

Processing and Properties of Ion Exchanged Glasses

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Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Security Administration under Contract DE-ACO4-94AL85000.





Recent Severe Hailstorm in New Mexico















Truck Bomb Attacks on Federal Buildings Have Focused Attention on Glass Behavior



**1995- Murrah Building,
Oklahoma City, OK**



1996- Khobar Towers, Saudi Arabia



**Embassy in Nairobi
August 7, 1998 Truck Bomb**



**Embassy in Dar Es Salaam
August 7, 1998 Truck Bomb**



Glass Fragment Character and Size, and Glass Strength Depend on Processing

Plate Glass- 5 kg Indent

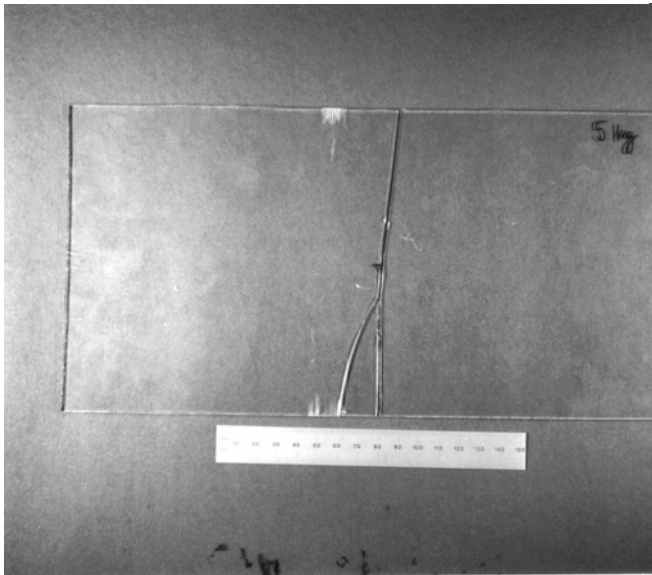
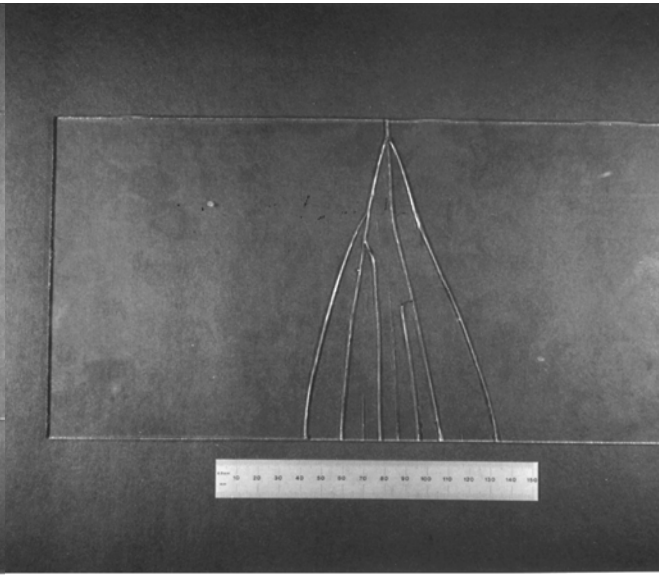
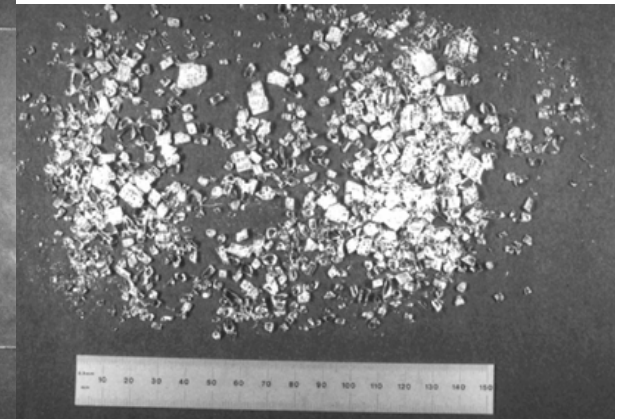


Plate Glass - As-Rec'd



Ion Exchanged





Video

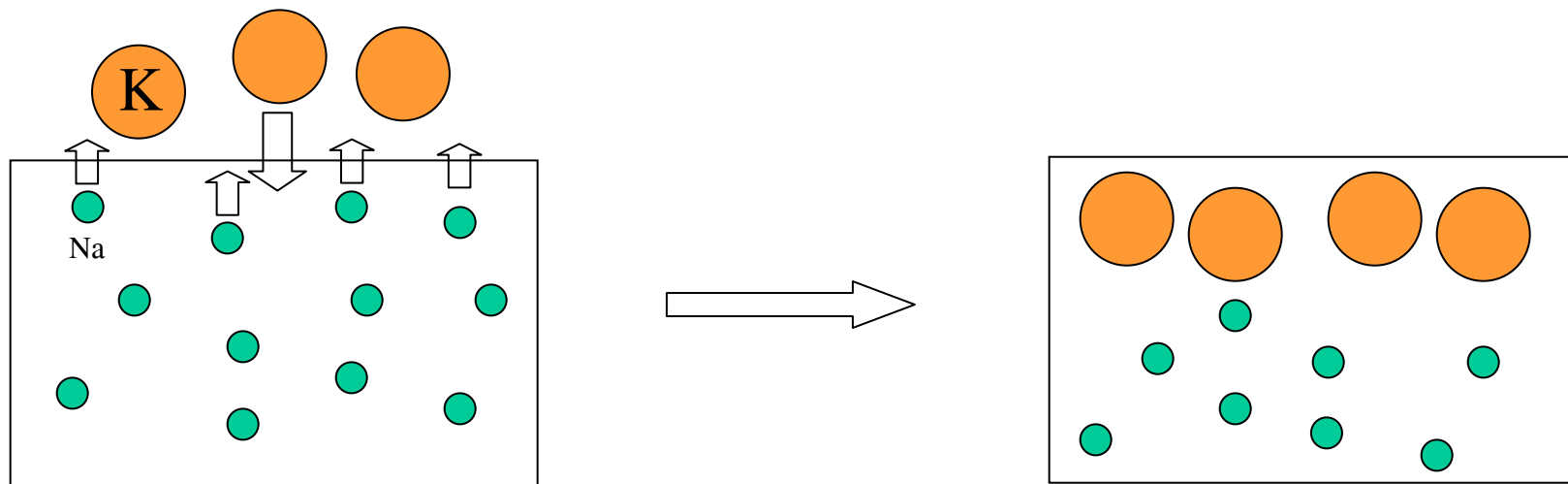
To view the video showing the breaking glass sheet for
standard glass compared to ion exchanged glass

