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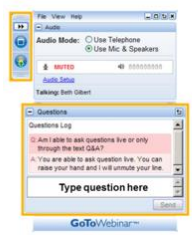

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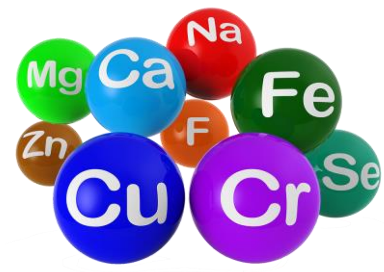

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2016 Material Science Series

http://bit.ly/2016MaterialScienceSeries

Thursday, February 4, 2016

The Chemistry of Hello: Transparent Circuitry

Tobin Marks, Professor of Catalytic Chemistry and Professor of Material Science and Engineering, Department of Chemistry, Northwestern University

Mark Jones, Executive External Strategy and Communications Fellow, Dow Chemical

* You are already signed for all 3 webinars in the "Chemistry of Hello" mini-series up so just save the date!

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Upcoming ACS Webinars®

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Thursday, January 21, 2016

"Is Your Etiquette Holding Back Your Career?"

Patricia Simpson, Owner/Consultant, Game Changing Etiquette
Director of Academic Advising and Career Services, University of Illinois

David Harwell, Assistant Director of Industry Member Programs, American Chemical Society

Thursday, January 28, 2016

2016 Drug Design and Delivery Symposium:
Drug-Target Kinetics in Drug Design

Robert Copeland, President of Research and Chief Scientific Officer, Epizyme, Inc.

Daniel Erlanson, Co-founder and President, Carmot Therapeutics, Inc.

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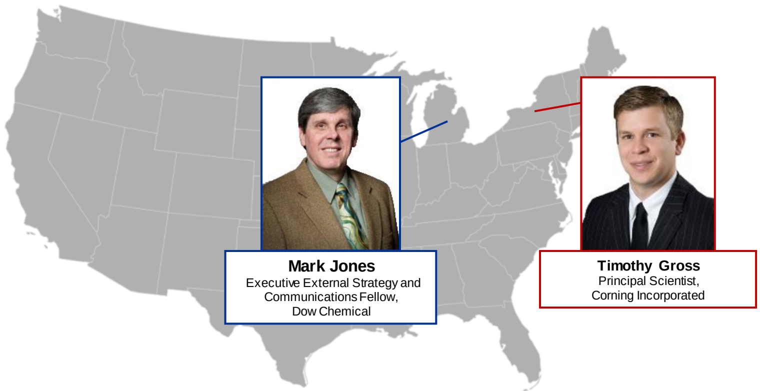
2016 Material Science Series

"The Chemistry of Hello: Making the Glass of Tomorrow"



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CORNING

Making the Glass of Tomorrow

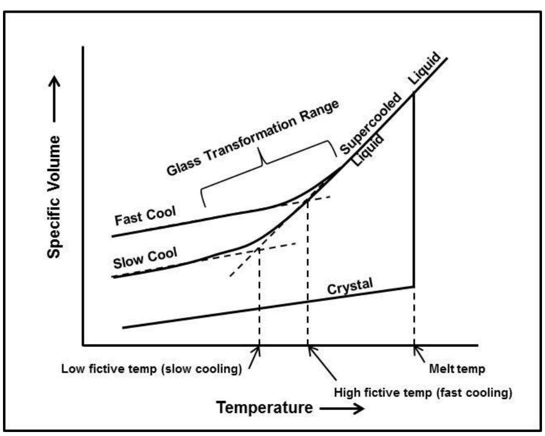
Timothy M. Gross, Ph.D.
Principal Scientist
Corning Incorporated Science and Technology Division

1/14/16

Overview

- Kinetic theory of glass formation – why do glasses form?
- Structure of basic alkali aluminosilicate glass
- Monovalent alkali ion-exchange and development of stress profile
- Fracture mechanics and damage resistance of Gorilla Glass

Description of the Glassy State



- At temperatures between the melting temperature and the glass transformation range, a super-cooled liquid will be transformed into the crystalline state if given enough time to do so.
- If the liquid is cooled sufficiently rapidly so that the atoms or molecules in the liquid have no time to rearrange to form a crystal, then the liquid becomes a glass.
- In principle, any material can be turned into a glass if it is cooled rapidly enough from the liquid state, however, some material sets much more readily form glasses than others.
- Depending on the cooling rate, glass can have different volume. The faster the quenching rate, the greater the volume. The temperature at which the glass structure is frozen-in is called the fictive temperature.

