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Mechanical Properties of Photomultiplier Tube Glasses for Neutrino Detection

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Photomultiplier tubes (PMT) are one of the primary components of water Cherenkov neutrino detection for the Long Baseline Neutrino Experiment (LBNE). Thousands of 10- to 12-inch diameter PMT bulbs are placed in the inner wall of a detection tank or a reservoir (e.g., deep mine) filled with 10,000 gallons of high purity water with a resistivity of 11–18.24 MΩ-cm. Long-term service of PMTs is vital to the success of neutrino detection projects. We report our results of our investigation on mechanical properties of PMT glasses from two vendors and the effect of ion exchange on their mechanical strength. Vickers indentation, four-point bend test, and ring-on-ring biaxial flexural strength test were used for evaluation of the mechanical strength. Chemical (potassium–sodium ion exchange) strengthening results show increased strength of 46% in one vendor glass and a 57% increase in the other, with no significant reduction in optical transmission in the ultraviolet-visible range of the electromagnetic spectrum that is critical to neutrino detection. Our results also show narrowing of the distribution of strength calculated using Weibull statistics with chemical strengthening for comparable exchange depths of 22–28 μm.

Introduction

Photomultiplier tubes (PMT) are extremely light sensitive glass-enclosed vacuum tubes that are used in large number in water Cherenkov neutrino detectors. Water Cherenkov detector uses the phenomena that light travels 25% slower in water than in vacuum thus enabling energetic particles to travel faster than light. This produces an optical boom analogous to sonic boom that takes the form of faint blue light called Cherenkov light (Pavel Alekseyevich Cherenkov, Nobel Prize, 1958). The measurement of the shape and size and the direction of the faint blue light help to determine the energy, type (muon, tau, or electron), and direction of the neutrino. Water Cherenkov detectors have been successfully implemented in many of the neutrino detection projects including Irvine-Michigan-Brookhaven (IMB), Kamiokande, Super-Kamiokande, and Sudbury Neutrino Observatory (SNO).

The detector is generally based on an enormous (200 × 10^6 kg) high purity water tank with semihemi-
spherical PMTs of diameters in the range of 25–50 cm for lining the inner walls. The detection of neutrinos requires reliable and stable PMTs for at least 20 years of time period with no electronic failure. The water pressure in the tank can be as high as \( \approx 890 \text{ kPa} \). A combination of high pressure with the corrosive nature of high purity water on glass surface can lead to a detrimental environment adversely affecting the mechanical property of the PMT glasses. Currently, no detailed investigation of mechanical properties of PMT glass for neutrino detection has been reported. To avoid a catastrophic failure of PMT bulbs similar to the event in Super-Kamiokande neutrino detector,\(^5\) we intend to provide a framework for mechanical properties of the PMT glasses, generate data sets, and advance our fundamental understanding of these glasses for this application. In this study, we report our results from Vickers indentation, four-point bend, and ring-on-ring (R-O-R) biaxial flexural strength tests carried out to measure the mechanical properties of the PMT glasses in as-melted and chemically strengthened conditions.

Four-point bend and R-O-R strength testing techniques are constrained by strength-limiting flaws. These flaws acting as stress concentrators, first suggested by Inglis,\(^6\) control the failure behavior of the glass. Typically for these tests, the flaws are predominantly surface flaws and can be introduced during grinding and polishing process and/or sample handing stage. We also report on an increase in strength of glass via ion exchange treatment. Ion exchange technique generates surface compression stress via exchange of smaller alkali ions in the glass matrix, such as sodium with larger ones, such as potassium ions at temperatures below the glass transition\(^7\) resulting in a strengthened glass. Weibull statistics is used to evaluate and compare the failure behavior of these glasses.

**Experimental Procedure**

**Sample Preparation**

Representative compositional range of PMT glass currently in use for neutrino detection of the two vendor glasses, Vendor 1 and Vendor 2\(^1\), primarily borosilicate composition, are (in weight %): \( \text{SiO}_2 = 65–70, \) \( \text{Na}_2\text{O} = 6–9, \) \( \text{Al}_2\text{O}_3 = 4–7, \) \( \text{ZnO} = 0–3, \) \( \text{BaO} = 0–3, \) \( \text{CaO} = 0–1, \) and \( \text{B}_2\text{O}_3 = 15–18. \) These glasses were received as cullet and remelted. The glasses were melted in a platinum crucible in an electrically heated high-temperature furnace at 1450°C for 12–24 h first and then at 1520–1550°C for half an hour before pouring into a cylindrical stainless steel mold of \( \approx 30 \text{ mm inner diameter} \) to produce 30-mm-diameter glass cylinder. To determine the annealing temperature, glass transition (\( T_g \)) temperatures of these glasses were measured using differential scanning calorimeter (DSC—TA Instruments Model 2910). Pieces of remelted cullet samples, held in platinum pans alongside an empty pan – as standard, were heated at a rate of 10°C from room temperature to 610°C under a nitrogen atmosphere.