use the link on the IMI-NFG website or

<http://rm1.cc.lehigh.edu:8080/dept/TMI/EdVideo/RealTime&SloMoGlassBreakage.mpg>



Ion Exchange Process Replaces Small Ions in Glass Surface with Large Ions

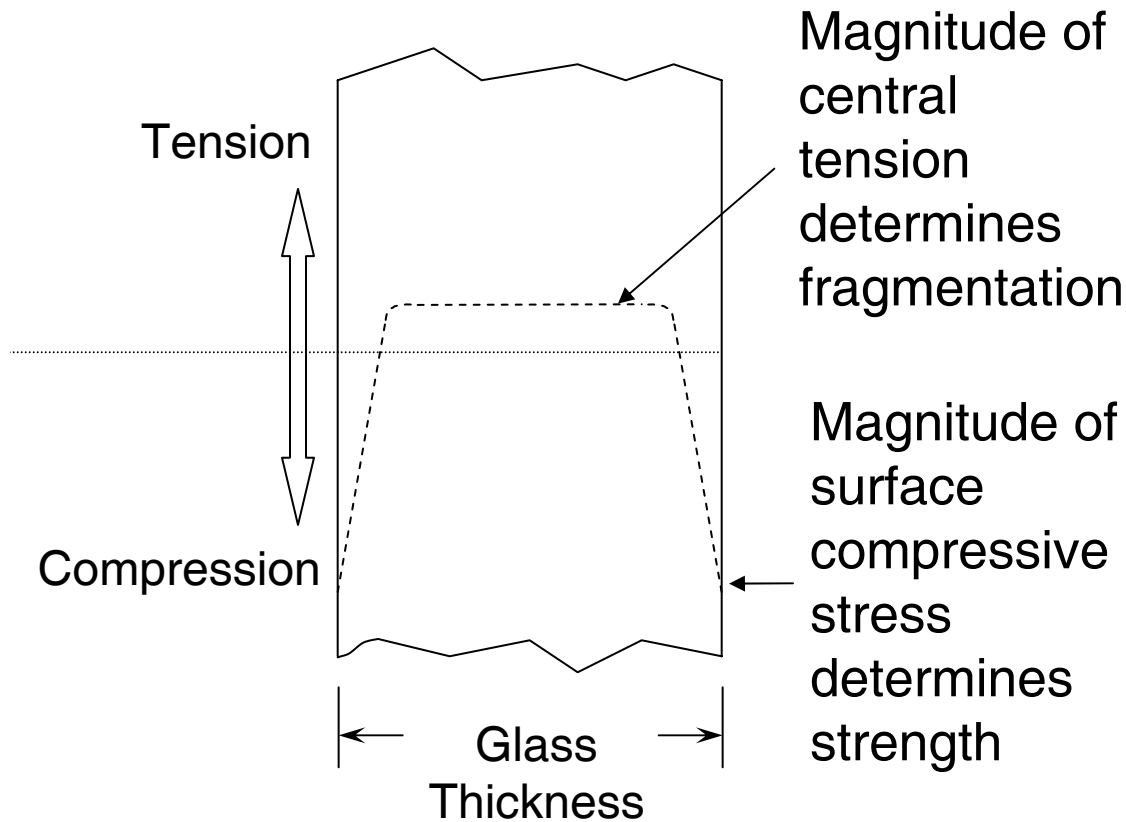


Exchange of a Na-containing glass in a bath of molten KNO₃ salt

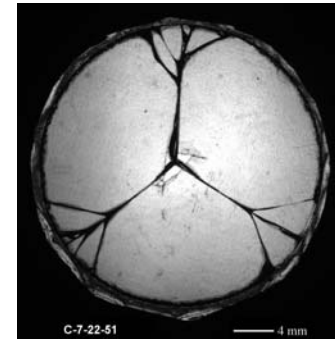


Ion Exchange Stress Profile Increases Glass Strength and Fragmentation

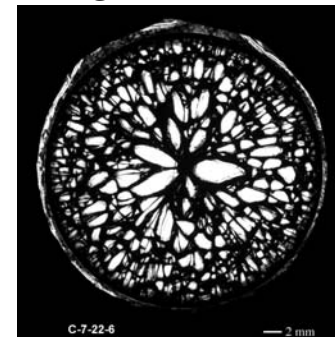
Stress profile = - - - - -



Disk diameter=1 inch



12 hr exchange
Strength = 759 MPa

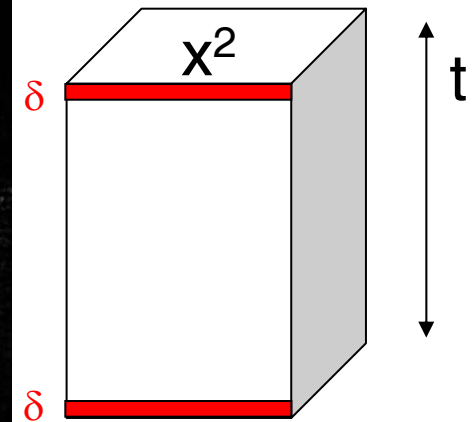
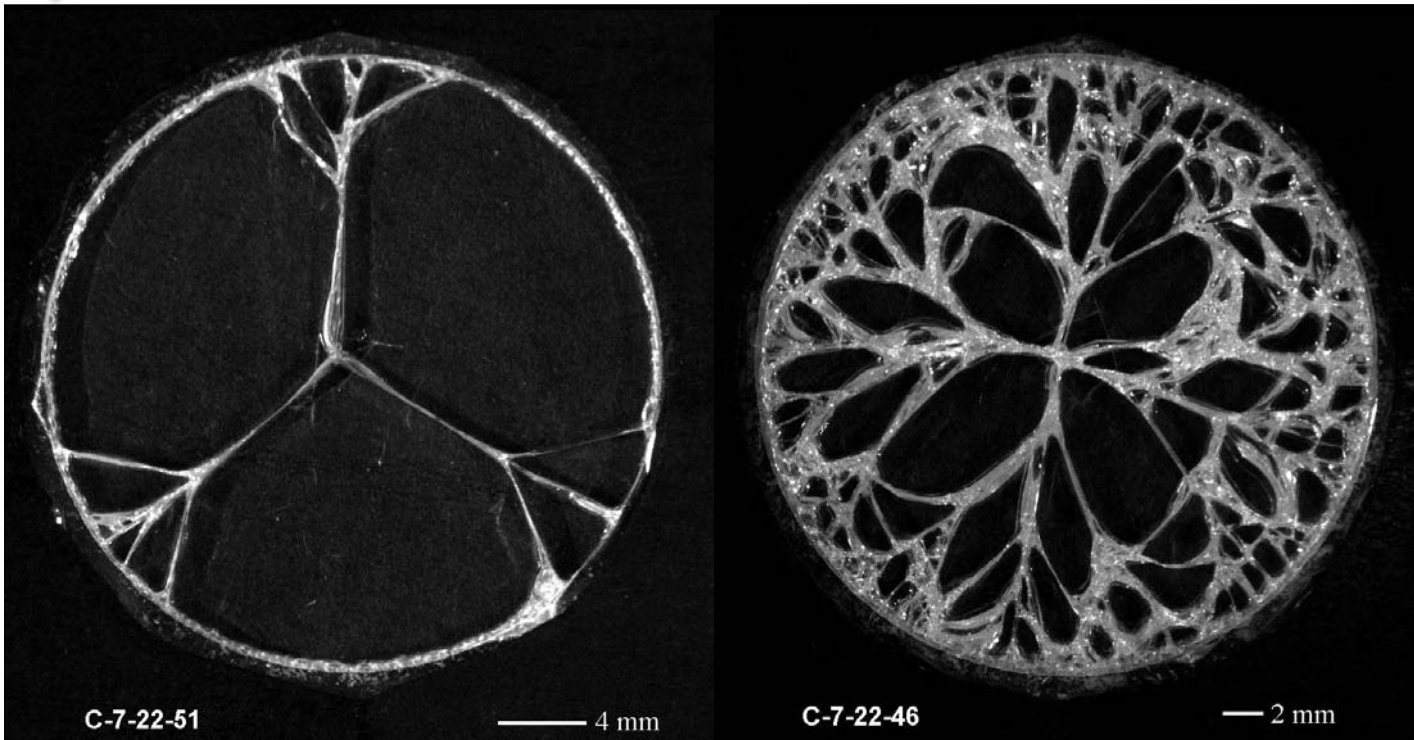


48 hr exchange
Strength = 706 MPa

The more a crack **branches** as it propagates the greater the degree of **fragmentation**.



Fragment Size Largely Determined by Central Tension, σ_t and Crack Branching Behavior



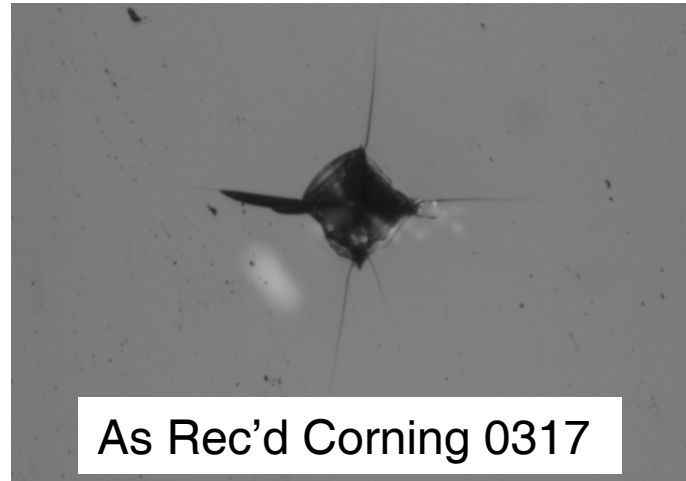
Fragment size,
$$x = \frac{K_{1c}^2}{\sigma_t^2} (1 + \nu) \frac{t}{(0.5t - \delta)}$$

