

Utilization of Borosilicate Glass for Boron-Neutron-Capture Radiation Treatment of Polymeric Interlayers within Assembled Laminated Glass Window Systems

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Abstract

A method has been investigated that utilizes borosilicate glass as the catalyst for the treatment of polymeric window interlayer material. This method will increase applications for the boron-neutron-capture radiation treatment technique previously developed and referenced herein. Analog laminated glass window panels, consisting of glass layer and EVA interlayer material, were assembled and irradiated, and quasi-static tensile tests were then performed on the irradiated EVA material. Neutron radiation treatment with borosilicate glass caused distinct increases in stiffness and toughness relative to the control specimen. The method proves the mechanical behavior of laminated glass polymeric interlayer material can be affected by radiation treatment even within existing laminated glass window systems, highlighting the effectiveness of neutron radiation treatment to effect target material at depth. Post-lamination treatment of polymers could be used for patterned stiffness profiles that disrupt the shockwave effects caused by blast or ballistic impacts. The stiffening or discoloration mechanism could help in the understanding of window system behavior in high neutron radiation environments such as nuclear reactors or even on the surface of Mars. Treatment methods could be further applied to Microelectromechanical systems (MEMS).

Keywords: Borosilicate Glass; Polymeric Interlayers; Laminated Glass; Neutron radiation; Polymer Material

Introduction

Polymer interlayer materials are utilized in laminated glass systems to provide increased resilience and post-cracking energy absorption during catastrophic events such as blast incidents [1-3]. The polymer chains within the interlayer material can benefit from material modifications that increase cross-linking between adjacent chains. One theorized method of targeted cross-linking is made possible through a boron-neutron-capture process. This process utilizes neutron radiation to bombard borated material, thus producing emissions of highly energetic particles into the polymer. Past efforts at the University of Missouri-Columbia have experimentally utilized the method for bulk material processing as well as surface treatment. The surface treatment process has been more extensively investigated to manipulate polymers commonly used as laminated glass window interlayer material [4,5]. Past evaluations have shown the material behavior changes

through methods of static tensile loading, dynamic tensile loading, indentation testing, and scratch resistance testing. Results have shown increased hardness, elasticity, stress capacity, and toughness.

The surface treatment technique has been utilized on interlayer material prior to lamination but has not had application in fully composite interlayer specimens laminated within glass panes. The obstacle for this technique would be the lack of boron available in typical glass, which is required to surface treat the laminated interlayer. The use of borosilicate glass could present an opportunity to develop laminated glass systems composed of borosilicate glass that can be treated post-lamination. This is made increasingly possible with neutron radiation because it can penetrate deep within material due to the neutron's low probability of interaction with most material.

Understanding a treatment technique via a post-lamination method can lead to the quantifiable application of increased system capacities that would improve the future design of window and building systems for safer, more secure, and resilient infrastructure. Applications with the borosilicate glass composite structure could further enhance resilience under high heat conditions characteristic of fire or nuclear blast. Furthermore, a better understanding of window system behavior in radiation environments can provide design considerations in space environments, the neutron radiation environment on Mars, and other high-radiation environments.

Background

To understand a useful link between laminated glass windows and radiation treatment of polymers, some additional background on these subjects is provided that includes a description of laminated glass panels and their behavior under blast loads, the radiation process utilized in this work, the interlayer material types that were evaluated, and the material characterization methods.

Window systems have advanced to utilize a layered combination of glass and polymer interlayer materials to protect against external hazards. The polymer interlayer material itself can vary in its composition, and its performance can be enhanced by variations in the material strength. Methods to manipulate the polymer material can help lead to increased strength and overall resilience of the window system.