High-speed saw (Struers Discotom-5, Copenhagen, Denmark) was used to slice the bulk cylindrical glass at spindle speed of 2850 rpm 50 Hz to disk samples of \( \approx 3.5 \text{ mm thickness} \). High-speed saw was preferred to slow-speed saw because the bulk cylindrical glass was taking extremely long time to cut a single sample. The time required to cut a single disk sample was reduced from at least couple of days to less than a minute. The disk samples were subjected to 8" wet/dry silicon carbide (SiC) abrasive grinding pressure-sensitive adhesive (P.S.A) premium disks (MetLab Corp., Niagara Falls, NY) for grinding procedure with either kerosene or mineral oil as lubricating media. The average grinding grain size in \( \mu \text{m} \) was progressively reduced from 30.4, 20.9, 14.0, and 9.2–6.2 on both sides of the disk samples. The samples were thoroughly washed with DI-water between switching sides and changing grinding disks. Furthermore, 3 \( \mu \text{m} \) and 1 \( \mu \text{m} \) polycrystalline (black) diamond particles suspension (MetLab Corp., Niagara Falls, NY) solution was used to polish the disk samples. The final polished glass was ultrasonically cleaned in DI-water and ethanol and stored in desiccant chamber for testing. The average of arithmetic mean surface roughness, \( R_a \), equation 1, and root-mean-square (rms) surface roughness, \( R_q \), equation 2, was determined by white light interferometer (ZYGO® surface profilometer Model NV5000–5032). MetroPro® software provided by Zygo® determined the numerical values. Multiple surface scans on the samples were measured to ensure the images were representative of the whole surface.

\[
R_a = \frac{1}{L} \int_0^L |z(x)|\,dx \tag{1}
\]
For rod samples, the glass was poured in a cylindrical graphite mold and annealed at 550°C. Glass rods hand-drawn using a technique called cane pulling. The rods diameter varied between about 3 and 6 mm because of the handmade nature of the process. Calculation of modulus of rupture (MOR) accounts for the variation; however, strength-limiting flaws are of primary concern for MOR tests. The rod samples were cut into lengths of approximately 50 mm and used for ion exchange treatment, discussed under the section “Ion Exchange Test”.

Mechanical Testing

We used a Shimadzu indenter—Micro Hardness Tester Model HMV-2000, Shimadzu Corporation, Kyoto, Japan—for indentation measurements. ASTM E384-99 was used for indentation experiment. Vickers hardness of the polished disk samples both before and after ion exchange was measured using different applied loads 200, 300, 500, and 1000 gf. Ten to twenty individual measurements were made for each load, with each sample held under load for 15 s. The diagonal of each indent was measured using in-built micrometer. The hardness is calculated using equation 3.

\[
H_v = \frac{1.8544F}{d^2}
\]  

(3)

where \( F \) = force in kgf and \( d \) = average of the two diagonal in mm.

The four-point bend testing fixture features four steel cylinders perpendicular to the test sample to allow the load to settle in the x direction, two above and two below, resulting in contact at four points along the rod. Below the two support rods, another steel cylinder runs lengthwise through the holder to offer freedom of movement in the z-direction, and below that a steel beam reinforces the plastic body. The fixture also features a point contact in the form of a steel ball bearing above the support span, which allows for 360-degree articulation during load application. The loading span of the test setup is 20 mm while the support span is 40 mm. The load applied at the time of failure was used to calculate modulus of rupture (MOR) for each sample using the following equation:\(^9\):

\[
MOR = \frac{5.09La}{d^3}
\]  

(4)

where \( L \) is the breaking load in N, \( a \) is the separation between adjacent support and loading edges in m, and \( d \) is the average diameter of the sample in mm. Thirty untreated samples of each vendor glass and 15 samples of each ion exchange schedule for each glass were selected for flexural strength testing. The diameter of the samples was measured in three places prior to testing to ensure limited variation along the length of each rod and to calculate an average diameter.