K_{1c} =toughness, t =thickness, δ =ion exchange depth

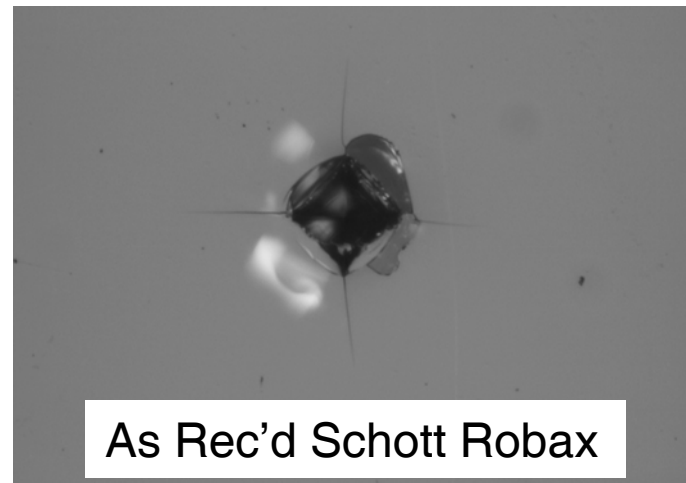


Schott Robax and Corning 0317 Glass Exchanged at 450 & 500C for Different Times

Wt% of oxides	Corning 0317	Schott Robax
SiO ₂	62.3	67.2
Na ₂ O	12.8	0.4
K ₂ O	3.5	0.23
CaO	0.3	0.05
MgO	3.3	1.1
Al ₂ O ₃	16.4	20.1
Fe ₂ O ₃	0.24	
TiO ₂	0.75	2.7
SnO ₂	0.04	
Li ₂ O		3.2
BaO		0.9
As ₂ O ₃		0.9
ZrO ₂		1.7
ZnO		1.7
Total	99.53	99.92



$H_V = 5.95 \pm 0.19$ GPa
 $K_{IC} = 0.82 \pm 0.03$ MPa√m



$H_V = 6.44 \pm 0.19$ GPa
 $K_{IC} = 1.09 \pm 0.03$ MPa√m





Comparison of Amount of Ion Being Exchanged

Corning Constituents	Wt%	Mol%
SiO ₂	61.96	65.33
Na ₂ O	11.88	12.14
K ₂ O	3.41	2.29
CaO	0.41	0.46
MgO	3.34	5.24
Al ₂ O ₃	16.50	10.25
Fe ₂ O ₃	0.08	0.03
TiO ₂	0.67	0.53
Li ₂ O	1.75	3.71
Total	100.00	100.00

Robax Constituents	Wt%	Mol%
SiO ₂	67.20	72.92
Na ₂ O	0.35	0.37
K ₂ O	0.23	0.16
CaO	0.05	0.06
MgO	1.06	1.71
Al ₂ O ₃	20.10	12.85
TiO ₂	2.65	2.16
Li ₂ O	3.15	6.87
BaO	0.88	0.37
As ₂ O ₃	0.87	0.29
ZrO ₂	1.73	0.92
ZnO	1.65	1.32
Total	99.9	100.0



Indentation Behavior of Exchanged Schott Glass Show Improved Damage Tolerance

R-11-19-11

R-11-19-22

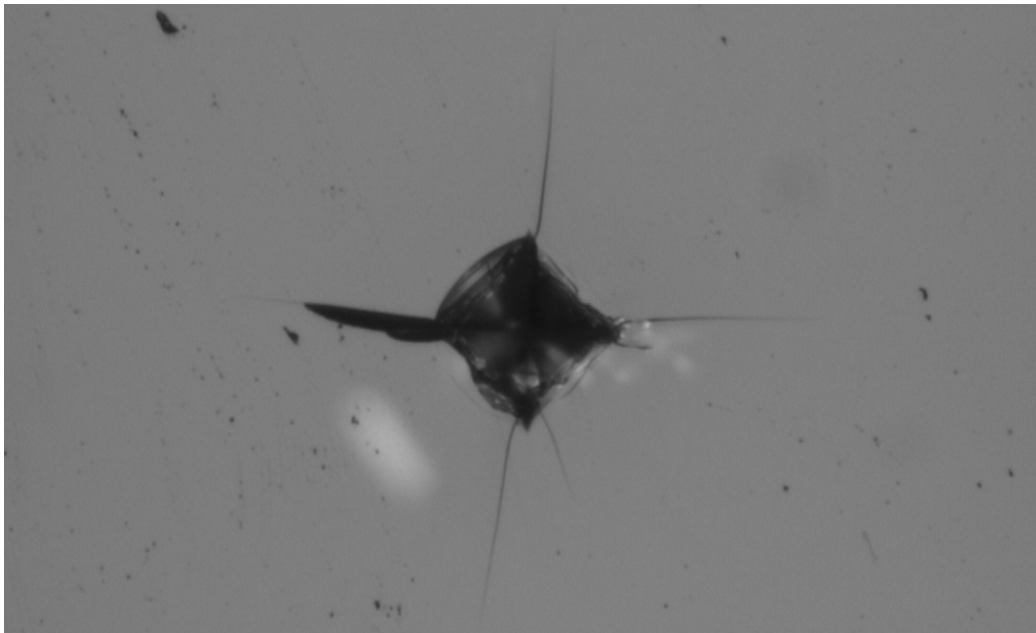


As received, 2000 g indent

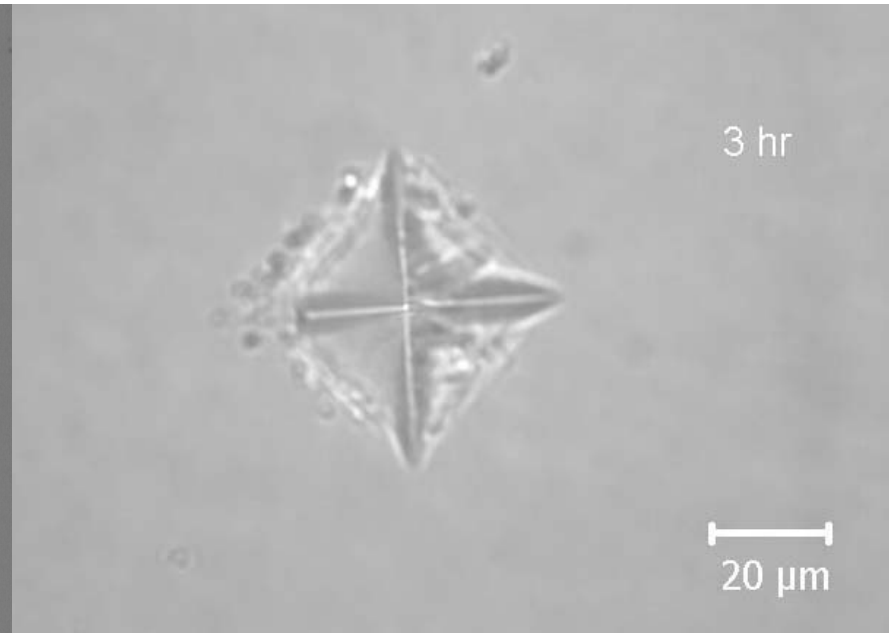
Exchanged at 500C, 6 hr, 2000 g



Indentation Behavior of Exchanged Corning 0317 Glass Shows Improved Damage Tolerance