A laminated glass panel consists of two or more layers of glass with a polymeric material between each glass layer, exemplifying the inherent strength and versatility of composite structures. Composite structures play a pivotal role in various industries, offering a remarkable blend of lightweight design, enhanced strength, and resilience, which is crucial for advancing the performance and safety of modern structures and systems [6,7]. The advantages of the polymeric interlayer are to act as a tensile membrane and to hold the fragments of the glass when the glass cracks, preventing damage and reducing the risk of harm due to flying glass shards [1,8,9]. Additionally, the interlayer provides post-cracking energy absorption to the window system, which in a blast event may prevent the pressure wave from entering the building and causing more damage to the occupants [10-12]. Past research has shown that the material characteristics of polymers can be manipulated through irradiation [4,5,13-17]. Specifically, the use of specific material doping, such as the utilization of 10Boron, can create conditions that enable cross-linking of polymer chains when exposed to a neutron field and thus manipulate the behavior of the polymer.

The isotope 10Boron has a very high neutron capture cross-section of roughly 3840 barns for low-energy thermal neutrons, making it a good candidate for capturing neutron radiation.

The resultant products of boron-neutron-capture are a stable Lithium-7 isotope and an Alpha (Helium ion) emission. The Alpha emission carries an energy of 1.78 MeV, and the Lithium-7 isotope recoils, conserving momentum and carrying an energy of 1.01 MeV [14]. These energetic particles are subsequently used in the cross-linking mechanism for the strengthening of polymers. For thin materials such as polymer window glass interlayers (0.381-0.762 millimeters or 0.015-0.030 inches thick), a surface treatment method was developed that could affect a significant depth of the material without the additional manufacturing step of boron doping.

A series of tests were performed on samples of commonly used interlayer materials. These tests exposed samples of the material to a low flux rate of thermal neutrons (8.4×10^8 N/cm²/sec). The neutron source was developed at the University of Missouri Research Reactor (MURR), and has been utilized for a variety of boron-neutron-capture research [18-20]. This research reactor has multiple irradiation positions that allow for various flux rates and neutron energies. The thermal beam port has been specially adapted to filter most of the fast neutron spectrum out of the beam, allowing for highly interactive thermal neutrons to be utilized. Specimen are lowered into a beam position and the beam is set to run for a specified length of time to achieve the desired total neutron fluence. The past irradiation set-up consisted of a layered system to gently compress the polymer interlayer material between a boron nitride plate and an aluminum plate. Once the neutrons bombard the boron nitride plate, the high cross-section 10Boron will interact with the neutrons and emit energetic particles into the surface layer of the polymer interlayer.

As discussed in previous work, 0.381mm Ethyl Vinyl Acetate (EVA) polymer samples that were irradiated at 8.4×10^8 N/cm²/sec neutron flux for varying time durations were tested with a quasi-static tensile testing process for comparison. This evaluation will provide insight into the effects of progressively larger doses of neutron radiation. Samples included irradiation durations of 0-, 10-, 40-, and 100-hours for comparison. Three individual test coupons were made for tensile evaluation regarding each irradiation, and the average of the three tensile tests was provided. Results from the study can be seen in Figure 1 [4,5].

As seen in Figure 1, all radiated specimens exhibited higher stiffness compared to the control specimen. There was significant stiffness behavior change at even the lowest 10-hour irradiation, specimen EVA-010. The 40-hour irradiation, specimen EVA-012, showed slightly higher stiffness than the 10-hour irradiation. As the duration reached the 100-hour length, it appears there may even be some degradation.

Experimental Description

Pyrex[®] is the brand name for a borosilicate glass with low thermal expansion. Commonly used in cookware, this product has

many applications beyond cookware, including high-temperature applications and even use in telescopes. Pyrex® has a composition that is approximately 4% naturally occurring boron, making

the material a good candidate for use in this particular surface treatment method of polymer interlayer materials [21].