A custom fixture designed in accordance with ASTM C1499-08\(^10\) was attached to Instron 5566 (Model 5566P6016, Instron, Norwood, MA) to measure R-O-R flexural strength. The support and load ring were 25 mm and 10 mm in diameter. Glass sample was concentrically placed between support and loading rings before load application. An average of thirty samples was tested for each of the vendor glasses. R-O-R strength test is a destructive test method. In one of the samples, multiple failures occurred around the top of the support ring, which was considered as an invalid test and discarded. Strength tests were carried out in displacement control at a rate of 0.5 mm/min. This rate is deemed rapid enough to avoid slow crack growth effects.\(^11\) The R-O-R equibiaxial flexural stress was calculated as follows:

\[
\sigma_f = \frac{3F}{2\pi rt^2} \left(1 + \nu \right) \ln \left( \frac{D_S}{D_h} \right) + \left(1 - \nu \right) \frac{(D_S^2 - D_h^2)}{2D^2}
\]  

(5)

where \( F \) = the applied force in N, \( \nu \) = the Poisson’s ratio, \( D_S \) = diameter of the support ring in mm, \( D_h \) = diameter of the loading ring in mm, \( D \) = radius of the glass sample in mm, and \( t \) = the thickness of the sample in mm.

Ion Exchange Test

The ion exchange technique consisted of immersion of the samples in a molten potassium nitrate (KNO\(_3\)) bath. Based on a study of soda-lime silicate glass,\(^12\) samples of each vendor glass were treated at two different temperatures, 470°C and 480°C, for a dwell period of 24 h. The use of potassium in the PMT glass composition is limited due to the presence of naturally occurring contamination of radioactive iso-
tope $^{40}\text{K}$ (0.1%), which can cause unwanted background counts.\textsuperscript{13,14} For this reason, the only use of pure $^{39}\text{K}$ is suggested for PMT development. An optimization of ion exchange parameters (time and temperature) is beyond the scope of this work.

Samples were arranged in a specially designed apparatus using two vertically aligned pieces of stainless steel mesh and a baseplate, which allowed the rods to be treated while upright, exposing the length of the rods to the surrounding molten salt. Disk samples were held similarly, using a narrower stainless steel mesh, shaped specifically for the task of exposing the surface to the salt bath. After treatment was completed, the sample holder apparatus was removed from the bath and allowed to cool to room temperature, at which point samples and holders were rinsed with water to dissolve excess salt deposits on the glass surface. The samples were stored in a desiccator to protect the surface treatments.

Depth of exchange in these glasses was examined using electron microprobe (JEOL JXA-8200, Tokyo, Japan) mapping of elements on cross sections of rod samples. A total of sixteen total rods, eight of each vendor glass, four of each ion exchange treatment, were selected to calculate an average exchange depth of $\text{K}^+$. Samples were cut into short lengths after the ion exchange bath and mounted vertically in epoxy and ground and polished with 1-μm diamond polish. Conductive sputter coating was performed to facilitate microprobe testing.

The electron microprobe was set up to align the beam approximately with the interface of the glass and epoxy mount. Measurements were carried out along 10 rows perpendicular to the sample edge, spaced 4 μm apart at 2-μm intervals along the row. Silicon, sodium, and potassium levels were tracked from the edge of the sample up to approximately 50 μm depth.

Neutrino detection requires good optical transmission through the PMT glass between 350 and 500 nm. These wavelengths were chosen because the majority of detectable Cherenkov light falls in this range.\textsuperscript{15} Absorbance spectrum of disk sample was measured using a Perkin–Elmer Spectrophotometer (Lambda 950 L6020036, Perkin-Elmer, Shelton, CT) before and after ion exchange treatment. The samples were cleaned with ethanol immediately before testing to eliminate any surface contamination. Any increase in the absorbance property would lead to reduction in signal detected, which is detrimental to the neutrino detection project.

Result and Discussion

Glass Transition Temperature

Glass transition temperatures for Vendor 1 and Vendor 2—estimated using the tangent intercept method on the temperature versus heat flow graph—were found to be 510°C and 540°C, respectively. A temperature of 550°C was deemed suitable to anneal the glass sample for an hour and cooled at 5°C per minute to room temperature.

