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Materials Science and Technology Division

GLASS STRIAE AND LASER SHOCK DAMAGE

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CONTENTS

LIST OF FIGURES

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DISCLAIMER

ABSTRACT

This study sought to examine if striae are preferential locations for damage initiation in vitreous silicate float glasses when they are subjected to shock. Striae are typically native to vitreous silicate float glasses and their formation is a consequence of incomplete or inhomogeneous mixing prior to glass solidification. Striae typically have a lamellar-like habit and their planar orientation is usually perpendicular to the glass tile thickness. Float glass, such as BOROFLOAT® borosilicate glass, is often used in transparent protective systems and therefore can be subjected to shock in a ballistic impact. Previous work by the Authors indicated that shock-induced damage in such glasses *could* be co-located with striae, so the objective of this study was to more closely examine that potential relationship.

Results showed shock-induced damage did not consistently initiate at those native striae with the employed test conditions, but the secondary crack propagation direction may have been altered by their presence. Additional testing using other test conditions and other float glasses is arguably needed to better support those results and interpretations.

1. INTRODUCTION

Striae are typically native to vitreous silicate float glasses and their formation is a consequence of inhomogeneous mixing or prematurely terminated diffusion or both prior to glass solidification. They are also known to form in various types of inorganic melts like lava and glass melts, their presence can affect some physical properties, and their local chemistry (e.g., SiO_2/Al_2O_3 ratio) is subtly different than the matrix material [1-4]. That local difference in chemistry results in there being a local difference in the index of refraction and being optically visible. Striae typically have a lamellar-like habit and their planar orientation is usually perpendicular to the glass tile thickness. Examples of are shown in Fig. 1 for BOROFLOAT® and Starphire vitreous silicate float glasses [5].

Float glass, such as BOROFLOAT® borosilicate glass, is often used in transparent protective systems. If that system is subjected to a ballistic impact, then shock can propagate into and beyond the glass constituent. Previous work by the Authors [6] indicated that shock-induced damage in such glasses *could* be co-located with striae. In that work, a formed damage mechanism (termed "microkernels") whose size was ~ 0.5 to 1 μm often had a planar habit, such as that shown in Fig. 2, and they tended to not be homogeneously distributed. These are similar characteristics of striae.

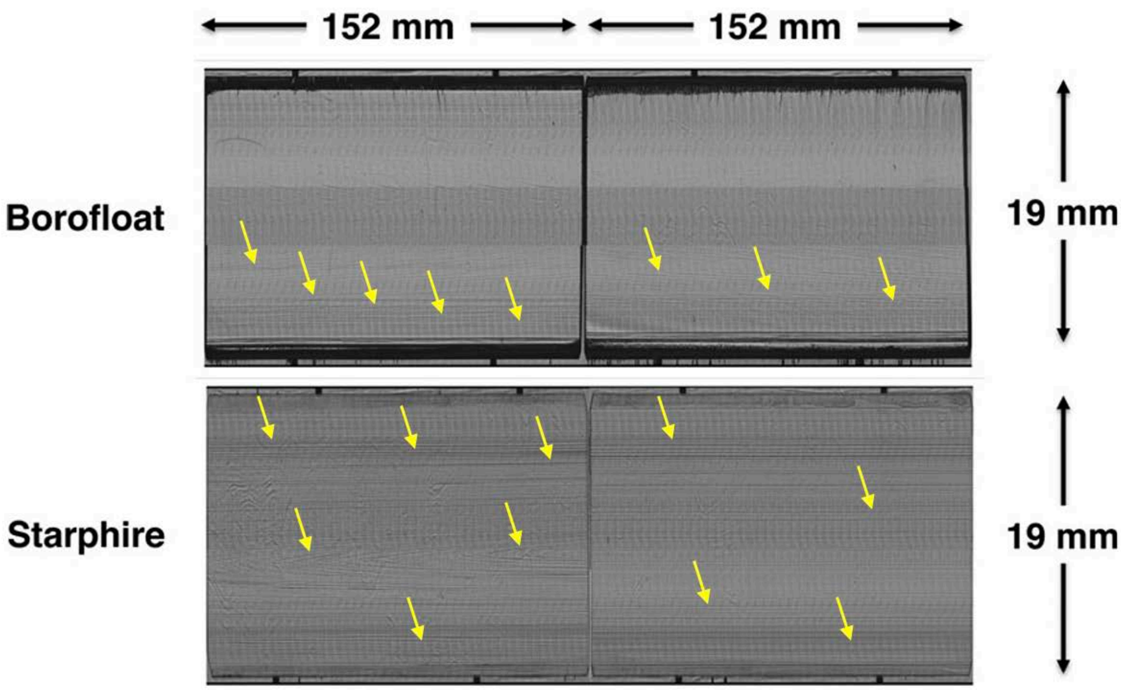


Figure 1. Examples of striagrams [5] with arrows pointing to striae.