As-Rec'd

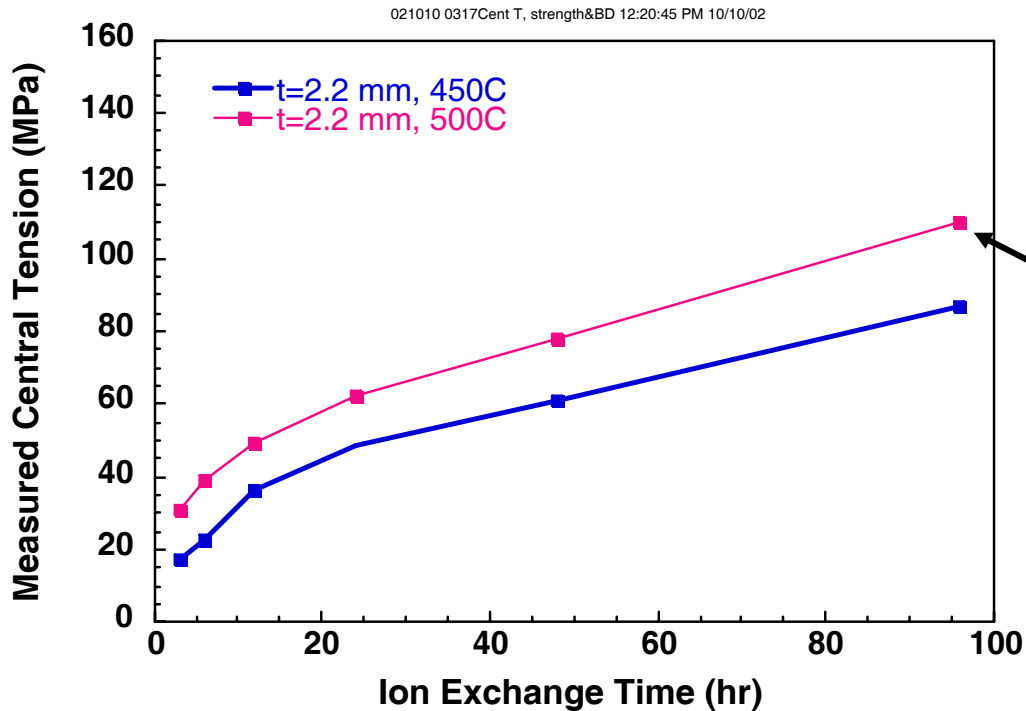


Exchanged at 450°C/3 hr

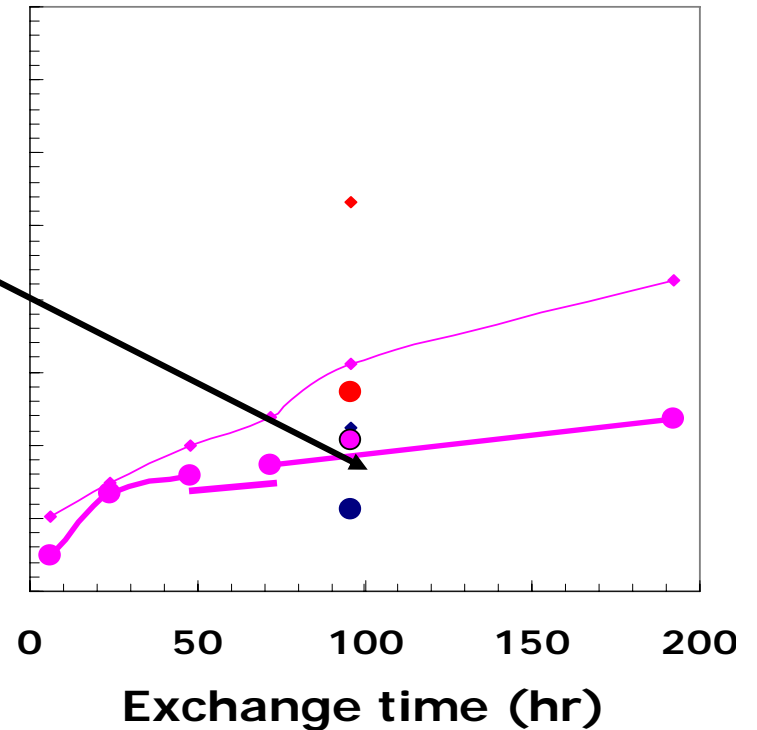


Central Tension as a Function of Ion Exchange Temperature and Glass Thickness

Corning 0317



Schott Robax

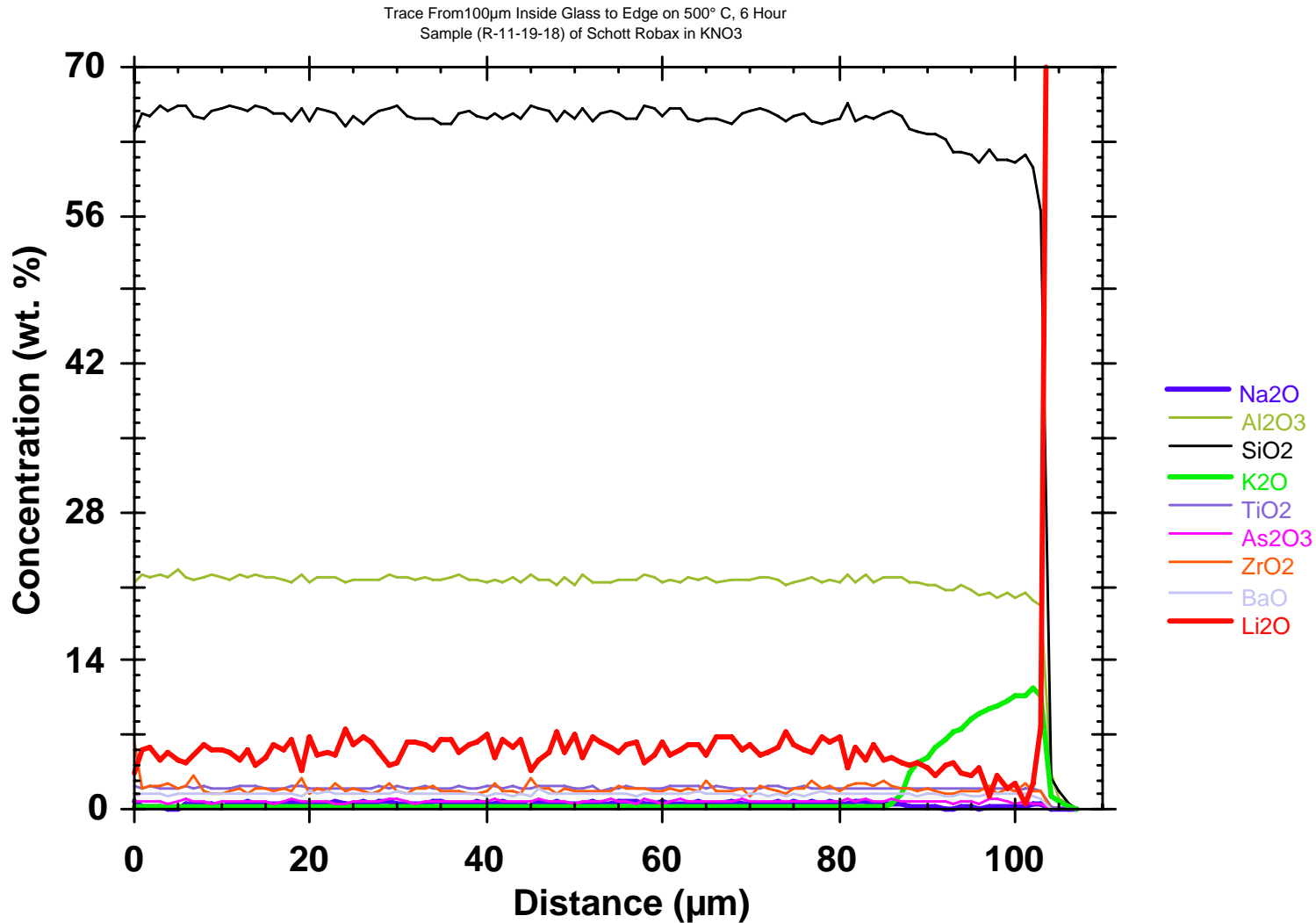


Arrow joins same ion exchange conditions (t & T) and glass thickness. 110 MPa vs. 42 MPa.