Roughness Characterization

The roughness parameters for polished Vendor 1 glass are $R_a$/μm = 6.62 ± 2.34 and $R_q$/μm = 9.63 ± 2.64. The value is averaged over eight individual scans on a sample. Figure 1 shows a representative 2-D interferometry scans of the glass. The cross-linked polishing marks seen in the figure can be associated with the repetitive nature of the polishing procedure. The change is color contrast indicate regions or points as possible stress concentrators, which are a major factors in flexural tests.

Indentation Hardness

In Fig. 2, Vendor 1 has higher Vickers hardness than Vendor 2 for all four loads; however, for both vendor glasses, the hardness decreases with increasing load. For ten indents performed for each the loads, the standard deviation reported is a factor of ten smaller than the difference in hardness number for the two
vendor glasses. This shows that the difference observed in hardness for different loads is real and inherent to the surface of the vendor glasses.

Figure 3 shows the measured hardness of the Vendor 1 glass at four different loads for treated and untreated samples in salt bath. There is no measurable change in hardness for either exchange process when accounting for error in the measurements up to a load of 500 gf, beyond which there is measurable decrease in hardness at 1000 gf. However, for Vendor 2 glass, Fig. 4 shows a measurable increase in hardness after the 470°C treatment for all indentation loads, as compared to untreated sample. It shows that for this particular treatment condition, a uniform compressive layer was formed on the surface. For indentation of samples treated at 480°C, the compressive layers close to the surface, indented by 200 and 300 gf loads, do not show an increase in hardness as compared to the untreated sample. This can be attributed to the relaxation of compressive layers. However, deeper regions indented by 500 and 1000 gf loads are still under compression; thus, we observe higher hardness values.

On top of the relaxation of glass during the high temperature exchange, structural relaxation during the indentation process also should be considered to evaluate the results. The compressive stress on the surface of the glass, introduced during ion exchange process, would increase hardness at greater depths of strengthened layer reached with higher indentation loads, while temperature-induced structural relaxation might be responsible for lower hardness at the shallow surface affected by the 200 and 300 gf loads. At 500 and 1000 gf, lower hardness measured may be attributed to local stress release at higher loads.

Average values of diagonals of the indentations made by 200, 300, 500, and 1000 gf loads are 25, 33, 38, and 55 microns, respectively. In the case of strengthened glass samples, the ion-exchanged layer thickness is about 15–20 μm. Recent developments in instrumented nanoindenters have enabled mapping of surface properties of with micrometer resolution.
These nanoindenters can measure the force and displacement during penetration thus allowing simultaneous determination of the hardness, elastic modulus, and fracture toughness of materials. Advancements in tribology of glasses indicate tribochemical reaction on the sliding wear of glasses that impact the wear resistance of the glasses. The Archard coefficient ($K$) used to describe sliding wear is defined as:

$$V = KA_r L = \frac{KLP}{H}$$

where $V$ is the volume loss, $A_r$ is the real contact area, $L$ is the sliding distance, $P$ is the applied load, and $H$ is the hardness of the softer surface. In our case, the indenter pushes through the ion-exchanged layer during the testing. Simple indentation does not capture the whole dynamics of the processes. Our hardness data calculated using equation 4 are intended to comparison purpose only. Detailed nanoindenter measurements will be needed to determine actual differences between the mechanical properties of the glasses.

**R-O-R Test**

Figure 5 shows the distribution of R-O-R equibiaxial stresses to sample thickness, showing the range of stresses. The mean flexural stresses ± standard deviations are $112.56 \pm 43.99$ MPa and $102.41 \pm 31.43$ MPa for glasses Vendor 1 and Vendor 2, respectively.

The lowest flexural stress recorded for a Vendor 1 sample with thickness of 3.235 mm is $60.99$ MPa, and the highest flexural stress, $235.09$ MPa for sample of 2.7925 mm for Vendor 1. Our data suggests that sample thickness does not affect the strength as the strength of glass strongly depends on surface finish, especially at subsurface-level damage.