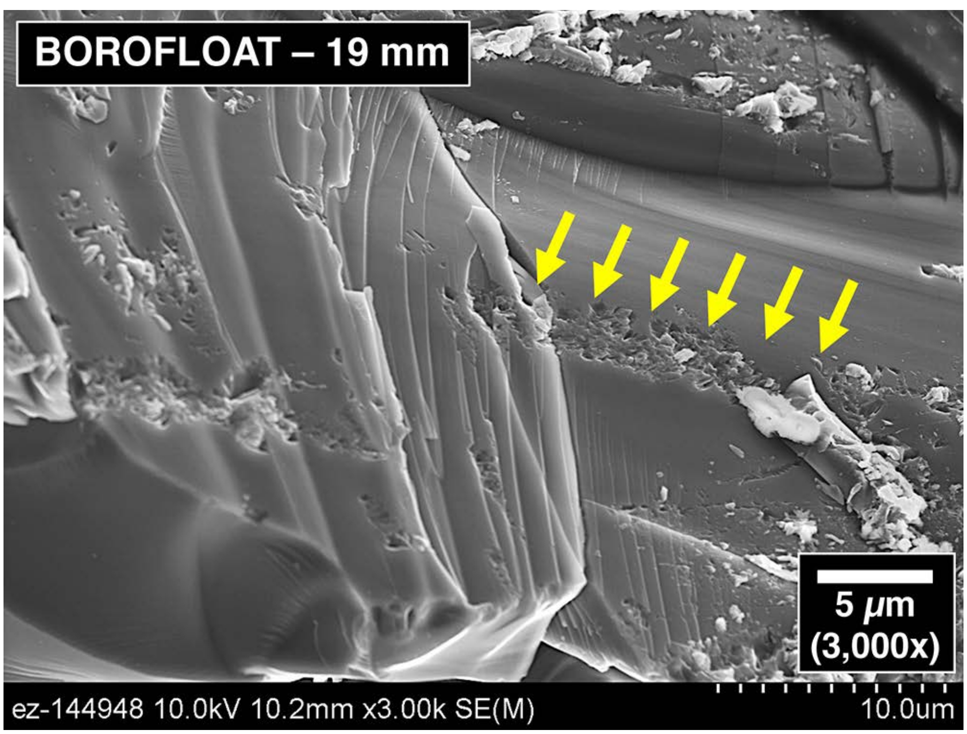


Figure 2. Example of planar habit of shock damage [6].

The shock influence on glasses and ceramics has been examined at ORNL for over 10 years now [7-10] through collaborations with Laser Shock Peening Technologies in Dublin, OH. A high-energy laser, whose energy density and time-dependent waveform are controlled, is shot at a sample like that schematically shown in Fig. 3. The surface location where the laser will impact is first taped with an opaque tape and continually flooded with water. The shot laser passes through the (transparent) water and impacts the tape. A plasma is generated but the water provide impedance to a reflection, and a shock is introduced into the sample.

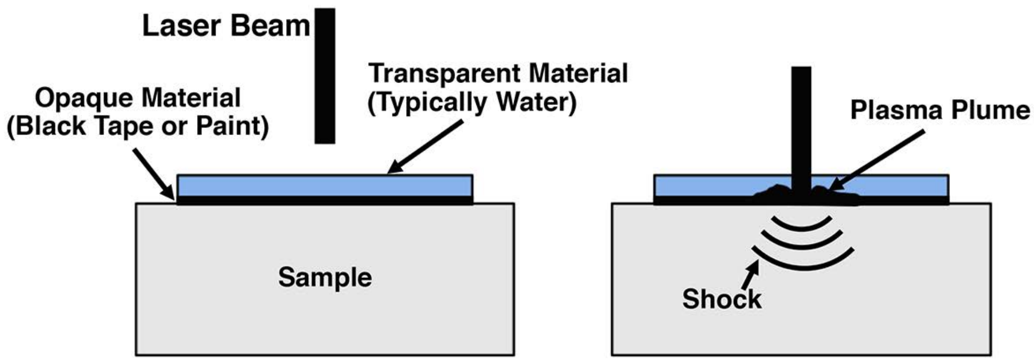


Figure 3. Laser passes through the water, impacts the black tape, creates a plasma the creates a shock pulse in the sample.

The chronology of the resulting generated shock is shown in Fig. 4. The shock wave propagates compressively through the thickness of same (Fig. 4.a.) and encounters the back surface (Fig. 4.b.) and impedance mismatch with the air behind it. The shock wave then gets reflected back as a tensile stress toward the surface where the laser shock impact had occurred (Fig. 4.c-d.).

That chronology also can affect the location and nature of any produced shock-induced damage in three ways. Examples of such damage are shown in Fig. 5 and in reference to Fig. 4.

- Impact surface. The initial impact can initiate surface-located damage [8-9]. In the case of glass, surface-located damage initiation is likely affected by surface finish characteristics and its densification response.

- Internally prior to back-face reflection. The compressive wave (and the tensile wave that immediately follows it) propagating through the sample can generate internal damage such as that shown in Fig. 6 [10]. The authors speculate such an event could be occurring with microkernel formation [6]. *The present study examines this in context to striae*.

- After back-face reflection. The reflected or rarefacted tensile shock can induce spall [7]. Polycrystalline glasses have "spall tensile strengths" similar to what are measured in quasi-static tensile or flexure testing; however, for glass, and it not having a granular microstructure, its tensile spall strength is much larger. Consistent with that, it is very difficult to cause spall in glass.