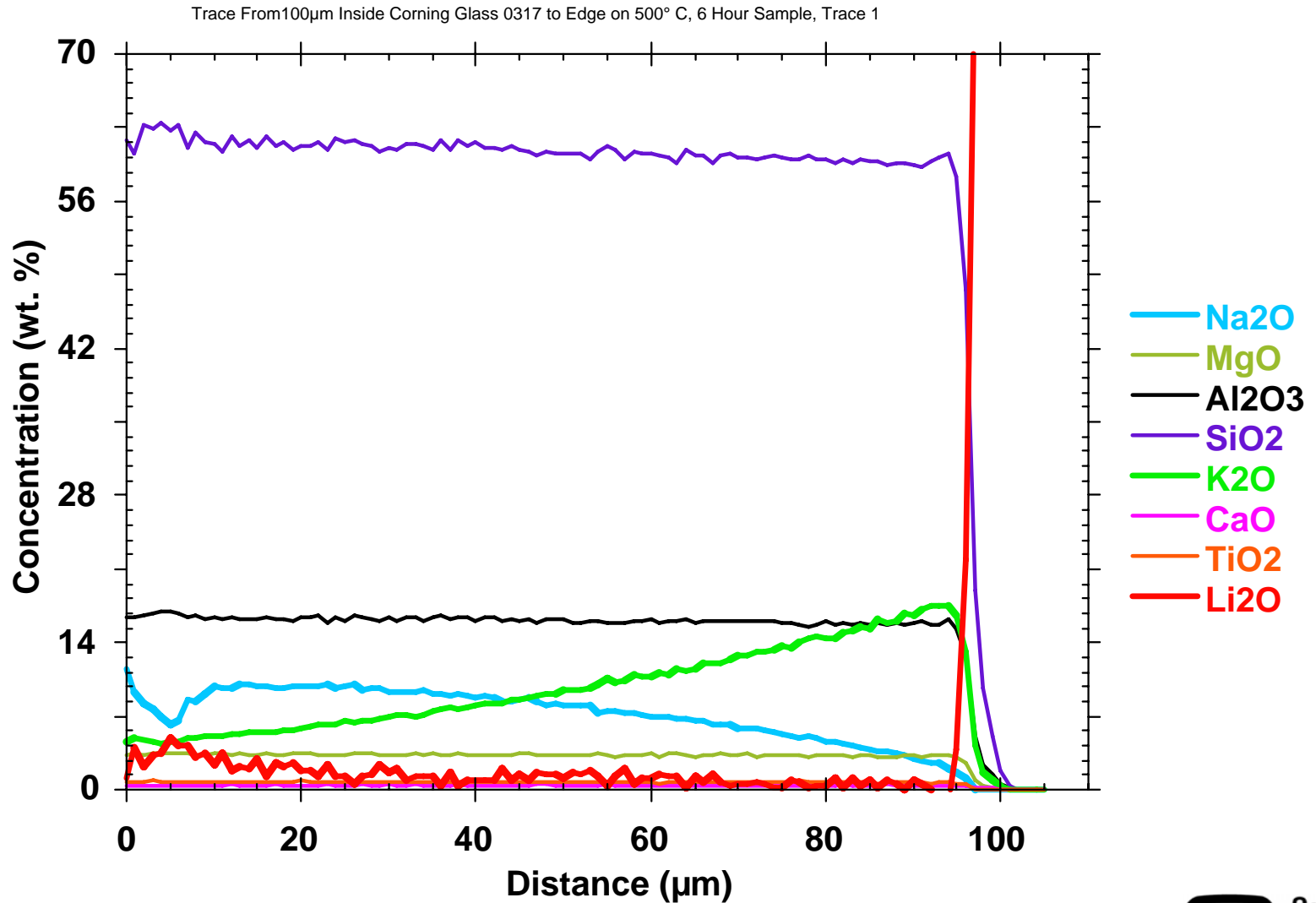
K Ion Exchange Depth for Schott Robax- 6 hr @500C



K penetration depth ~ 20 microns.



K Ion Exchange Depth for Corning 0317- 6 hr @500C



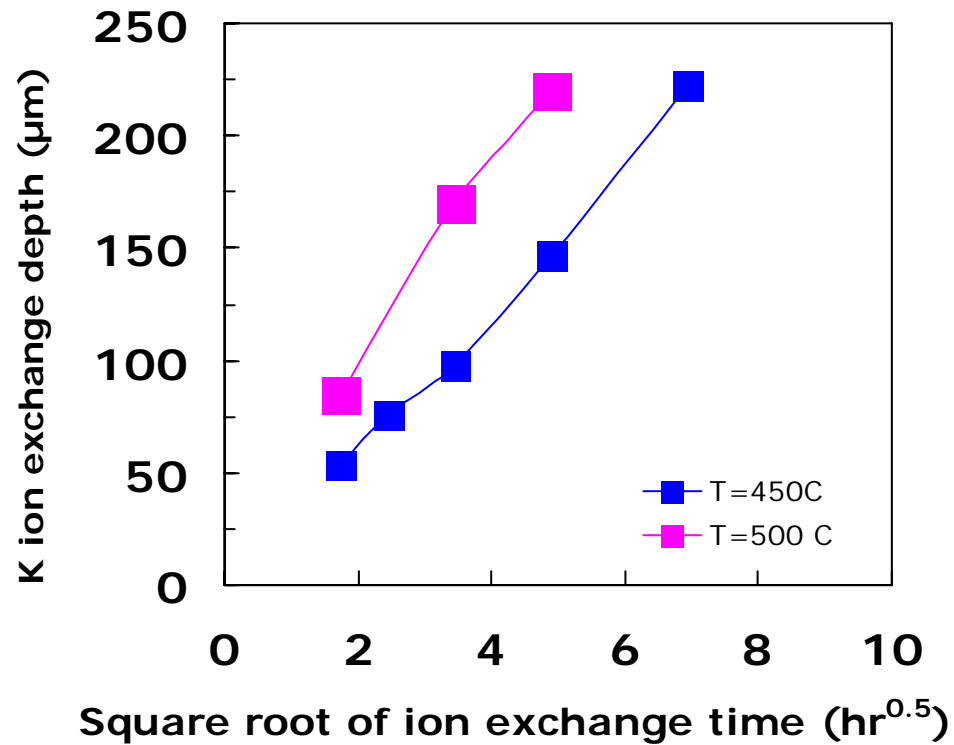
K penetration depth ~ 90 microns.



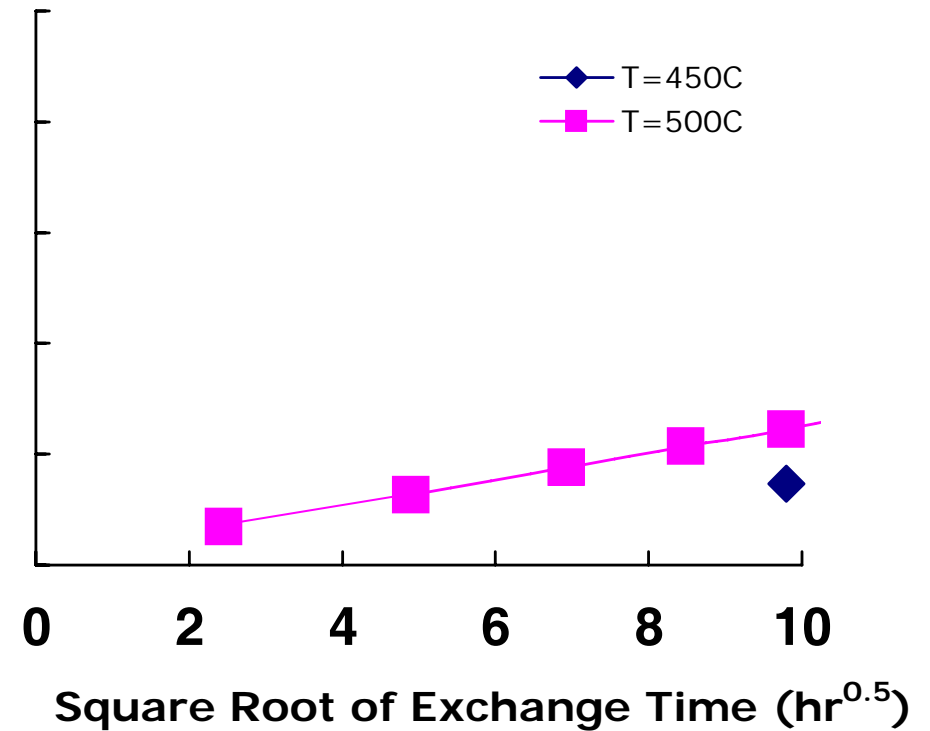


Schott Robax Has a Much Smaller Ion Exchange Depth than Corning 0317 for the Same T and t

Corning 0317



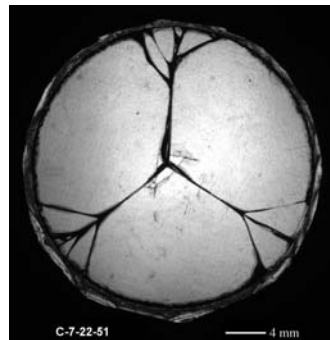
Schott Robax





Comparison of Critical Central Tensions for Crack Branching in 1" Diameter Disk

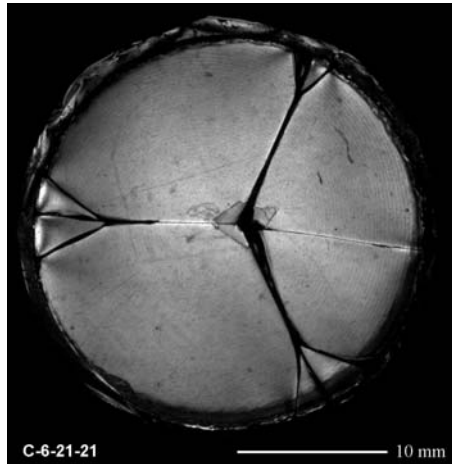
	Measured Values for Critical Central Tensions (MPa) for Branching in 1" Disks				K_{IC} (MPa√m) (Indentation)
	t=1.0	t=1.2	t=1.8	t=2.2	
Thick.▶ Glass ▼					
Corning 0317	42		40	34	0.82
Schott Robax		~ < 47		~ < 47	1.09





