation in glass composition

- **Silica glasses do not strengthen**
- **Poor strengthening (surface compression only about 50 -150 MPa):**
  - Pb-crystal glasses
  - Low expansion borosilicate glasses
- **Medium strengthening (300 MPa; about 15-35 µm case depth)**
  - Medium expansion borosilicate (Type 1)
- **Good strengthening (400 - 700 MPa, about 15-35 µm case depth)**
  - Soda lime silicate
- **Very good strengthening (300 – 1000 MPa, about 50 µm to 1 mm case depth)**
  - Alkali aluminosilicate glasses
How to test strengthened glass?

(1) Test surface compression and case-depth

Glass fractures from the application of a high tensile stress acting on a (generally) surface flaw. The flaw tip acts as a stress concentrator and the surrounding environment affects the crack velocity.

ASTM C-1422

(2) Test strength

Apply tensile stress in a defined geometry till specimen fails. Statistics of handling flaws often requires a group of specimens to be broken. Strength performance can still vary at the hands of a consumer.
Fig. 2. (a) Dark-field photoelastic fringe patterns as viewed through the (b) unstrengthened surface of the Ion-Armor™ glass using white light in a circular polariscope. Also shown is (c) an enlarged image of the bottom edge of the unstrengthened face which illustrates the large number of fringes and increasing fringe density toward the edge. Note that the camera was positioned and focused on the bottom edge of the sample to eliminate perspective foreshortening.

Fig. 3. Optical micrograph of the isochromatic fringe patterns as viewed through the unstrengthened face using a 10× objective and a circular polariscope (dark-field arrangement) with a green optical bandpass filter centered on 550 nm. A total of 15 whole fringes in the compressive region and two whole fringes in the tensile region are observed.

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Strength Tests

• Bird-shot
• Ball drop tests
• Tests to meet hurricane code
• Ring-on-ring or 4-point strength testers
• Abrasion resistance

https://www.youtube.com/watch?v=8ObyPq-OmO0
Ball Drop Test

The typical ball test for strengthened glass is to drop a 128 gram steel ball from a height of 1 meter onto the surface of the glass.

With increasingly demanding requirements for cover glass, we've made the test for Corning® Gorilla® Glass even tougher.

Our latest innovations, including Gorilla® Glass 2 and advanced finishing technologies have been put to the test by increasing drop height by 80%.

Using a Ball that is greater than 4 times heavier.
Important Applications of chemically strengthened glass

- Aircraft cockpit windshields
- Display windows in mobile personal electronic devices (e.g., mobile phone, MP3 players, iPad)
- Autoinjector glass cartridges for emergency antidote against bee-stings, peanuts and other allergens that cause one to go into anaphylactic shock
Aircraft cockpit windshield

**FEDERAL AVIATION REGULATIONS**

- Home > Aviation Regulations > Parts Index > Part 25 > Sec. 25.775 - Windshields and windows.
- **Sec. 25.775 — Windshields and windows.**
  - (a) Internal panes must be made of nonsplintering material.
  - (b) Windshield panes directly in front of the pilots in the normal conduct of their duties, and the supporting structures for these panes, **must withstand, without penetration, the impact of a four-pound bird when the velocity of the airplane (relative to the bird along the airplane's flight path) is equal to the value of \( V_C \), at sea level, selected under §25.335(a).**
  - (c) Unless it can be shown by analysis or tests that the probability of occurrence of a critical windshield fragmentation condition is of a low order, the airplane must have a means to minimize the danger to the pilots from flying windshield fragments due to bird impact. This must be shown for each transparent pane in the cockpit that—
    - (1) Appears in the front view of the airplane;
    - (2) Is inclined 15 degrees or more to the longitudinal axis of the airplane; and
    - (3) Has any part of the pane located where its fragmentation will constitute a hazard to the pilots.
  - (d) The design of windshields and windows in pressurized airplanes must be based on factors peculiar to high altitude operation, including the effects of continuous and cyclic pressurization loadings, the inherent characteristics of the material used, and the effects of temperatures and temperature differentials. The windshield and window panels must be capable of withstanding the maximum cabin pressure differential loads combined with critical aerodynamic pressure and temperature effects after any single failure in the installation or associated systems. It may be assumed that, after a single failure that is obvious to the flight crew (established under §25.1523), the cabin pressure differential is reduced from the maximum, in accordance with appropriate operating limitations, to allow continued safe flight of the airplane with a cabin pressure altitude of not more than 15,000 feet.
  - (e) The windshield panels in front of the pilots must be arranged so that, assuming the loss of vision through any one panel, one or more panels remain available for use by a pilot seated at a pilot station to permit continued safe flight and landing.