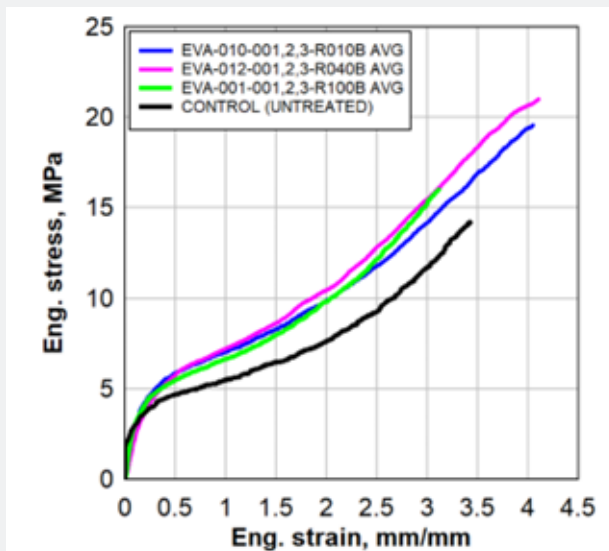


Figure 1: Dose effects of EVA with surface treatment.

Past efforts have focused on pre-treatment of the polymer interlayer material prior to lamination within glass panels. With the use of borosilicate glass, laminated glass window systems could be assembled with one glass surface made up of borosilicate sheet glass, and the window system could be treated post-assembly. The use of only one panel of borosilicate glass is recommended as the neutrons flux may be diminished while passing through the any material that contains boron. If the neutron beam is positioned such that the neutrons pass through normal float glass, through the polymer, and then strike the surface of borosilicate glass; then it is expected that neutron flux should be sufficient to instigate material behavior change in the polymer. The resulting interaction at the interface of the polymer and the borosilicate glass is expected to produce similar results to past research.

Approximate calculations show that total neutron flux can be adjusted to obtain similar treatment results as those found conducted in previous work. The boron nitride plate had been composed of approximately 43.4% boron by weight and the borosilicate glass is composed of approximately 4.0% boron by weight. The density of the boron nitride plate is 2.214 grams/cm³ and the density of the borosilicate glass is 2.214 grams/cm³. Adjusting for density between the two materials, the borosilicate would have a slightly 5.3% higher percentage of material density than the boron nitride block, and that would be similar to a 4.21% density adjusted boron percentage. This brings the comparison between the two materials to a factor of 10.31 in adjusted boron quantities. Therefore, for this experiment, neutron flux will be adjusted with an increase of ten times the total neutron flux for

the borosilicate glass material. This could meet expectations to develop similar results between irradiations of the boron nitride block at 10 hours and irradiations of the borosilicate glass at 100 hours.

A series of testing was proposed to determine boron-neutron-capture treatment effects on laminated glass that utilizes EVA interlayer material and borosilicate glass. Material will not be fully laminated, but rather only simulated layers without full lamination protocol followed (no heating or high-pressure treatment). Steps required are as follows:

- Procure EVA interlayer material.
- Procure float glass.
- Procure borosilicate glass.
- Cut material as required for irradiation.
- Assemble layered specimen in the order of:
 - Group 1 (Specimen 20, 21, 22) Float glass, EVA, boron-nitride plate. Apply moderate clamping force.
 - Group 2 (Specimen 23, 24, 25), Float glass, EVA, borosilicate glass. Apply moderate clamping force.
 - Group 3 (Specimen 26, 27, 28), Virgin EVA. Control group.
- Irradiate Group 1 for 10 hours.
- Irradiate Group 2 for 100 hours.

- Punch out 3 standard test coupons specimen for each sample (see full test matrix in Table 1).
- Perform quasi-static tensile test on all test coupons.
- Post-process video of each test using the Digital Image Correlation (DIC) method to derive the strain-time histories.
- Plot engineering stress-strain responses and evaluate results.

Table 1: Static Tensile Test Matrix of EVA Specimens.

Material	Specimen number	Subset	Treatment
EVA-	020-	001	No Radiation ,virgin material(N000N)
EVA-	021-	002	No Radiation ,virgin material(N000N)
EVA-	022-	003	No Radiation ,virgin material(N000N)
EVA-	023-	001	Radiation at Low Flux ,10 hour, Boron plate (R010B)
EVA-	024-	002	Radiation at Low Flux ,10 hour, Boron plate (R010B)
EVA-	025-	003	Radiation at Low Flux ,10 hour, Boron plate (R010B)
EVA-	026-	001	Radiation at Low Flux ,100 hour, Pyrex Glass (R100P)
EVA-	027-	002	Radiation at Low Flux,100 hour, Pyrex Glass (R100P)
EVA-	028-	003	Radiation at Low Flux,100 hour, Pyrex Glass (R100P)