Kinetic Theory of Glass Formation

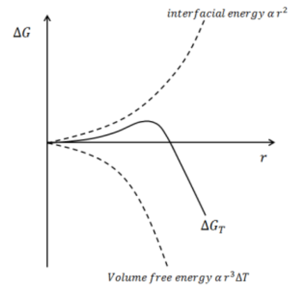
Review crystallization process to understand glass formation.

Crystallization involves nucleation and growth processes.

Nucleation involves energetically competing terms:

$$\Delta G_T = \underbrace{\frac{4}{3}\pi r^3 \Delta G_V}_{\text{Product of the volume of a nucleus and the free energy decrease per unit volume}} + \underbrace{4\pi r^2 \gamma_{SL}}_{\text{Product of the new surface area and surface energy per unit area}}$$

$$\Delta G_V = \frac{L_V \Delta T}{T_m}$$



For a nucleus to become stable it needs to attain a critical size, otherwise it will dissolve back into the melt

At $\frac{\partial \Delta G_T}{\partial r} = 0$ the critical radius is achieved where $r^* = -\frac{2\gamma_{SL}}{\Delta G_V}$ and the energy barrier to nucleation is given by $\Delta G_T{}^* = \frac{16\pi}{3}\frac{\gamma^3}{(\Delta G_V)^2}$

Kinetic Theory of Glass Formation

Molecules in a liquid are constantly fluctuating, some of them join together to temporarily form a crystal. This process can lead to the formation of a nucleus with a size greater than the critical size.

The number of nuclei of critical radius r^*, $n_r{}^*$, and the total number of molecules in the system, n_o, are related by Boltzmann statistics:

$$n_r{}^* = n_o exp\left(-\frac{\Delta G_T{}^*}{kT}\right)$$

The rate of nucleation, I (number of nuclei formed per unit time in a unit volume) is given by:

$$I = \nu n_S n_r{}^*$$

Where ν is the frequency of one molecule successfully joining the critical nucleus to make it stable and n_s is the number of molecules facing the critical nucleus.

$$\nu = \nu_o \exp\left(-\frac{\Delta E_D}{kT}\right)$$

Where ν_o is the molecular jump frequency and ΔE_D is the activation energy for transport across the nucleus-matrix interface.

Kinetic Theory of Glass Formation

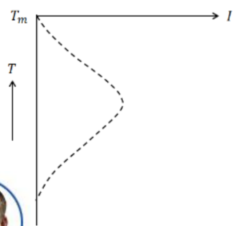
The transport process in the melt is controlled by viscosity, η, which is reciprocally proportional to the frequency of molecular jumping, ν:

$$\nu \; \alpha \; \frac{1}{\eta} \qquad \eta = \eta_o exp\left(\frac{\Delta E_\eta}{kT}\right)$$

ΔE_η is the activation energy for viscous flow and is equal to ΔE_D

Thus the rate of nucleation can be written as:

$$I = \frac{K_1}{\eta_o} exp\left(-\frac{\Delta G_T{}^*}{kT}\right) exp\left(-\frac{\Delta E_\eta}{kT}\right)$$



At high temperatures near the melting point, the driving force for crystallization approaches zero as ΔG_T* approaches infinity.

Recall: $$\Delta G_T{}^* = \frac{16\pi}{3}\frac{\gamma^3}{(\Delta G_V)^2} \qquad \Delta G_V = \frac{L_V \Delta T}{T_m}$$

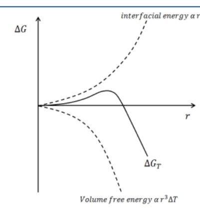
At low temperatures, the viscosity becomes extremely high, also making the rate of nucleation low.

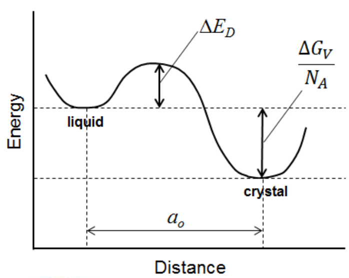
Kinetic Theory of Glass Formation

Crystal Growth

Once a stable nucleus is formed, i.e. the radius is large enough to be on the right hand side of the ΔG_T curve, a crystal can grow.

During crystal growth, the energy of the material is reduced as molecules in the liquid state join the growing crystal.