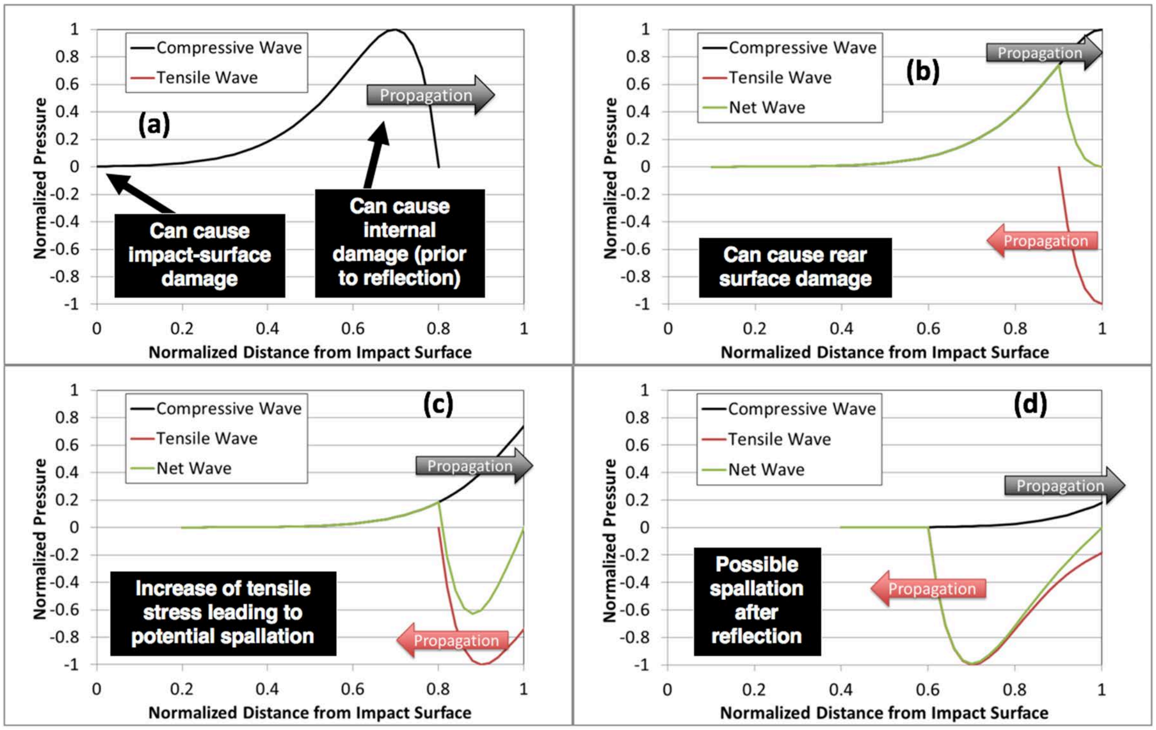


Figure 4. Chronology of (a) shock wave approaching the back sample back surface, (b) as it reflects at the back surface, (c) shortly after reflection, and (d) later on after the reflection.

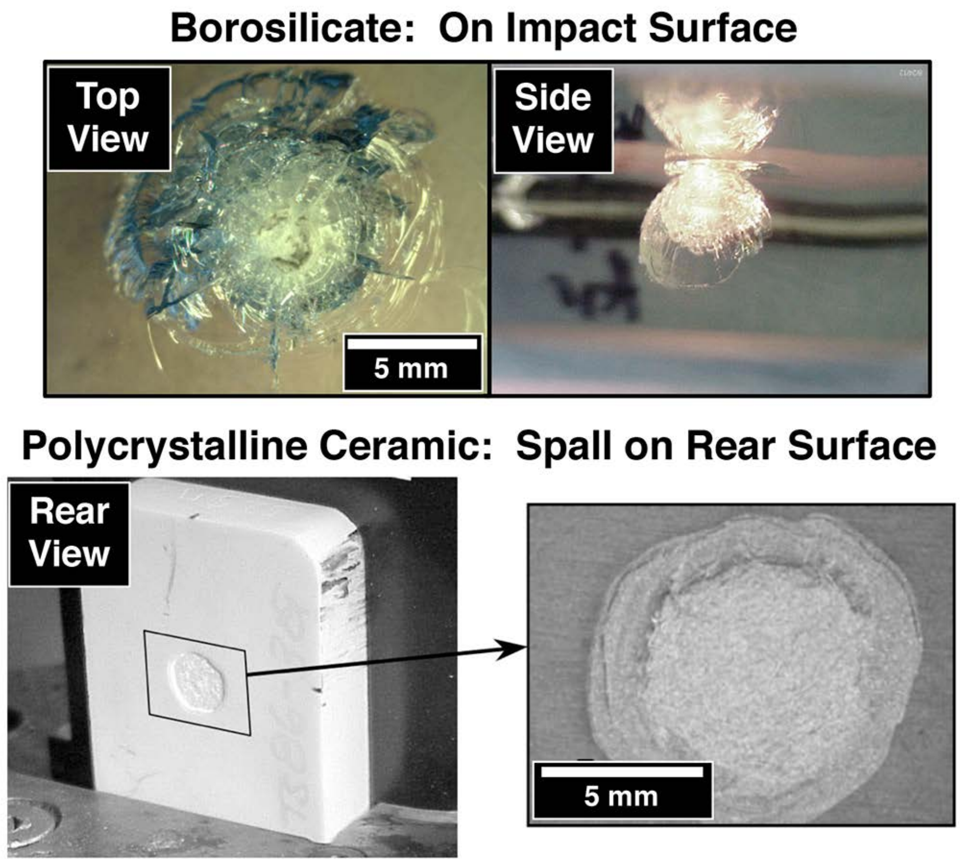


Figure 5. Examples of shock damage. Contact-damage (top left), internal damage (top right), and spall damage (bottom).