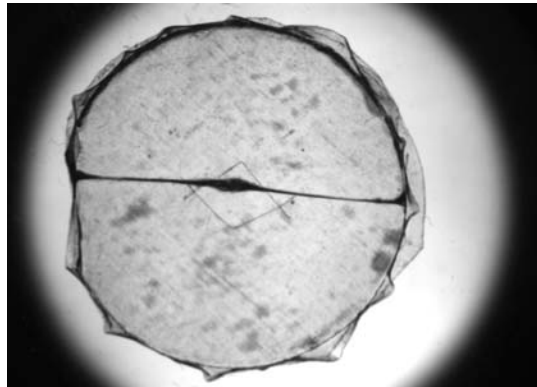
Fragment Size Comparisons for Two Glasses with Equivalent t 's and σ_t 's

Corning 0317



C-6-21-21
 $t=0.085''=2.2$ mm
500C/6 hr
 $\sigma_t=38.7$ MPa

Schott Robax

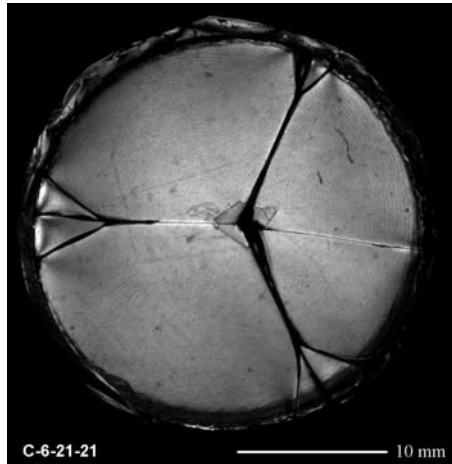


R-11-20-6
 $t=0.085''=2.2$ mm
500C/96 hr
 $\sigma_t=41$ MPa



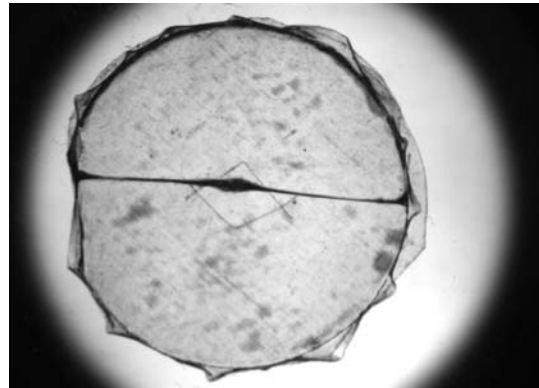
Fragment Size Comparisons for Two Glasses with Equivalent t 's and σ_t 's

Corning 0317



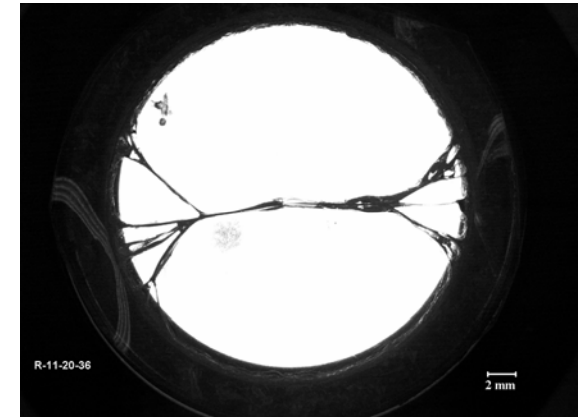
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Schott Robax



R-11-20-6
 $t=0.085''=2.2$ mm
500C/96 hr
 $\sigma_t=41$ MPa

Schott Robax

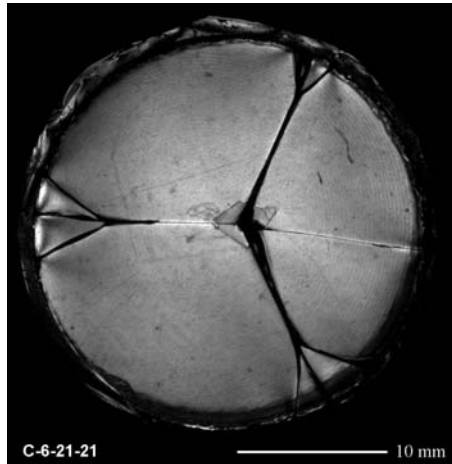


R-11-20-36
 $t=0.085''=2.2$ mm
500C/192 hr
 $\sigma_t=47$ MPa



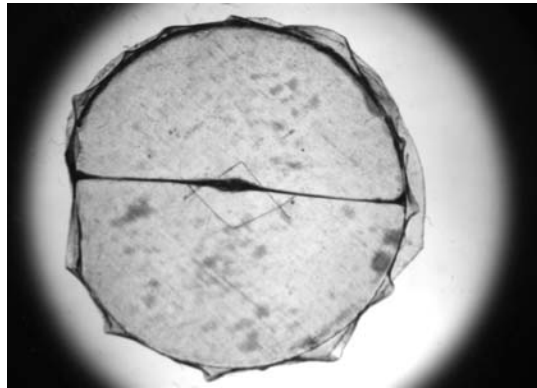
Fragment Size Comparisons for Two Glasses with Equivalent t 's and σ_t 's

Corning 0317



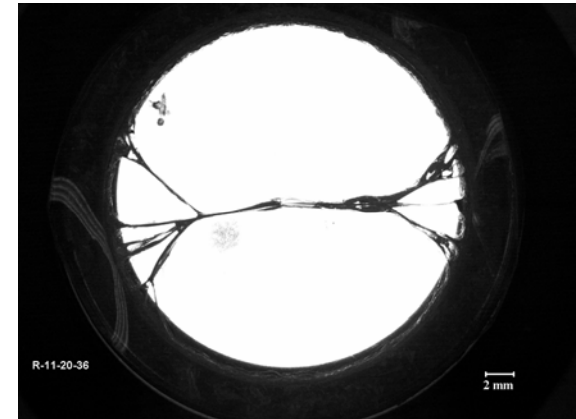
C-6-21-21
 $t=0.085''=2.2$ mm
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 $\sigma_t=38.7$ MPa

Schott Robax



R-11-20-6
 $t=0.085''=2.2$ mm
500C/96 hr
 $\sigma_t=41$ MPa

Schott Robax



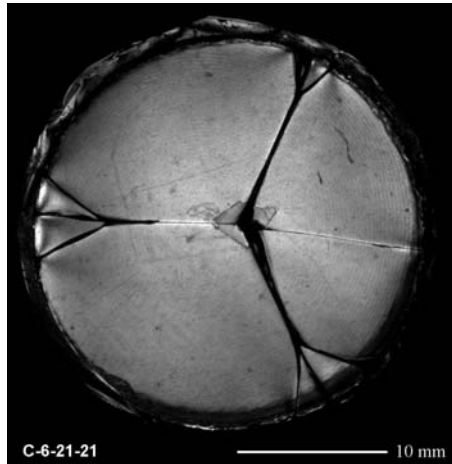
R-11-20-36
 $t=0.085''=2.2$ mm
500C/192 hr
 $\sigma_t=47$ MPa