Frequency of a molecule going from liquid to crystal

$$\nu_{lc} = \nu_o exp\left(-\frac{E_D}{kT}\right)$$

Frequency of a molecule going from crystal to liquid

$$\nu_{cl} = \nu_o \exp\left(-\frac{\Delta E_D}{kT}\right) \exp\left(-\frac{\Delta G_V}{N_A kT}\right)$$

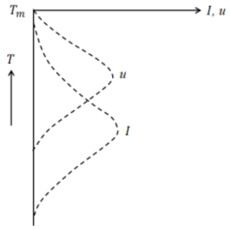
Crystal growth rate by size a_o is then:

$$u = a_o(\nu_{lc} - \nu_{cl})$$

Again, using the inversely proportional relationship between jump frequency and viscosity the rate of nucleation equation is derived:

$$u = \frac{K_2}{\eta_o} exp\left(-\frac{\Delta E_\eta}{kT}\right)\frac{\Delta G_V}{RT}$$

Nucleation and Growth



$$u = \frac{K_2}{\eta_o} exp\left(-\frac{\Delta E_\eta}{kT}\right)\frac{\Delta G_V}{RT}$$

$$I = \frac{K_1}{\eta_o} exp\left(-\frac{\Delta G_T{}^*}{kT}\right) exp\left(-\frac{\Delta E_\eta}{kT}\right)$$

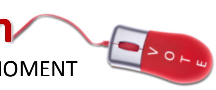
- Typically the temperature at the maximum rate of nucleation is lower than the temperature at the maximum rate of crystal growth.
- In order for crystallization to take place, both nucleation and growth need to take place at a reasonable rate.
- If the activation energy for viscous flow is large, both nucleation and growth rates become slow.
- Materials with large $\Delta E\eta$ like SiO_2 and B_2O_3 will tend to form glass easily.
- Understanding of nucleation and growth kinetics in glass have resulted in several important Corning inventions in the glass-ceramic material space. One example is Corningware.

material	T_m (K)	ΔE_η at T_m (kJ/mole)
SiO_2	2007	753
B_2O_3	723	167
H_2O	273	21
Na	371	6.3
LiCl	886	33.5
Benzene	278	3.4

From M. Tomozawa Textbook of Glass Science 2008

Audience Survey Question

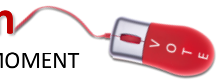
ANSWER THE QUESTION ON BLUE SCREEN IN ONE MOMENT



Which material most readily forms a glass when cooling from the molten state?

- H_2O
- SiO_2
- ZrTiCuNiBe (Vitreloy 1 metallic glass composition)
- Benzene

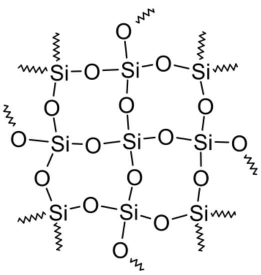
Audience Survey Question



ANSWER THE QUESTION ON BLUE SCREEN IN ONE MOMENT

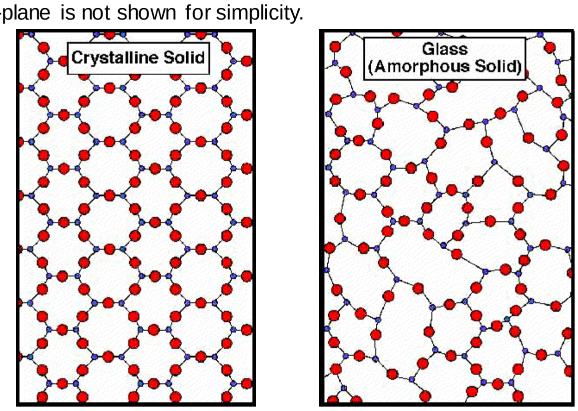
Which material most readily forms a glass when cooling from the molten state?

- SiO_2



Glass vs. Crystals

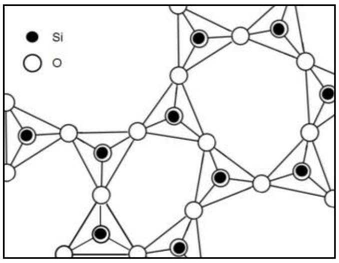
2D schematic representation of crystalline and amorphous silica. The simplified planar representation of the silica tetrahedra only show 3 in-plane oxygens per tetrahedron. The fourth out-of-plane is not shown for simplicity.



- Silica glass consists of the same structural units (tetrahedra) as in the crystalline forms.
- While both types of solids have the same local structure, glass do not possess long range order as do crystals.