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The “chicken cannon”
• Ionex® Strengthened glass cartridge in the EpiPen
Saxon Glass Technologies, Inc.
Pharmaceutical autoinjector cartridges

EpiPen Type I
Borosilicate
glass cartridge
before and after
chemical
strengthening

Cumulative failure probability

Load-at-Failure (lb)

Unstrengthened

IONEX®

Raw Set 1  Raw Set 2  Raw Set 3
× Ionex® Set 1  × Ionex® Set 2  ● Ionex® Set 3

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\[ Q = \ln[\ln(1/(1-F))] \]

\[ F = \text{cumulative failure probability} \]

Failure probability reduced to less than 1 ppm

Weibull Plot

\[ y = 5.63x - 29.74 \]
\[ y = 6.25x - 33.39 \]
\[ y = 6.36x - 33.45 \]
\[ y = 5.92x - 37.70 \]
\[ y = 6.18x - 39.54 \]
\[ y = 4.98x - 32.08 \]

\[ Q = \ln[\ln(1/(1-F))] \]

\[ F = \text{cumulative failure probability} \]

Failure probability reduced to less than 1 ppm
IONEX®

1996 sales 1M units
2017 sales 30 M units

Helps save human lives during a life-threatening emergency event...
### Thin glasses as cover screens for personal mobile electronic devices

<table>
<thead>
<tr>
<th>Physical Property</th>
<th>Units</th>
<th>SINGLE CRYSTAL</th>
<th>Gorilla Glass&lt;sup&gt;TM&lt;/sup&gt; (Corning)</th>
<th>Xensation&lt;sup&gt;T M&lt;/sup&gt; (Schott)</th>
<th>Dragontrail&lt;sup&gt;T M&lt;/sup&gt; (AGC)</th>
<th>Unstrengthened Soda lime glass</th>
<th>Silica glass</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density</td>
<td>g/cm³</td>
<td>3.97</td>
<td>2.42</td>
<td>2.48</td>
<td>2.48</td>
<td>2.5</td>
<td>2.2</td>
</tr>
<tr>
<td>Young's modulus</td>
<td>GPa</td>
<td>345</td>
<td>71.5</td>
<td>74</td>
<td>74</td>
<td>73</td>
<td>73</td>
</tr>
<tr>
<td>Mean flexural strength</td>
<td>MPa</td>
<td>895</td>
<td>800-900*</td>
<td>700-800*</td>
<td>750-850*</td>
<td>40</td>
<td>50</td>
</tr>
<tr>
<td>Shear modulus</td>
<td>GPa</td>
<td>145</td>
<td>29.6</td>
<td>30</td>
<td>30</td>
<td>30</td>
<td>31</td>
</tr>
<tr>
<td>Fracture toughness</td>
<td>MPa√m</td>
<td>2.3</td>
<td>0.68</td>
<td>---</td>
<td>----</td>
<td>0.7-0.8</td>
<td>0.66</td>
</tr>
<tr>
<td>Knoop hardness</td>
<td>GPa</td>
<td>18.6</td>
<td>6.17</td>
<td>6.26</td>
<td>6.95</td>
<td>6.03</td>
<td>4.9</td>
</tr>
<tr>
<td>Vickers hardness</td>
<td>Kg/mm²</td>
<td>2200</td>
<td>649</td>
<td>681</td>
<td>673</td>
<td>580</td>
<td>630</td>
</tr>
<tr>
<td>Dielectric constant</td>
<td>k</td>
<td>9.39</td>
<td>7.23</td>
<td>6.7</td>
<td>----</td>
<td>7.75</td>
<td>3.82</td>
</tr>
<tr>
<td>Refractive index</td>
<td>n</td>
<td>1.76</td>
<td>1.51</td>
<td>1.52</td>
<td>1.51</td>
<td>1.52</td>
<td>1.45</td>
</tr>
</tbody>
</table>
Alfred University Develops Stronger Glass for Use in Mobile Appliances

Have you ever dropped your cell phone or accidentally hit your MP3 player against something and broken its display glass? Now researchers at the Kazuo Inamori School of Engineering at Alfred University in New York have found a way to strengthen glass used in mobile appliances so they will be able to withstand falls, bumps and hits without breaking.