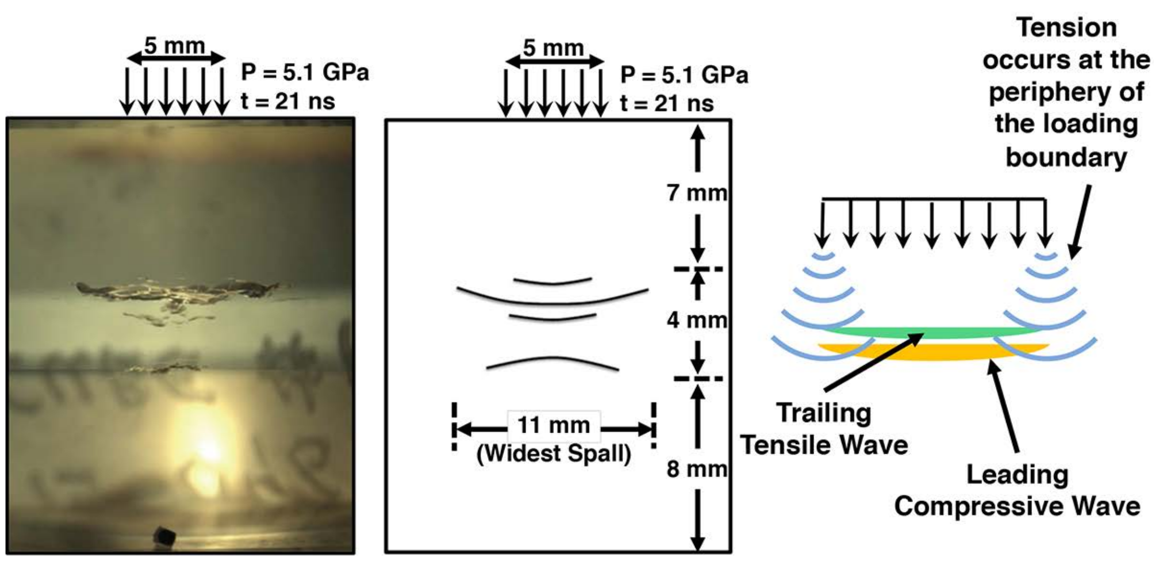


Figure 6. Stresses generated from laser shock that produce internal damage [10].

Given the above, the objective of this study was to more closely examine the potential relationship between shock and whether native striae can be preferential locations for damage initiation due to high-energy shock.

2. EXPERIMENT

Four descriptions are provided associated with the test vitreous silicate glass, the laser shock testing, imaging of the striae, and other post-testing characterization.

2.1 Material Description and Sample Preparation

BOROFLOAT® 33 (or also referred to in this report as just BOROFLOAT®) borosilicate float glass was tested. It is fabricated by SCHOTT North America. A detailed description of BOROFLOAT® can be found in Ref. [5]. Nine-mm- and 19-mm-tile thicknesses were tested, but the post-testing characterization focused on the latter.

2.2 Laser Shock Testing

Laser shock testing, schematically shown in Fig. 3, and whose test setup is shown in Fig. 7 was conducted at Laser Shock Peening Technologies (LSPT) in Dublin, OH. Laser energies were chosen to be sufficiently high to cause internal damage that would enable the study of the possible interaction between shock damage and striae in the glass. The air side of the float glass tiles was impacted. A more detailed description of the laser shock testing can be found elsewhere [7-10] and its principal can be examined at LSPT's website [11].