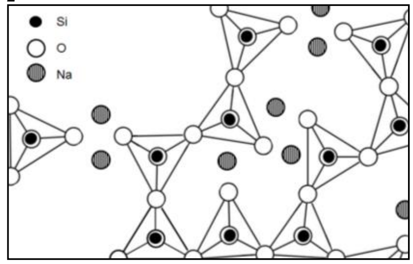
Silica vs. Sodium Silicate Glass

Silica glass



Silica glass modified with sodium

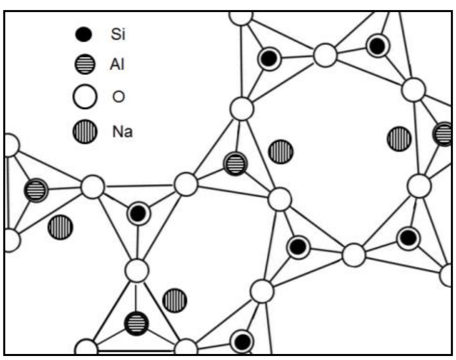
$$Na_2O + \equiv Si-O-Si\equiv \;\Rightarrow\; \equiv Si-O^-Na^+ \quad Na^+O^- - Si\equiv$$



- Adjacent tetrahedral units are connected by shared oxygens (referred to bridging oxygens).
- Tetrahedra are only linked at corners (not edges) and each oxygen is linked to only two silicon cations.
- The silica glass network has high connectivity resulting in high viscosity and free volume.

- Glass modifiers can be added to the silicate glass network to change glass properties, e.g. viscosity, density, refractive index, Young's modulus, etc
- Glass modifiers are not considered good glass formers, so would require extreme quench rates to form amorphous structures if used as the primary constituent. However, a limited amount can be added to a glass former while maintaining good glass forming capability.
- Incorporation of sodium oxide into the SiO_2 network creates non-bridging oxygens (NBOs), thus breaking up the connectivity of the silica tetrahedral network.
- Incorporation of NBOS leads to a lower viscosity, but more highly packed network

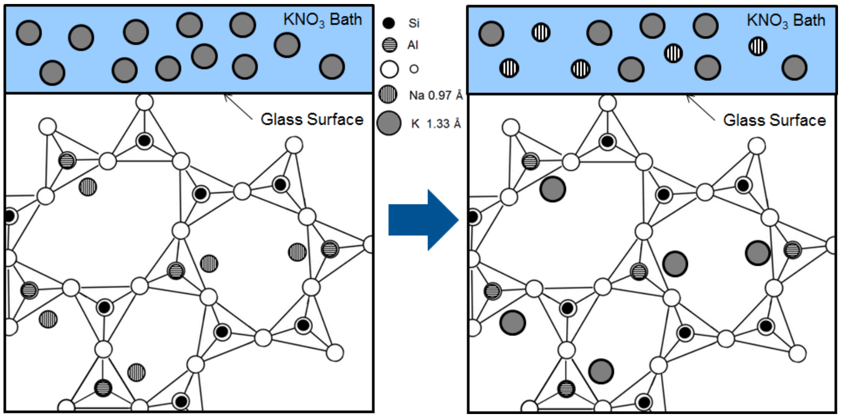
Addition of Intermediate Al_2O_3



- Al_2O_3 can be added to the glass composition and substitutes into tetrahedral positions (Na^+ charge compensates the Al^{3+}, so that it acts as a 4+ cation).
- If Al_2O_3 and NA2O are added on a 1:1 basis, the network is again free of non-bridging oxygens.
- This increases the viscosity and forms a more open network structure when compared to sodium silicate glass with large concentrations of NBOs.

Basic ternary Na_2O-Al_2O_3-SiO_2 system is the basis for highly ion-exchangeable glasses including Corning® Gorilla® Glass.

Ion-exchange Diffusion of Monovalent Cations



- Glass is immersed in a molten salt bath at temperatures where stress relaxation is minimal, yet diffusion rate is suitable for processing time.
- Ion-exchange processing of alkali aluminosilicates is typically performed at ~400°C, substantially less than the strain pt. of these glasses (550 to 650°C).
- Strain pt. is a reference temperature defined at $\eta = 10^{13.7}$ Pa s, where stress relaxation occurs over the course of hours.