$$x = \frac{K_{1c}^2}{\sigma_t^2} (1 + \nu) \frac{t}{(0.5t - \delta)}$$



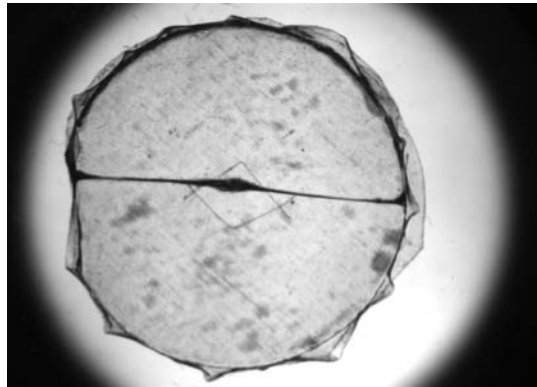
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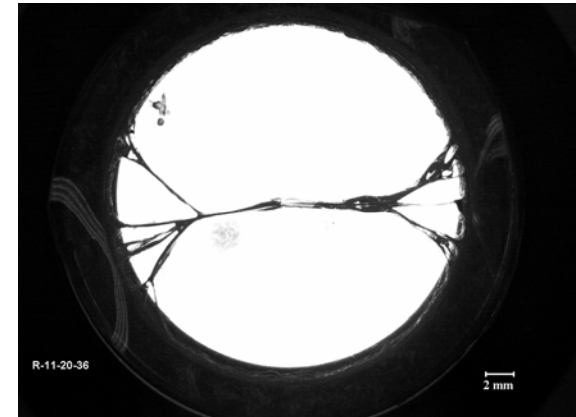
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 $t=0.085''=2.2$ mm
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 $\sigma_t=47$ MPa

$$x = \frac{K_{1c}^2}{\sigma_t^2} (1 + \nu) \frac{t}{(0.5t - \delta)}$$

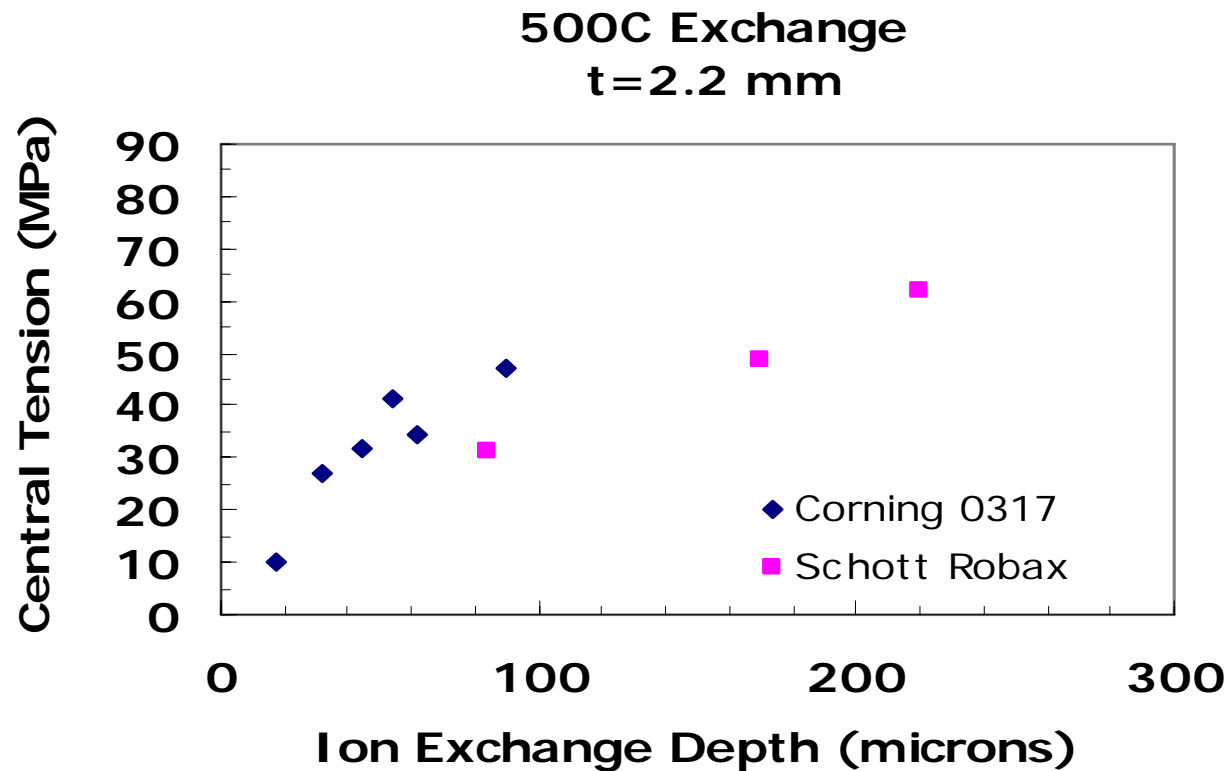
Critical σ_t is ~ 34 MPa for Corning and 47 MPa for Robax (ratio=0.72)

Toughness ratio=0.75 (0.82 vs. 1.09 MPa·m^{0.5})



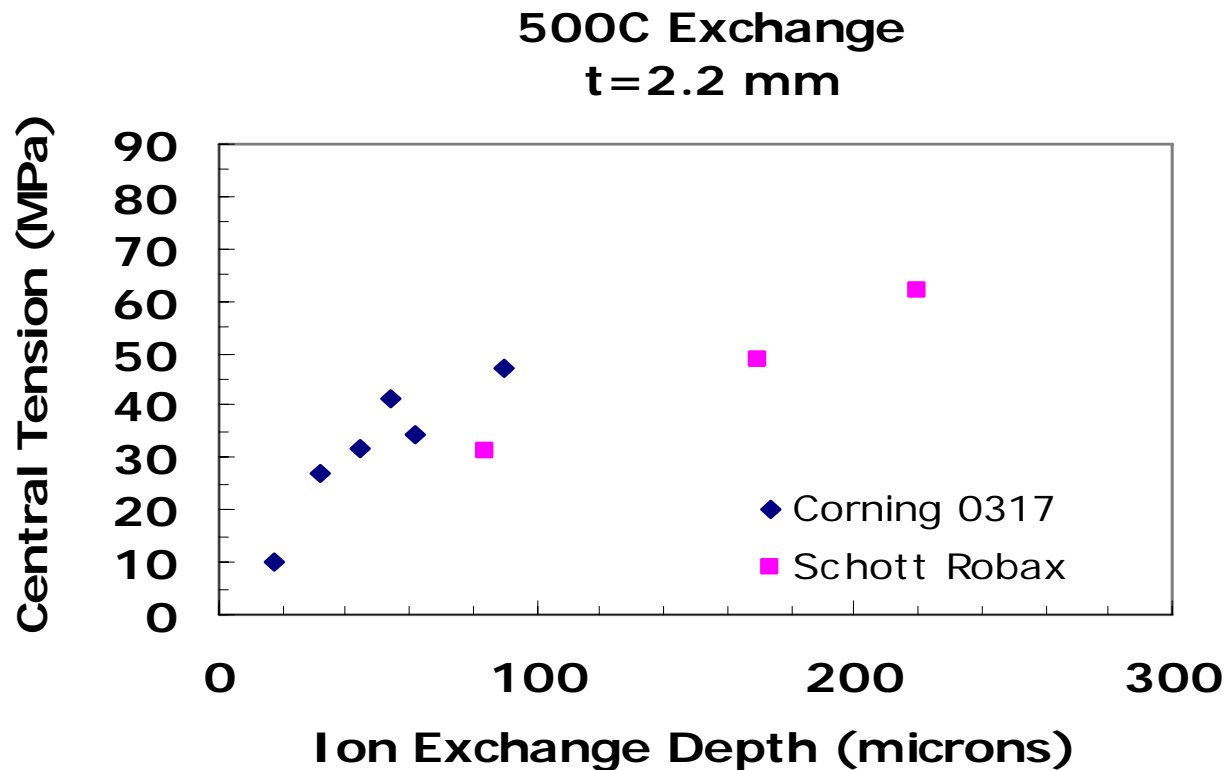


For a Given Ion Exchange Depth There is More Central Tension in the Corning Glass





For a Given Ion Exchange Depth There is More Central Tension in the Corning Glass