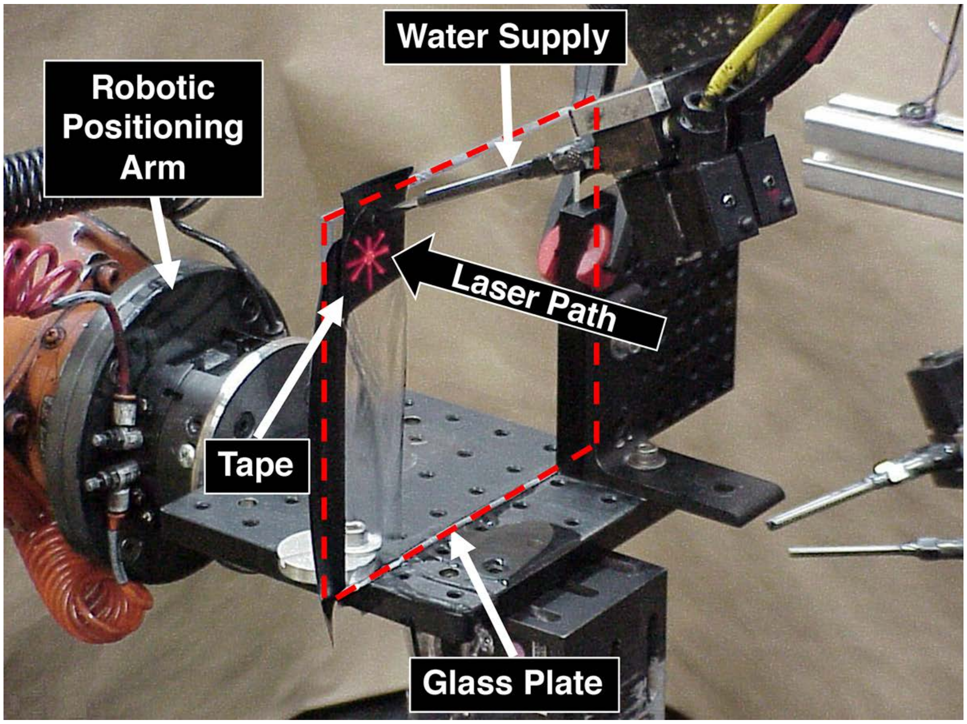


Figure 7. Laser shock test setup.

2.3 Imaging of Striae

Imaging of the striae, whose resulting images are called "striagrams", was performed by NEWTE (Teplice, Czech Republic). NEWTE's Striatter 1000 was used, see Fig. 8, and is an automated instrument for generating striagrams of transparent glasses using a classical technique that exploits a stria's different index of refraction and that has been used glass manufacturers for decades [12]. Testing is done in an index-of-refraction-matched liquid (which manages reflections), and images are produced with a monochromatic and collimated light that is horizontally moving through the glass and captured with a CMOS camera. Details of the instrument can be found at the vendor's website [13].

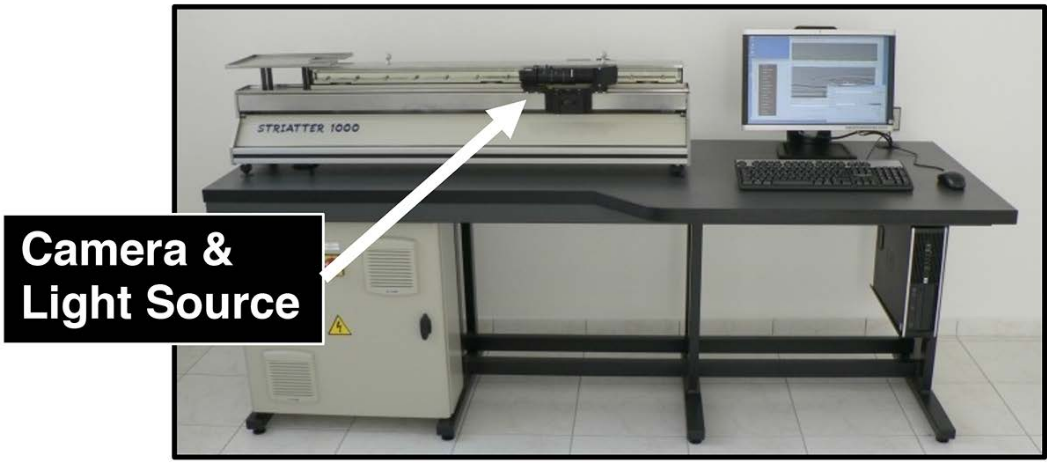


Figure 8. STRIATTER 1000 used to image striae.

2.4 Post-Testing and Supplemental Analysis

Analysis primarily involved optical microscopy of the resulting shocked damage and its proximity to the striae. This was aided with imaging through a polariscope, and photoelastic principals with glass [14-15]. A stress-optical coefficient value, or Brewster's Constant, of 3.8 x 10^{-6}/MPa for borosilicate [15] was used for the calculation of residual stress.

To examine possible differences between the striae and the matrix glass, Energy Dispersive Spectroscopy was pursued to see if a measurable chemical difference existed, a chemical etching (hydrofluoric acid) trial was conducted to examine if differences in chemical reaction susceptibility existed, and nanoidentation using a Berkovich indenter was performed to see if there was a local difference in hardness and elastic modulus.

3. RESULTS AND DISCUSSIONS

The 9-mm and 19-mm thick plates of the investigated BOROFLOAT® borosilicate float glass indeed had striae, and they were more concentrated near the glass's tin side. Examples of striae are shown in Fig. 9.

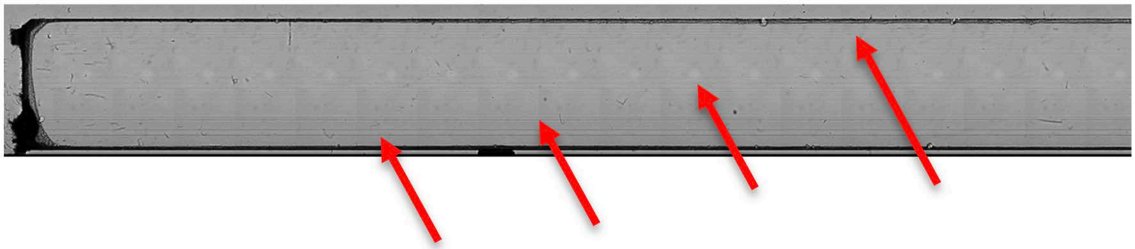


Figure 9. Examples of striae in a cross-sectioned and polished BOROFLOAT® tile prior to its laser shock testing. Thickness of the shown tile is 19 mm, and the tin side is located at the bottom.