Procedure

Tests were performed as in past work [4,5]. Two major differences from past efforts were included, both of which should lead to less pronounced results, but still expected to show similar behavior changes. The first difference in process was the use of float glass, as opposed to the prior use of aluminum on the incoming neutron side of the specimen. Aluminum has much greater transparency to neutrons in comparison to float glass, so it is expected to reduce the received flux by a marginal amount. However, the use of the float glass should more closely represent the application within a fully laminated glass panel. The second difference is the use of a thicker EVA material. As the maximum penetration of the treatment technique is expected to be within 10 microns of the surface, a thicker specimen may not see as significant of percent change of polymer material behavior as a thinner specimen. The polymer material used is twice as thick as the past research focus (0.762mm thick rather than the previously evaluated 0.381mm thick EVA).

Treatment

In this study, all test specimens were cut from the same sheet of EVA EVGuard material having 0.762mm-thickness. All specimens were kept under the same temperature and storage conditions. The Boron Nitride Plate and Borosilicate Glass panels were procured from an online supplier, and the float glass panels were procured from a local architectural glass supplier. The following shows the treatment methods for each testing group in this study:

Group 1:

- EVA material was placed between a panel of Float Glass

on one side and a Boron Nitride Plate on the other side.

- Simple clips were used to add light compression to the EVA material.
- The composite specimen was placed in the neutron beam port with the Float Glass panel on the incoming beam side and the Boron Nitride Plate on the opposing side.
- The specimen was irradiated with a thermal neutron beam at 8.4×10^8 n/cm²/sec for 10 hours.
- The specimen was removed and allowed to rest for several months.
- Test coupons were made from the irradiated specimen and evaluated using a quasi-static tensile test.

Group 2:

- EVA material was placed between panels of Float Glass on one side and Borosilicate Glass on the other.
- Simple clips were used to add light compression to the EVA material.
- The composite specimen was placed in the neutron beam port with the Float Glass panel on the incoming beam side and the Borosilicate Glass panel on the opposing side.
- The specimen was irradiated with a thermal neutron beam at 8.4×10^8 n/cm²/sec for 100 hours.
- The specimen was removed and allowed to rest for several months to allow for any activated material to decay.
- Test coupons were made from the irradiated specimen and evaluated using a quasi-static tensile test.

Group 3:

- a. Control group – no radiation was performed.
- b. Test coupons were evaluated using a quasi-static tensile test.

Tensile Testing

In this study, the sample geometry was, according to ASTM D638-10 [13], a standard Type IV specimen, as shown in Figure 2a. To ensure the accuracy of the specimen dimensions, a steel cutting die was manufactured (Figure 2b) [1–3]. All tensile test specimens were cut from aged virgin EVA sheets of 0.762-mm thickness. The 25-mm (1-inch) central gauge length, L_g , and the grip locations

were marked with thin black lines using a permanent marker pen prior to testing (Figure 3a). High contrast clips were attached to the test specimen to enable accurate measurement of very large strain throughout the test using the DIC method (Figure 3b). Digital calipers were used to measure the thickness and width of the test section at three locations to an accuracy of ± 0.0125 mm (± 0.0005 -inches).

Specimens were loaded into the static tensile testing device as shown below in Figure 4a. A camera was used to film each test so that the strain-time histories could be derived in post-processing using the DIC method (Figure 4b). Specimens were pulled in tension at a displacement rate of 2 in/min, which corresponds to an average strain rate of 0.013 s^{-1} , until failure.