The development is the result of research conducted by graduate student Jeff Olin; his research advisor, Arun Varshneya, professor of Glass Science & Engineering; and employees at Saxon Glass Technologies Inc., a business housed in Alfred's Ceramic Corridor Innovation Center.

“We believe that the strengthened glass can withstand handling much better than other glass products,” Varshneya says.

The professor/entrepreneur anticipates a growing market for cell phones in Brazil, Russia, India and China, based on predictions that half the world’s population will possess cell phones before the end of the next decade. “We know the demand will be about a billion of these strengthened glass windows a year,” he says.

Arun Varshneya, professor of Glass Science & Engineering at Alfred University and also the president of Saxon Glass Technologies.
Recent developments in Science & Technology of Ion-Exchange Strengthening

• Buildup and relaxation of stress during ion exchange
• Analogy to indentation science; effect of glass topology
• Engineered stress profile. “ESP”
• High-surface compression/FAST technologies
• Warp control of thin float-produced glasses
• Automobile windshield

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“The Dilation Anomaly” (1975)

\[ B = \frac{1}{3} \ln \left( \frac{V_2}{V_1} \right) / C \]

For ion exchange in 15Na_2O.10CaO.75SiO_2 glass:

- \( V_1 \) is the molar volume of 15Na_2O.10CaO.75SiO_2 glass = 24.55 cm³
- \( V_2 \) is the molar volume of 15K_2O.10CaO.75SiO_2 glass “CEAM” = 26.61 cm³
- \( C = 15\% \)
- \( B = 0.0019 / \text{mol}\% \)

Biaxial stress \( \sigma = - \frac{BEC}{1 - \nu} = -2.5 \text{ GPa} \)

Measured stress is usually only about -500 MPa.

Where have all the pascals gone?
Molecular dynamics simulation of a 6,000 atom cube at 723K and various boundary conditions of NPT and NVT. 15 Na$_2$O.10CaO.75SiO$_2$
Expansion of glass upon ion exchange

CEAM = “compositionally equivalent as-melted”
Biaxial stress build-up and relaxation

Fraction of picoseconds after ion exchange

2.6 GPa

Relaxation time few picoseconds

2.1 GPa

Relaxation time few nanoseconds

1.5 GPa

Stress-driven viscous flow hours/days at ion exchange temperature

1.2 GPa

Thermal contraction loss after cooling to ambient
Tyagi/Varshneya 1998 "Laboratory reported zero-time stress"

0 MPa

Stress-free Parent glass “Blue Volume”

0 MPa

Stress-free “Orange volume”

0 MPa

Stress-free CEAM “Green volume”

2018 GOMD Morey Award Lecture
A. Tandia, K. D. Vargheese, J. C. Mauro

2018 GOMD Morey Award Lecture
Warp of thin glasses

*“Tin side”* has ~10 wt% Sn (over a couple of microns).

*“Air side”* has near-zero.

*Interdiffusion coefficient of $K^+ \leftrightarrow Na^+$ is different for the two surfaces.*

• *Tin side has lesser penetration $\Rightarrow$ Lesser compression $\Rightarrow$ net negative bending moment of the air side about the mid-plane $\Rightarrow$ the air side develops convexity to counteract the negative bending moment from ion exchange.*

WARP !!

• *If the glass is thin, warp is measurable.

Warp is undesirable for proper lamination of various layers in personal mobile electronic devices such as cell phones.

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Example of Differential spray method

“Strengthened glass and methods for making using differential density”

Vary the availability of the exchange medium

1. As-received coupon
2. Coat both surfaces with salt + clay of different thickness
3. Chemically strengthen (both sides) in air

Tin side
Air side

Coupon with improved flatness

2018 GOMD Morey Award Lecture
Warp reduction

KNO₃ bath treatment

Grid = 2 mm

Competition →

Saxon process; patent pending
Non-contact profiler scans across 50 mm soda lime silicate float glass coupon 0.4 mm thick

\[ CS = 630 \text{ MPa}; \quad \text{DOL} = 11 \mu\text{m} \]

Caution: x-axis scale = 5000 microns; y-axis scale = 20 microns
Engineered stress profile ("ESP")
D. J. Green, V. Sglavo, R. Tandon

Strength variation would decrease

The future

* More efficient and faster ion exchange strengthening processes
* Pharma glass container applications
* Hurricane-resist architectural windows
* Automobile applications
* Thin glass substrates for solar energy harvesting
* Armor development
Analogy of chemical strengthening process to indentation process

• Indenter is like a big ion….  
• During indentation, the glass is sheared and densified (both yield strengths are exceeded)
• Upon retracting, there is a permanent shape change; stresses appear in the vicinity
• Likewise, during ion exchange, the larger ion shears the interstitial space and densifies it.
Influence of network topology
...according to Varshneya...ICG 2007

• Optimized connectivity leads to a maximization of shear yield strength; not much effect on dilatational yield strength.