Ion-Exchange Diffusion Equations

Interionic Diffusion Coefficient, $\widetilde{D}$, is increased with open network structures, smaller diffusing ions:

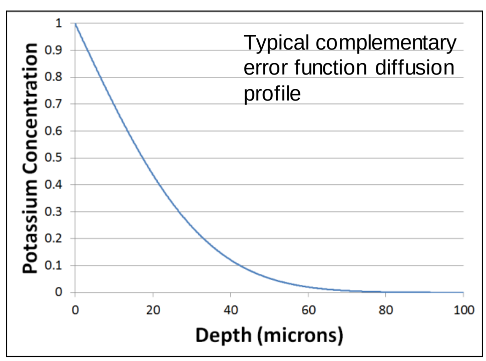
$$\widetilde{D} = \frac{D_{K^+} D_{Na^+}}{N_{K^+} D_{K^+} + N_{Na^+} D_{Na^+}}$$

D_i is the diffusion coefficient of species i
N_i is the mole fraction of species i

Concentration of K+ as function of position and time:

$$\frac{C(z) - C_o}{C_S - C_o} = \mathrm{erfc}\left(\frac{x}{2\sqrt{\widetilde{D}t}}\right)$$

$C(z)$ is the concentration of invading ion at depth z.
C_s is the concentration of the invading ion at the surface.
C_o is the initial bulk concentration of the invading ion.



Generation of Ion-Exchange Stress Profile

The concentration of the exchanging K^+ ion at depth z produces a strain, ε. The constant of proportionality between the concentration change and strain is referred to as the lattice dilation coefficient, B.

$$\varepsilon = B(C(z) - C_o)$$

For an isotropic, linear elastic material like glass, the stress in the x and y directions at depth z is given by the following expression:

$$\sigma(z) = \frac{BE}{1-\nu}\left(C_{avg} - C(z)\right)$$

E : Young's modulus
ν : poisson's ratio

The above stress equation is a simplified form assuming zero stress relaxation, i.e. we are operating at an ion-exchange temperature where η of the glass is high relative to the strain pt. viscosity of $10^{13.7}$ Pa s). A stress relaxation function takes the following form:

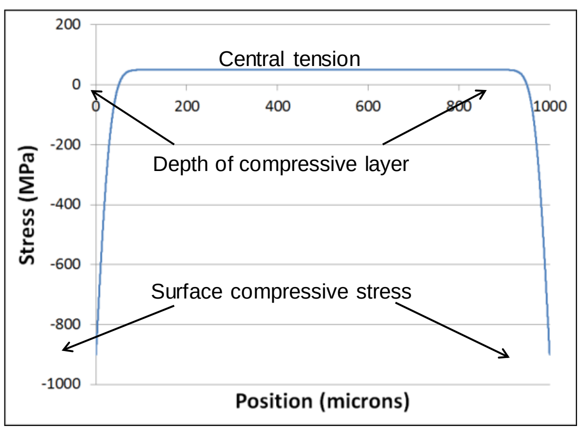
$$\sigma_t = \sigma_o exp\left(-\frac{Gt}{\eta}\right)$$

G: Shear modulus

Typical Ion-Exchange Stress Profile

+ tensile stress

- compressive stress



- The stress profile takes on an complementary error function form since it follows the potassium diffusion profile.
- The resulting compressive stress shown extending from the surface to the depth of compressive layer is balanced by the stored central tension.
- The compressive stress is important since a surface flaw cannot extend to failure under compression.

Audience Survey Question

ANSWER THE QUESTION ON BLUE SCREEN IN ONE MOMENT

What mathematical function is typically used to describe the diffusion and stress profiles in ion-exchanged glasses?

- Parabola
- Linear fit
- Complementary error function
- Half Gaussian

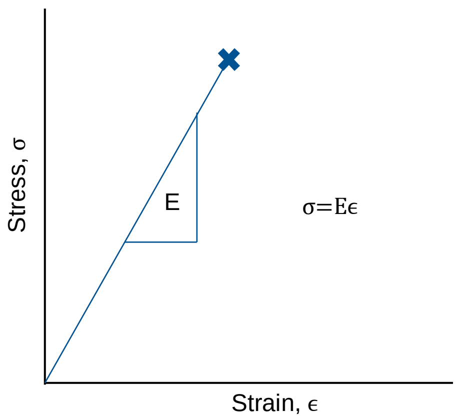
Audience Survey Question

ANSWER THE QUESTION ON BLUE SCREEN IN ONE MOMENT

What mathematical function is typically used to describe the diffusion and stress profiles in ion-exchanged glasses?

- Complementary error function

Basics of Glass Fracture



- Bulk glass deformation is entirely elastic. Unlike many other materials, a permanent deformation mechanism is not available.
- Glass only fails under tension.
- The stress at failure is not an instrinsic property and will vary from sample to sample.
- Based on the strong covalent/ionic bonding in oxide glass the theoretical strength is extremely high (exceeding 14 GPa).
- In practice, the strengths are much lower (0.014 to 0.14 GPa).
- The practical strength of glass is an extrinsic property that depends on the surface quality. A geometric discontinuity such as a surface crack locally amplifies the stress.