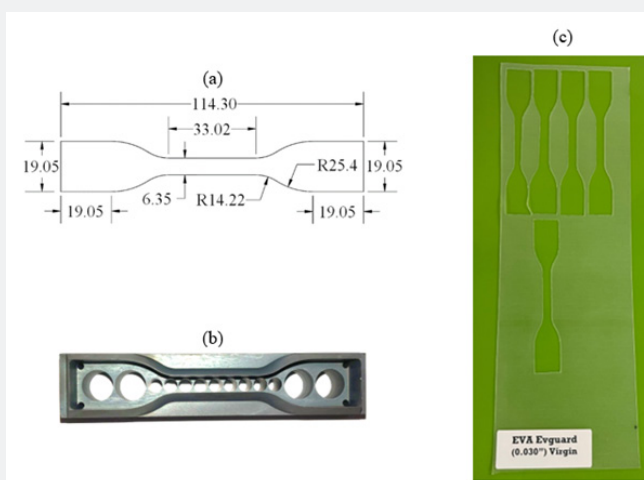


Figure 2: Specimens stamping: (a) Static specimen geometry; (b) Static cutting die; (c) EVA EVGuard Material, control sample with test coupons punched for tensile testing. Dimensions are in mm.

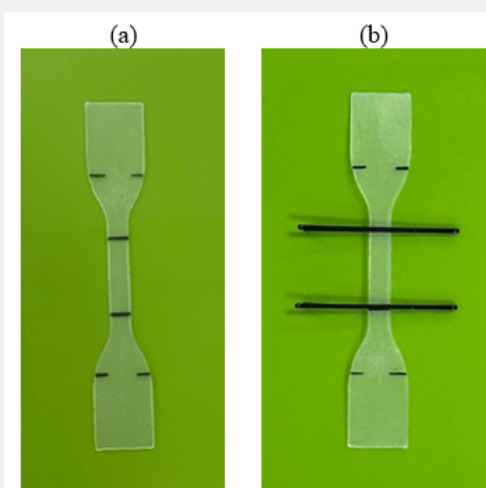


Figure 3: Specimen preparation: a) Tensile test specimen with gage length and gripped distance marked, and b) additional markers for ideal DIC analysis.

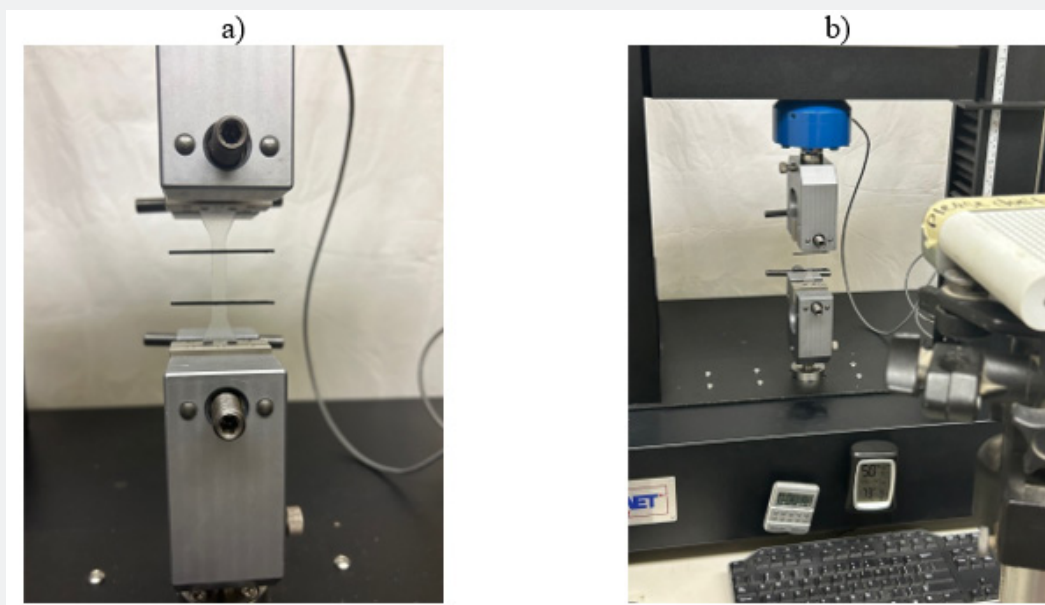


Figure 4: Tensile test setup: a) specimen placed in grips for tensile testing, and b) overall setup with camera for DIC.