• Elastic deformation is more in \( <r> = 2.40 \) solids.

• Hence, surface compression should be higher in such solids.

• High alkali content glasses have high NBO, hence, low \( \sigma_s \).
Prediction

- Alkali containing glasses that show lesser deformation during microindentation should chemically strengthen better.
- Chemical composition is such that they are optimally connected networks.
Super-CS technology


- Electric-Field-assisted process, Commercializable, Improve glass surface compression and case depth, less than 1 hour process time
- Two step process is more useful.

It’s electric!

<table>
<thead>
<tr>
<th>Sample Set</th>
<th>Surface Compression Average (MPa)</th>
<th>Case Depth Average (micron)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gorilla3/Super-CS</td>
<td>1140</td>
<td>9.5</td>
</tr>
<tr>
<td>SLS/Super-CS</td>
<td>796</td>
<td>19.2</td>
</tr>
<tr>
<td>SLS/Reference 1</td>
<td>688</td>
<td>6.2</td>
</tr>
<tr>
<td>SLS/Reference 2</td>
<td>486</td>
<td>17.8</td>
</tr>
</tbody>
</table>
Large-missile resistant for building located < 30’ above ground level.
2”x4”x6’ lumber ~ 9 pounds, fired at speed of 50 fps (34 mph). Impact should not create hole larger than 1/16” x 5" in the interlayer of the glass.
Must pass positive and negative wind loads for 9,000 cycles.

Small-missile resistant
10 ball bearings speed of 80 fps (50 mph). The product is then subjected to wind loads for 9,000 cycles.
Automobile windshield glass

M. Dejneka, GOMD
Madison WI 2016

- 2.1 mm Annealed Soda Lime

- 2.1 mm Annealed Soda Lime

1.75” (44mm) Ice Ball Impacting Windshield Laminate at 72mph

Test Results

<table>
<thead>
<tr>
<th>Test Case</th>
<th>Result</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.1 ASLG / 2.1 ASLG</td>
<td>Break</td>
</tr>
<tr>
<td>2.1 ASLG / 1.0 ASLG</td>
<td>Break</td>
</tr>
<tr>
<td>2.1 ASLG / .7 GG</td>
<td>No Break</td>
</tr>
<tr>
<td>2.1 ASLG / .55 GG</td>
<td>No Break</td>
</tr>
</tbody>
</table>

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Surface compression $\sim 1$ GPa
Case-depth $\sim 1$ mm
Antimicrobial Glass: Aqueous IOX uses less Ag\(^{+}\), keeps it at the surface to kill germs, and maintains full mechanical strength!

M. Dejneka, GOMD Madison WI 2016
Nagging issues.....

(1) Surface relaxation to tension in a mixed-alkali glass

Donald & Hill 1988
Robert Schaut & Melodie Schmidt & experiment

<table>
<thead>
<tr>
<th>Glass RAS Composition</th>
<th>Mole Percent</th>
</tr>
</thead>
<tbody>
<tr>
<td>Li₂O</td>
<td>30.00</td>
</tr>
<tr>
<td>MgO</td>
<td>10.00</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>10.00</td>
</tr>
<tr>
<td>P₂O₅</td>
<td>1.00</td>
</tr>
<tr>
<td>SiO₂</td>
<td>50.00</td>
</tr>
</tbody>
</table>

Performed in-situ NaNO₃ stress measurement during exchange at 385°C

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(2) Hardness increase well beyond neutral line

Data of Jannotti *et. al.*
On Ion-Armor™ Glass

Why is hardness increase observed as deep as 2.4 mm?
Oxide glasses containing alkali ions can be chemically strengthened to as high as 1000 MPa with 200 – 1,000 µm DOL.

Alkali aluminosilicates are the better glasses for chemical strengthening.

Applications: Display (thin); armor (thick)

- Small amount of impurities can reduce the surface compression and case depth significantly.
- The science of stress build-up and relaxation is close to being understood!
- Market for pharma container applications is likely to grow.
- Hurricane-resist glass, armor, automotive applications and solar energy harvesting substrates are around the corner.
Guardian Industries for sponsorship of the Morey Award

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Thank you....