Basics of Fracture Mechanics

The stress intensity at a surface flaw is given by the following equation:

$K_I = Y\sigma\sqrt{a}$

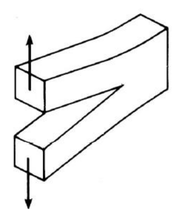
Y is a dimensionless parameter that depends on both specimen and crack geometries

σ is the applied stress

a is the crack depth

When the stress intensity, K_I, reaches a critical value, K_{IC}, instantaneous failure occurs.

Mode I crack opening displacement



K_{IC} , also known as the fracture toughness. Unlike the failure stress for glass, fracture toughness is an intrinsic material property.

For oxide glasses the fracture toughness is in the range of 0.6 – 0.9 MPa$\sqrt{m}$

Audience Survey Question

ANSWER THE QUESTION ON BLUE SCREEN IN ONE MOMENT

The strength of glass is: ____________________?

- An intrinsic property determined through calculation of bond strength
- An extrinsic property dependent on stress concentration at surface flaw locations

Audience Survey Question

ANSWER THE QUESTION ON BLUE SCREEN IN ONE MOMENT

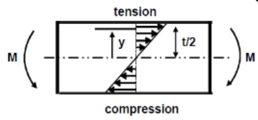
The strength of glass is: ____________________?

- An extrinsic property dependent on stress concentration at surface flaw locations

Example case where ion-exchange greatly improves the surface strength for glasses containing small flaws (allows glass flexibility)

Consider bend induced stress on a 1 mm thick glass plate:

$$\sigma_{bend} = E\frac{t}{2R}$$



Recall: Glass can only fail under tension!

$K_I = Y\sigma_{bend}\sqrt{a}$ $\quad Y = 0.65\sqrt{\pi}$ for semicircular "half-penny" shaped crack

$$K_I = 0.65\sigma_{bend}\sqrt{\pi a}$$

Using, E = 75 GPa, K_{IC} = 0.7 MPa$\sqrt{\mathrm{m}}$, and a = 1 micron, we can calculate the bend stress and bend radius required to exceed critical stress intensity for non-strengthened glass:

$$\sigma_{bend} = \frac{0.7MPa\sqrt{m}}{0.65\sqrt{\pi*10^{-6}m}} = 607 \text{ MPa}$$

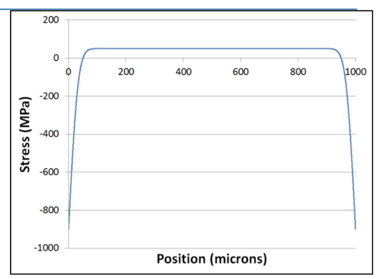
$$R = \frac{75000MPa*10^{-3}m}{2*607MPa} = 0.062\text{m} = 6.2 \text{ cm}$$

If we now ion-exchange the same glass plate, we allow for bending to much tighter bend radii

Now we need to also consider the ion-exchange stress:

$$K_I = 0.65(\sigma_{bend} + \sigma_{IOX})\sqrt{\pi a}$$

We still require 607 MPa of tensile stress to reach the critical stress intensity of 0.7 MPa$\sqrt{\mathrm{m}}$, but now we require 1507 MPa of bend induced stress to overcome the surface compressive stress of -900 MPa.

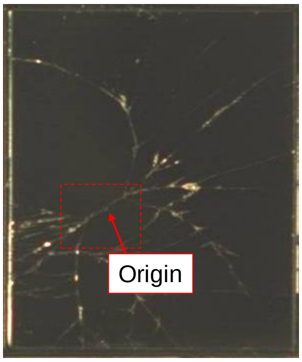


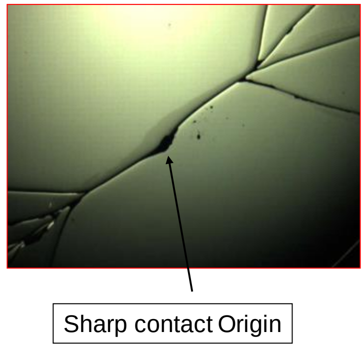
The bend radius now required to exceed critical stress intensity for non-strengthened glass:

$$R = \frac{75000MPa*10^{-3}m}{2*1507MPa} = 0.025\text{m} = 2.5 \text{ cm}$$

- The applied bend induced stress to failure is 2.5X greater for ion-exchanged glass.
- In terms of bend radius, the ion-exchanged glass can be taken to a 2.5X tighter bend.
- Flexible ion-exchanged glass may be a critical component in new flexible display technologies.