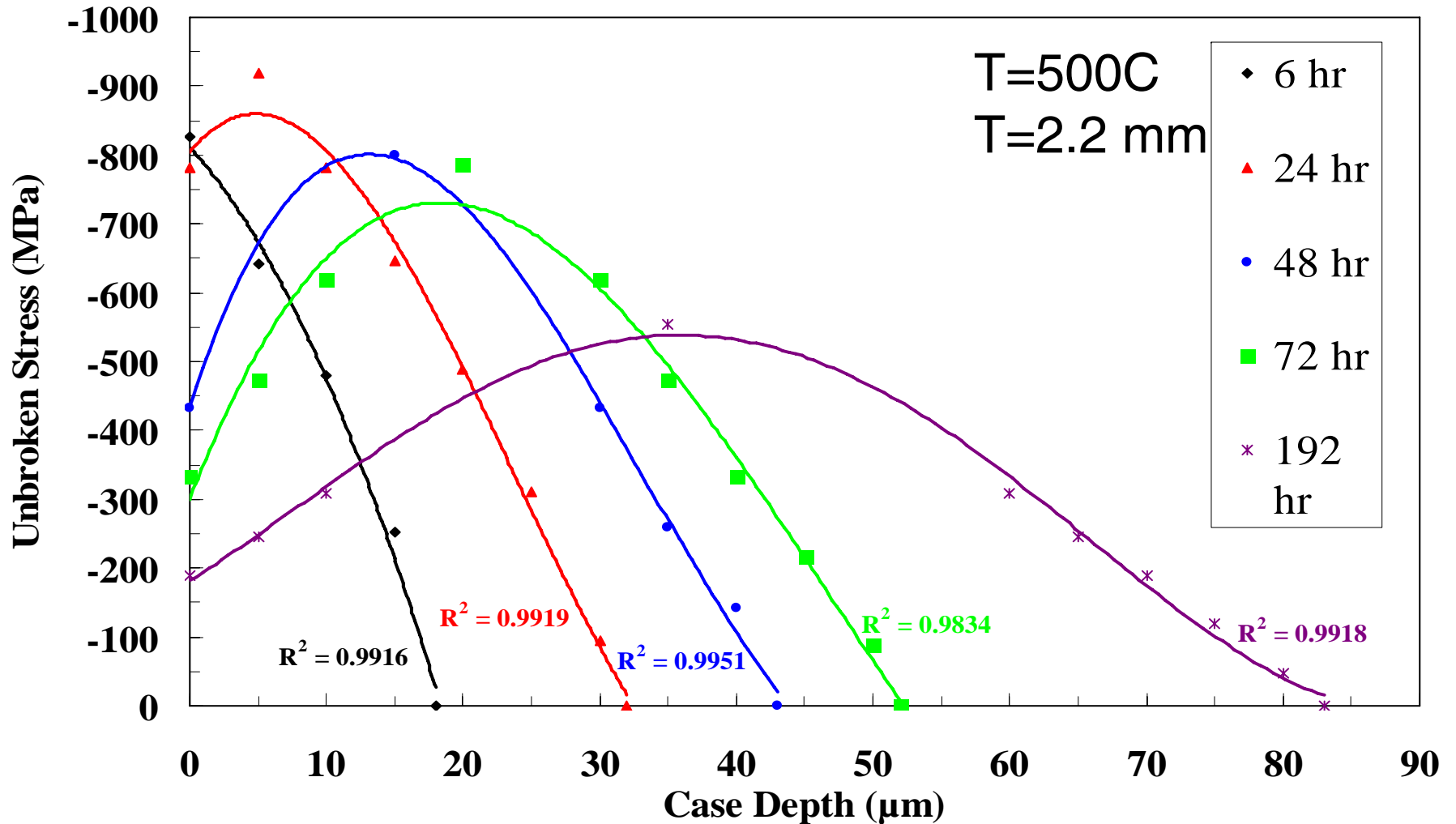
Corning: 12.14 mol% Na_2O (+3.71 mol% Li_2O) being replaced by K_2O

Robax: 6.87 mol% Li_2O + 0.37 mol% Na_2O being replaced by K_2O

Pauling's ionic radii: $\text{Li}^+=1.2\text{\AA}$, $\text{Na}^+=1.90\text{\AA}$, $\text{K}^+=2.66\text{\AA}$



Surface Residual Stress Profile and Case Depth Measurements for Schott Robax

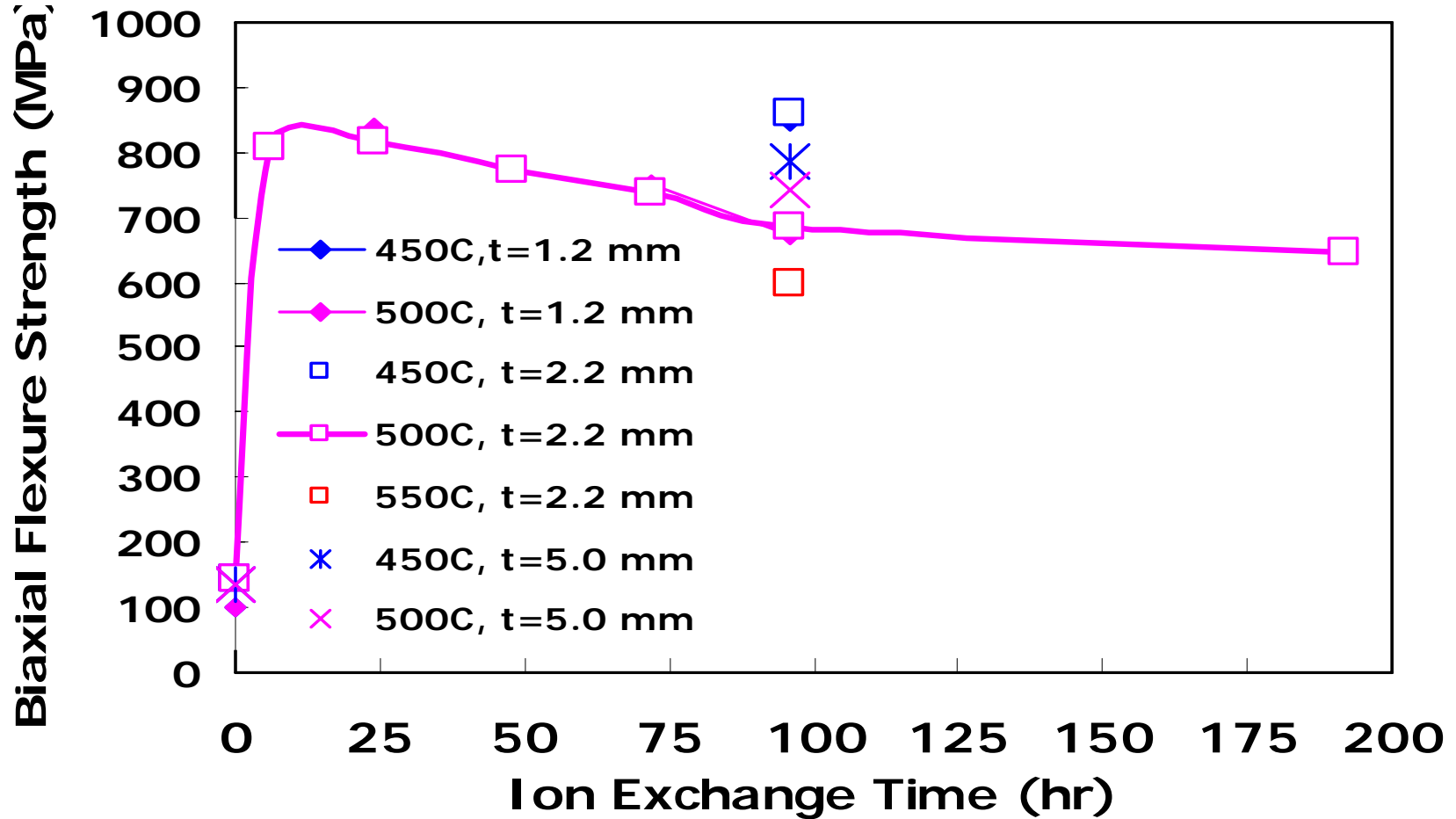


Measurements by I. Spinelli, Saxon Glass Technology, Inc.





Schott Robax Biaxial Flexure Strength vs. Ion Exchange Time





Summary and Conclusions

- Ion exchange provides remarkable damage tolerance and strengthening of glass
- Indentation induced fragment size determined by branching distance (central tension, t , δ) and number of branches
- Corning 0317 has greater ion exchange depth and central tension than Schott Robax for a given exchange time and T
- For similar values of central tension Corning has greater degree of fragmentation than Robax because of lower toughness (0.82 vs. 1.09 MPa·m^{0.5}, ratio=0.75)
- Critical σ_t for fragmentation is ~ 34 MPa for 2.2 mm Corning and just below 47 MPa for Robax (ratio=0.72)
- Glasses with higher K_{IC} require higher central tensions to achieve fragmentation and have larger fragments for a given σ_t
- High values of surface compression and strengths can be achieved for Schott Robax - K⁺ for Li⁺ exchange
- High strengths, even for long exchanges, suggest that fracture controlled by compressive maximum



Acknowledgments

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- D. Gerling, Sierracin/Sylmar Corp., Sylmar, CA

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