Results

Test specimens were strained to rupture failure for each case, and the engineering stress-strain responses were plotted. Failed

specimen underwent significant strain, and in all cases were more than 7 mm/mm of strain. Strain beyond 7mm/mm has been removed from final combined result graphs. Pictures of failed specimen can be seen in Figure 5.



Figure 5: Failed tensile test specimens.

The engineering stress-strain responses are for the control EVA Group 1, the 10-hour Boron Nitride radiation Group 2, and the 100-hour radiation Pyrex and float glass Group 3 are shown below in, Figure 6 a), b), and c), respectively. For each group, an average curve from the three test specimens is shown in black,

and the individual specimen data are depicted by the blue, red, and magenta markers. For each group, the results from each of the three test specimens show a very good correlation, with little variance in stress and failure strain.

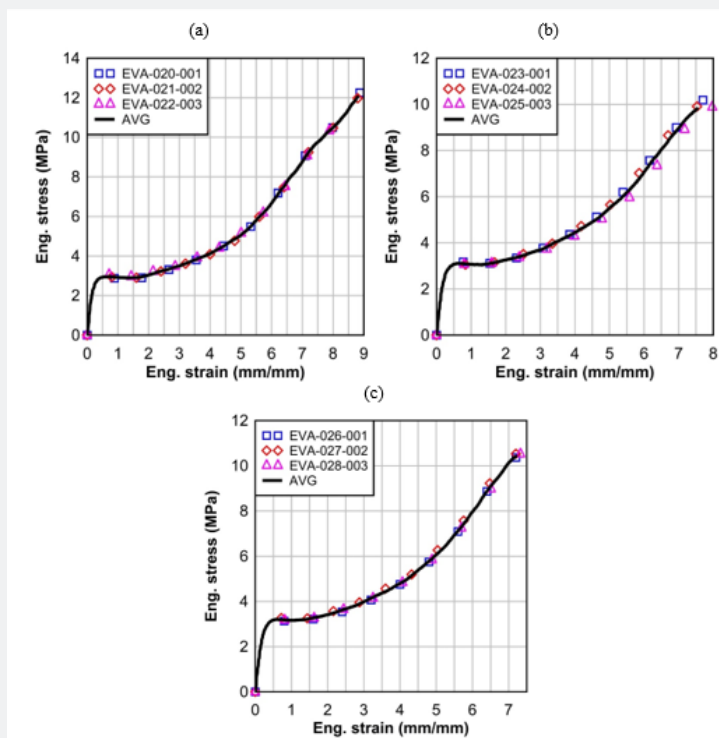


Figure 6: Engineering stress vs. strain for a) EVA control group, b) EVA samples irradiated with boron nitride plate, 10-hour irradiation, and c) EVA samples irradiated with Borosilicate Glass backing, 100-hour irradiation.

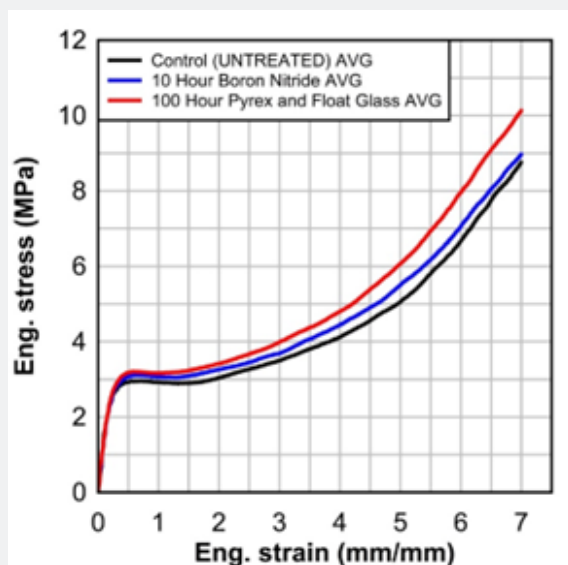


Figure 7: Average engineering stress-strain response of each sample set; Control, 10-hour radiation boron nitride, 100-hour radiation Pyrex and float glass.