For the cover glass application we see that the failure mode is local sharp contact that generates large flaw that penetrated the depth of compressive layer



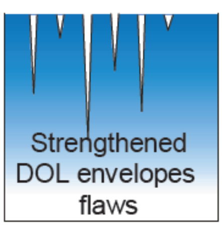


- Sharp contact deformation is defined by the glass response. It occurs when the contact load is distributed over small contact area and elastic limit is exceeded resulting in permanent deformation (only under highly local deformation does this occur).
- Strength limiting flaw formation initiates within the permanent deformation region.
- Crack extension to failure occurs as contact flaws extend through the depth of compressive layer (typically associated with small global bending stress).

By designing glass with capability to be ion-exchanged to deep depth of compressive layer we can keep large flaws under compression

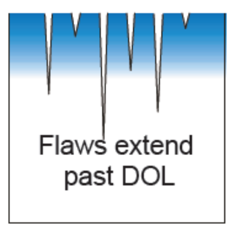
Corning® Gorilla® Glass is designed to have superior surface compressive stress and deeper depth of compressive layer than normal soda-lime silicate glass.

Typical surface flaws are well contained in the compressive layer for Corning® Gorilla® Glass



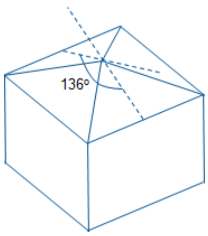
CS ~900 MPa
DOL ~50 microns

Typical surface flaws easily penetrate the compressive stress layer for soda-lime

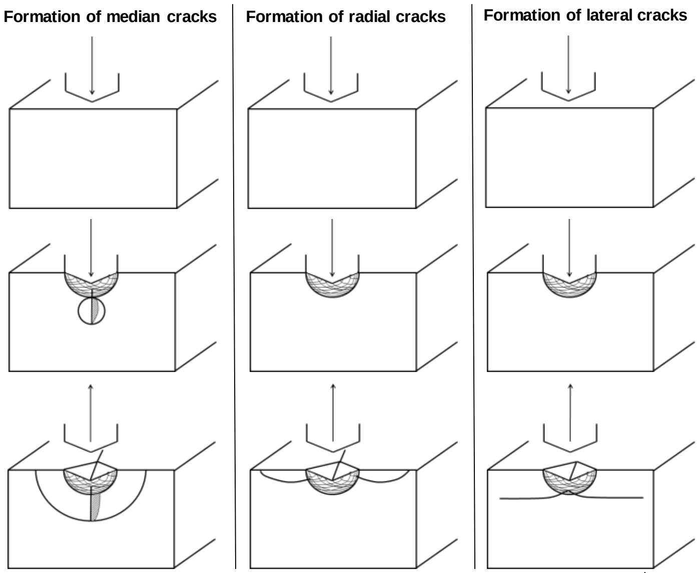


CS ~650 MPa
DOL ~10 microns

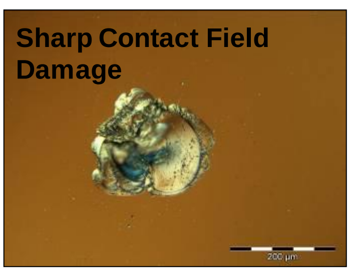
Using diamond indentation to mimic large flaw formation from sharp contact

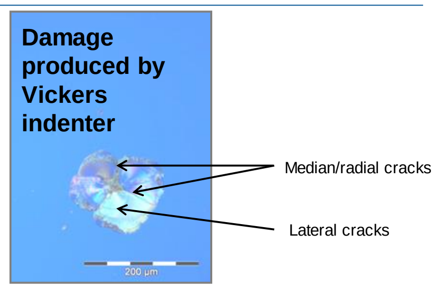


The Vickers diamond indenter is a 4-sided pyramid with angle between opposite faces = 136°

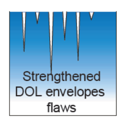


Comparison of field damage with damage produced by a Vickers indenter

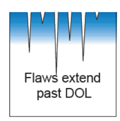




To create flaws that penetrate the depth of compressive layer requires a Vickers indentation load of ~10 kgf for ion-exchanged alkali aluminosilicate with CS = 900 MPa and DOL = 50 microns.