The striae chemically etched differently than the matrix glass (see Fig. 10) indicating its chemical composition was different; however, its difference was subtle because it was not detectable using Energy Dispersive Spectroscopy whose chemical resolution is ~ 0.1%. Additionally, the elastic modulus and hardness were statistically equivalent to those of the matrix glass as measured using nanoindentation.

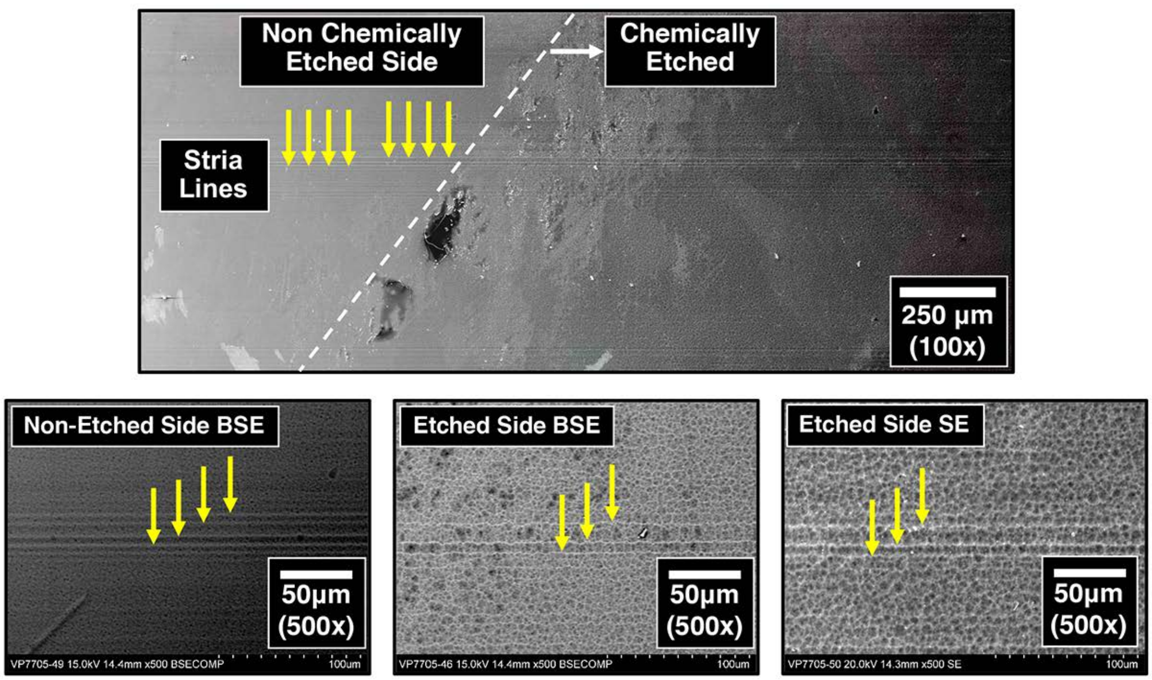
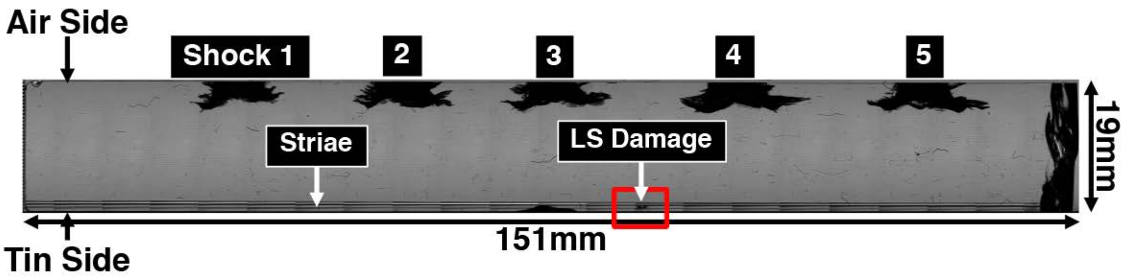


Figure 10. Etching with hydrofluoric acid shows striae can respond differently to etching.

Shock-induced damage did not consistently initiate at those native striae, but secondary crack propagation direction may have been altered by their presence. The analysis that supports that observation are shown in Figs. 11-12. Peak or initial shock pressures between 5-6 GPa caused surface-located damage as well as damage on the face of the tile's back. The back-face-located damage became the focus of the post-testing because that was the tin-side of the tiles and where the striae were more highly concentrated. The striae were co-located in the vicinity of the shock damage as shown in Figs. 11.b and 12.b, but it difficult to confidently conclude if the striae were preferential locations for initiation and directional changes in crack propagation. Additional testing using other test conditions and other float glasses is arguably needed to better support those results and interpretations.

Photoelasticity results suggested that were permanent residual stresses on the order of a few MPa in close proximity of the laser shock, see Fig. 13 Based on other work of the authors [16] and the peak contact pressures the glass was subjected to, that residual stress was likely due to complex, local, multiaxial confinement of densified glass and local crack patterning.