For comparison, the average curves of all three groups are plotted together in Figure 7. To compare the effect of neutron radiation treatment relative to the control at the same strain values, all curves were cut off at a strain of 7mm/mm.

The engineering stress-strain response of EVA can be generally characterized by four distinct regions: (1) linear elastic region until strain of about 0.25, (2) strain softening until local maximum stress, (3) stress relaxation region as specimen undergoes plastic deformation, and (4) strain hardening until failure. The effect of radiation treatment on the initial stiffness in the linear region is relatively small, +1.9% and +4.1% for Group 1 and Group 2 compared to the control. However, when considering the stiffness of the material through the strain-softening region, the effect of radiation treatment is significant. The effect is quantified below in Table 2. Due to the radiation treatment, both Groups 1 and 2 have higher local maximum stresses and the local maximum stresses are at lower strain values relative to the control group. The

secant modulus, defined as the slope of the line through the local maximum, is 12.8% and 27.6% greater than the control group for Group 1 and Group 2, respectively.

The increase in stiffness in the material response due to radiation results in increased toughness relative to the control group, as shown in Figure 8. For Group 1, the increase in toughness is relatively constant after 2.0 strain, showing that the radiation treatment had a negligible effect on the mechanical response in the strain-hardening region. For Group 2, the most dramatic increase in toughness is in the linear region, after which the toughness difference continues to increase linearly. Therefore, the radiation treatment of Group 2 shows a significant effect on the total mechanical response, even throughout the hardening region.

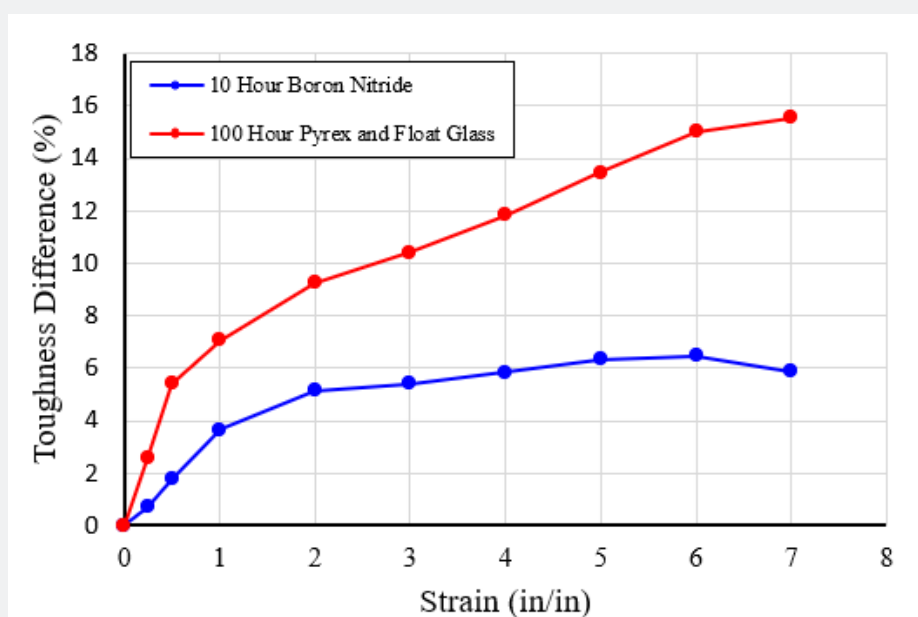


Figure 8: Toughness increase (%) due to radiation treatment relative to the control group.

Table 2: Local maxima data.

	Local Maximum Stress (Mpa)	Strain at Local Maximum (mm/mm)	Secant Modulus (Mpa)	Secant Modulus Difference (%)
Control	2.96	0.713	4.15	-
10-Hour Boron Nitride	3.12	0.666	4.68	+12.8
100-Hour Pyrex and Float Glass	3.21	0.606	5.3	+27.6

Another notable outcome was the effect of the irradiation on the Borosilicate Glass Panel, as seen in Figure 9. The neutron beam can clearly be seen as the 150mm diameter discoloration on the borosilicate glass panel. There was no discoloration seen in the float glass. The float glass did have a small bit of radioactivity that appeared to be coming from a single point. This radioactivity did not dissipate after several months, and the specimen was not removed from the facility.