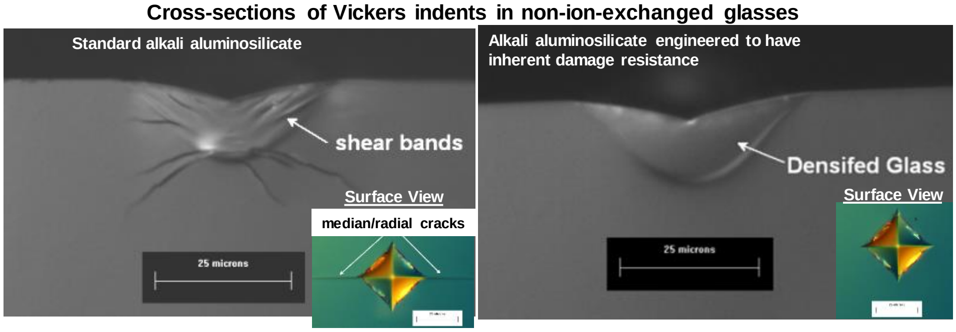


For ion-exchanged soda-lime silicate with CS = 650 MPa and DOL = 10 microns, the load required to puncture the depth of compressive layer is less than 1 kgf.



Since the local sharp contact and the resulting large flaw formation is the primary failure mode in cover glasses, Corning has used compositional understanding to improve the inherent crack resistance of the base glass.

Cross-sections of Vickers indents in non-ion-exchanged glasses



- Glass compositions designed to have a combination of optimal free volume and network connectivity will permanently deform under a sharp contact without creating the subsurface damage that lead to glass failure.
- When ion-exchanged, the glass with high inherent damage resistance requires Vickers indentation loads exceeding 30 kgf to create a flaw that penetrates the depth of compressive layer.

Summary

- The glass kinetics of formation overview provided the theory of glass formation and describes why glasses form from a melt.
- The structure of a simple sodium aluminosilicate system was described since this type of glass is the basis for highly ion-exchangeable glass compositions.
- Ion-exchange diffusion was described to understand the mechanism by which larger potassium ions exchange with smaller sodium ions and create a compressive stress profile.
- Fracture mechanics was briefly described and used to show the strength improvement (improved flexibility) for ion-exchanged glasses over non-ion-exchanged glass.
- The primary failure mode of cover glass (sharp contact) was described and it was shown how stress profile and glass design is used to prevent this failure mechanism.
  - Research is ongoing at Corning to further improve the strength and damage resistance of Corning® Gorilla® Glass through optimization of compressive stress profile and mechanical properties of the glass.
  - The ultimate goal that we are moving towards is the virtually unbreakable glass.

Thank you for your attention!

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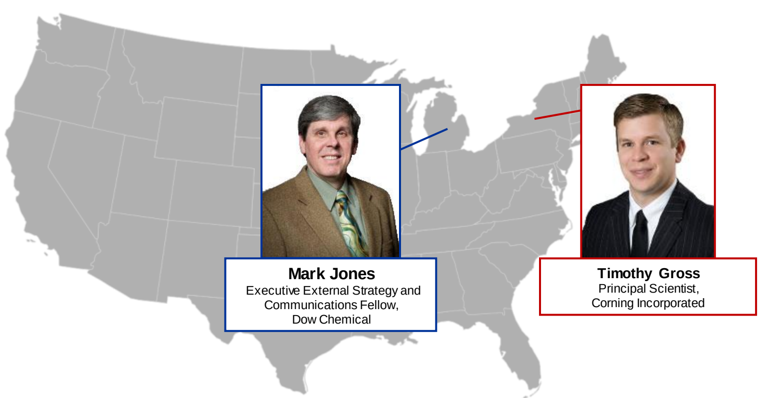

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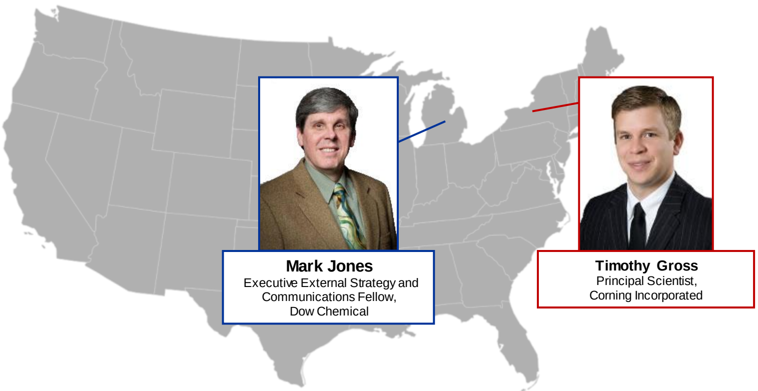

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