(a)

Laser Shock Location	Energy (J)	Spot Size (mm)	Rise Time (ns)	Pulse Width (ns)	Energy Density (GW/cm²)	Peak Pressure (GPa)
1	19.4	5	4	20.1	4.92	5.66
2	19.5	5	4	20.3	4.89	5.64
3	20.8	5	4	20.5	5.17	5.81
4	20.2	5	4	20.0	5.14	5.80
5	20.4	5	4	20.7	5.02	5.72

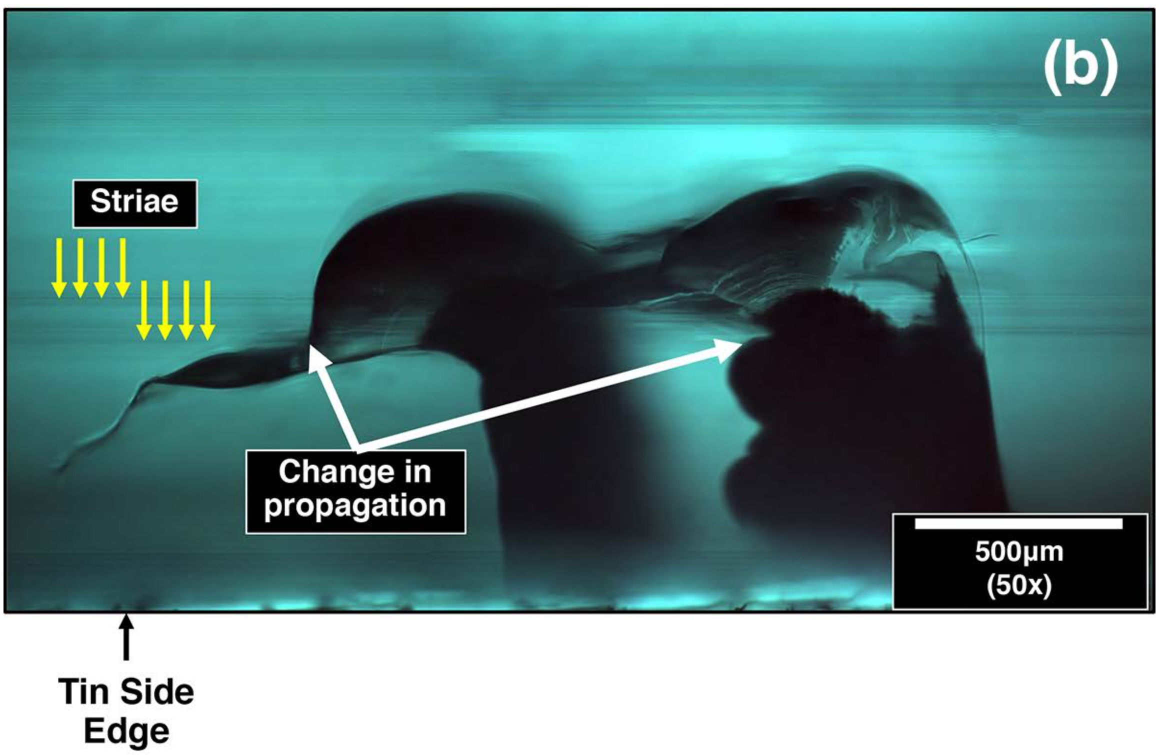
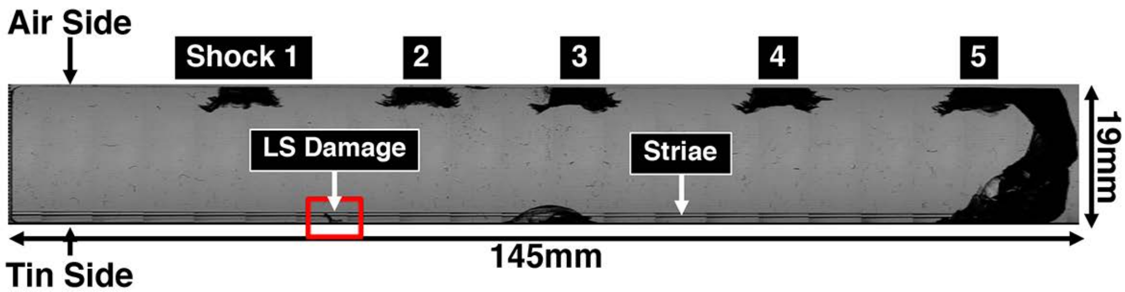


Figure 11. Laser shock damage of sample Tile #1.



Laser Shock Location	Energy (J)	Spot Size (mm)	Rise Time (ns)	Pulse Width (ns)	Energy Density (GW/cm²)	Peak Pressure (GPa)
1	16.2	5	4	20.6	4.01	5.08
2	17.0	5	4	20.6	4.20	5.21
3	18.3	5	4	21.2	4.40	5.33
4	18.2	5	4	20.9	4.44	5.36
5	16.4	5	4	21.0	3.98	5.06

(a)