Conclusion

Overall, the testing was successful in showing the potential treatment of polymeric interlayers within assembled laminated glass window systems. Borosilicate glass was used to initiate in-situ neutron radiation treatment of polymer interlayers with verifiable material behavior changes. These behavior changes are most apparent from the increased stiffness of the EVA material following neutron radiation treatment.

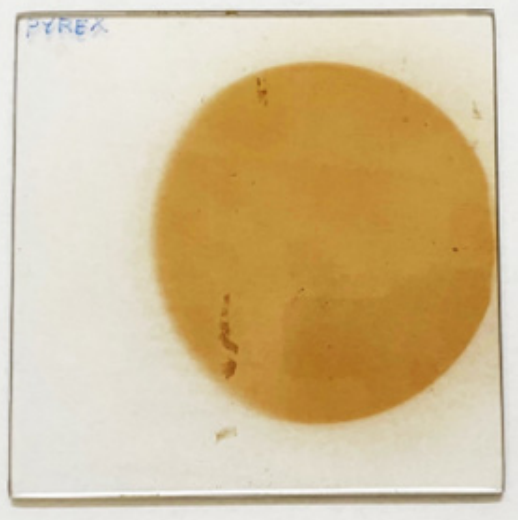


Figure 9: Borosilicate Glass following irradiation in the 150mm diameter neutron beam. Note discoloration. The small amount of tape residue from securing specimen not relevant.

Hypothesized correlations between the total neutron fluence effects of the Boron Nitride Panel and the Borosilicate Glass Plate were reviewed and showed results that trended in the same direction but did not prove to be an overlapping match in the quantified results. The Borosilicate Glass Panel showed a more highly pronounced increase in stiffness; better than expected between the two catalyst materials tested. Although not all mechanisms are fully understood, it is reasonable to expect that the surface contact differences between the two materials may be the cause of this difference in the results. The Boron Nitride Panel has a relatively rougher and more porous surface as compared to the smooth surface of the Borosilicate Glass Panel. This smooth surface appears to create a much better interaction between the energetic particle emissions and the EVA material, as the total travel distance is limited to approximately 10 microns. The EVA was very well adhered to the Borosilicate Glass Panel.

The discoloration of the Borosilicate Glass Panel has shown that methods to minimize clarity issues during treatment should be addressed. The discoloration is most likely due to the boron-neutron capture within the material, but the exact cause of the discoloration will require further investigation. A thinner layer of borosilicate glass could reduce the effect. Decreased amounts of radiation may be utilized in future testing to determine a tolerable limit that maintains visual clarity.

The float glass showed no noticeable change in clarity but did activate in one spot on its surface. This is likely from an impurity in the glass that had a highly activating material composition, so care must be taken to avoid contaminants during the glass manufacturing process. Repeated tests will indicate if this becomes a continuous problem.

These overall results have shown Borosilicate Glass can certainly have a pronounced effect on the material stiffness behavior of adhered polymeric material. This experiment has also shown the profound advantages of treatment with penetrating neutron radiation. Due to the low interaction of neutrons with most materials, the radiation can pass through thick layers of non-target material and then affect its target at depth. Post-lamination treatment of polymers could be used for patterned stiffness profiles that disrupt the shockwave effects caused by blast or ballistic impacts.

Treatment methods could be further applied to Microelectromechanical systems (MEMS) that utilize borosilicate glass substrates. Some MEMS rely on a pre-characterized stiffness coefficient of the system to process the voltage outputs for usage in accelerometers, gyroscopes, and more. The stiffness coefficient could be adjusted through neutron treatment. Furthermore, borosilicate glass-polymer composite systems utilized in high neutron flux environments could be susceptible to variation in voltage output beyond the expected functionality. This could be especially relevant in space-based or other nuclear related systems.

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