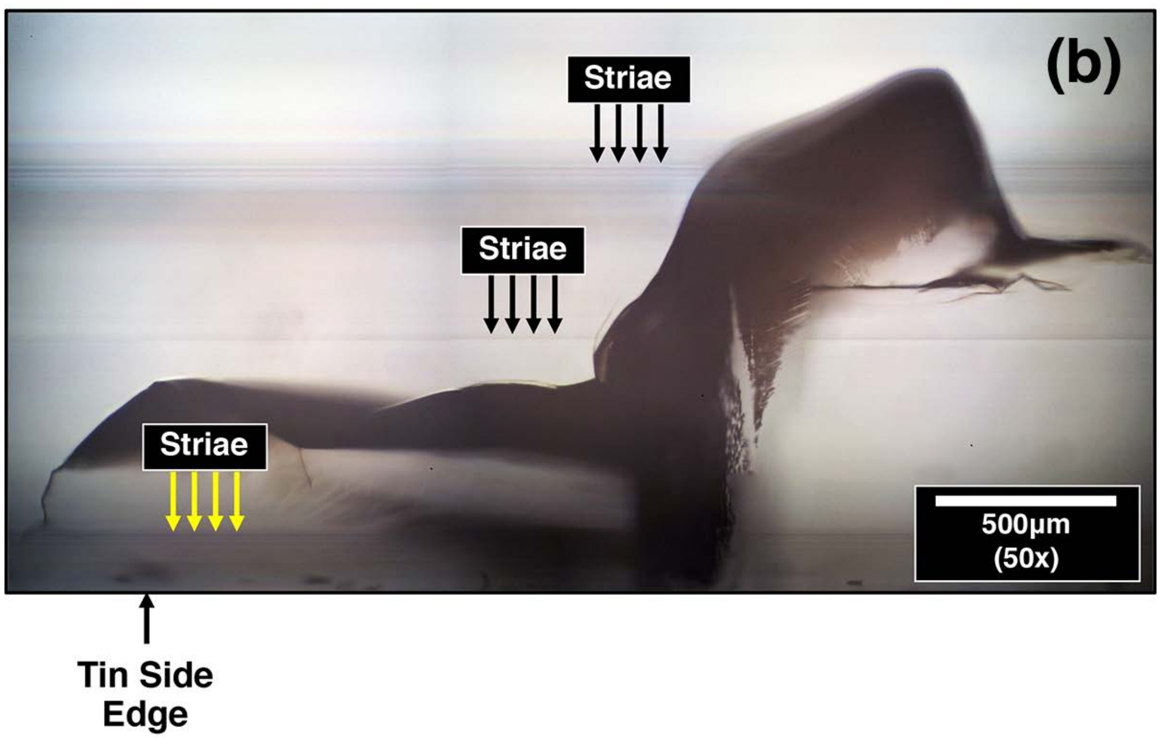
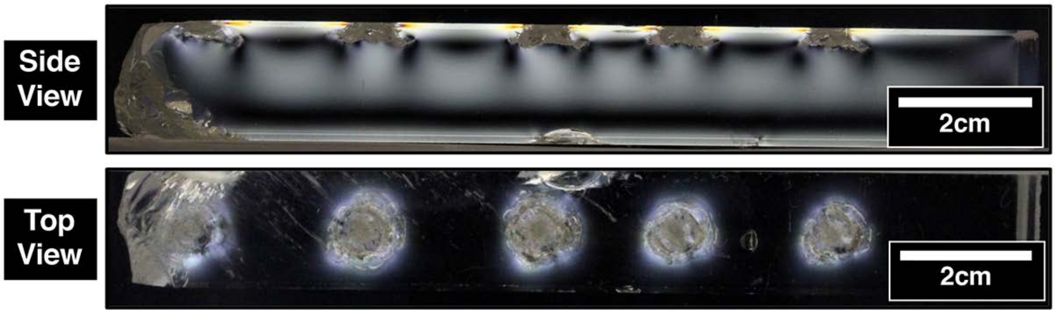


Figure 12. Laser shock damage of sample Tile #2.



$$\sigma = \frac{\delta}{t \times C_B}$$

Where:

$\sigma = Stress$

$\delta = Retardation$

$t = Thickness$

$C_B = Brewster's\ Constant$

Color Observed	Retardation δ (nm)	Stress σ(MPa)
Black	0	0.0
Grey	150	2.0
White-Yellow	250	3.5
Yellow	300	4.2
Orange	450	6.2

Figure 13. Images from a polariscope and residual stress estimation using photoelasticity. A Brewster's Constant of 3.8 x 10^{-6}/MPa [15] was used for BOROFLOAT®.

A closing comment about striae and damage initiation that can occur there. Several additional tests were conducted driven by curiosity where the black tape shown in Fig. 3 was not used. The laser and its high energy were able to penetrate and travel through the entire glass sample. For these tests, it appeared that damage initiation *did* initiate at the native striae. However, that damage was a consequence of direct interaction with the laser (and not shock) and is a complex phenomenon outside the scope of this study.

4. CONCLUSIONS

The investigated BOROFLOAT® borosilicate float glass had striae in 9-mm and 19-mm thick plates. They were more concentrated near the glass's tin side. The striae chemically etched differently than the matrix glass indicating its chemical composition was different; however, its difference was subtle because it was not detectable using Energy Dispersive Spectroscopy whose chemical resolution is ~ 0.1%, and its elastic modulus and hardness were statistically equivalent to the matrix as measured using nanoindentation.

Shock-induced damage did not consistently initiate at those native internal striae when impacted from the air side, but secondary crack propagation direction may have been altered by their presence. Additional testing using other test conditions and other float glasses is arguably needed to better support those results and interpretations.

Permanent residual stress on the order of a few MPa existed in close proximity of the laser shock. Based on other work of the authors and the peak contact pressures the glass was subjected to, that residual stress was likely due to complex, local, multiaxial confinement of densified glass and local crack patterning.

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