Weibull parameters are modulus, $m$, and characteristic strength, $\sigma_0$. Characteristic strength is defined as the strength at which there is a 63% probability of failure of a given sample. This is calculated by applying

![Fig. 6. Weibull plot of Vendor 1 and Vendor 2.](image)

![Fig. 7. Weibull plot of 4-point bend strength of Vendor 1 glass at various exchange treatments.](image)
linear regression to the failure data for each glass treatment. Modulus values for Vendor 1 and Vendor 2 are 3.00 and 3.50, respectively. Characteristic strengths (MPa) for Vendor 1 and Vendor 2 are 126 and 114, respectively. Figure 6 shows a Weibull distribution plot. It is on the lower end of typical practical values of $m$. In the ideal case of a perfectly uniform flaw population, the Weibull modulus would approach its theoretical upper limit of $m \to \infty$. Larger values of $m$ indicate a greater predictability and hence greater reliability. The high scatter of data points are attributed to the presence of flaws, we do not know the size or volume or distribution of the flaws in these glasses.

### Four-point Bend Test

Four-point bend tensile test was performed to examine the strength change between untreated and ion exchange-treated glass samples. Vendor 1 glass showed 47.5% increase in strength when treated at 470°C, and 43.9% when treated at 480°C. Vendor 2 glass results show a 58.3% and 28.8% increase in strength after treatment at 470°C and 480°C, respectively.

Figures 7 and 8 show Weibull distributions for each vendor glasses and ion exchange-treated samples. The characteristic strengths of both vendor glasses increased for the two different temperature treatments. The strength of Vendor 1 increased by 46.1% and 39.5% for glass samples treated at 470°C and 480°C, respectively. The Weibull statistic had a tighter distribution as modulus significantly increased as compared to the untreated samples. For the Vendor 2 glass, the strength increased by 56.5% after 470°C treatment and 28.5% after 480°C treatment. Improvement in Weibull modulus of Vendor 2 was only marginal compared to Vendor 1.

### Optical Absorbance

Figures 9 (a) and (b) show absorbance before and after ion exchange treatment for Vendor 1 and Vendor 2 glasses, respectively. The results were normalized to a

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Fig. 8. Weibull plot of 4-point bend strength of Vendor 2 glass rods at various exchange treatments.

Fig. 9. Normalized absorbance of light between 350 and 500 nm wavelengths of (a) Vendor 1 glass samples “A” and “B” before and after exchange and (b) Vendor 2 glass samples “A” and “B” before and after exchange.
"per millimeter" absorbance to eliminate thickness factor of the selected samples. In addition, percent change in absorbance across the required wavelength was determined using:

$$\text{% Difference} = \frac{|A_U - A_T|}{A_U} \times 100$$  \hspace{1cm} (7)

where $A_U$ is the normalized absorbace of the untreated sample and $A_T$ is the normalized absorbance of the treated sample. Absorbance of Vendor 1 glass was comparable to that of Vendor 2 glass. Vendor 1 sample “A” showed an average of 8.88% decrease in absorbance per mm before and after treatment, while Vendor 1 sample “B” showed 3.51% decrease. For Vendor 2 sample “A,” 12.12% decrease was calculated and 2.56% for Vendor 2 sample “B”. In both cases, sample A had the lowest absorbance across the wavelength of the measurement. Our results show chemical strengthening has not significantly affected the optical transmission of these glasses.

**Ion Exchange**

The exchange profile of potassium for each ion exchange treatment was measured using electron microprobe mapping. Figure 10 shows intensity of the three elements, Na, Si, and K tracked against the depth of measurement for (a) Vendor 1 glass treated at 470°C:

![Fig. 10. Exchange profile of (a) Vendor 1 glass treated at 470°C for 24 h, (b). Vendor 1 glass treated at 480°C for 24 h, (c). Vendor 2 glass treated at 470°C for 24 h, and (d). Vendor 2 glass treated at 480°C for 24 h.](image-url)
Table 1. Summary of Mechanical Properties and Chemical Exchange Profiles

<table>
<thead>
<tr>
<th>Glass composition</th>
<th>Exchange treatment</th>
<th>Average MOR (MPa)</th>
<th>Standard deviation (MPa)</th>
<th>Weibull modulus</th>
<th>Characteristic strength (MPa)</th>
<th>Exchange depth (µm)</th>
<th>Maximum K⁺ intensity (counts)</th>
</tr>
</thead>
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<tr>
<td>Vendor 1</td>
<td>None</td>
<td>133.96</td>
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<td>5.9</td>
<td>144.8</td>
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<td></td>
<td>470°C, 24 h</td>
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<td>6.8</td>
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<td>22.45</td>
<td>11.4</td>
<td>202.0</td>
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<td>3392</td>
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<tr>
<td>Vendor 2</td>
<td>None</td>
<td>156.43</td>
<td>37.56</td>
<td>4.6</td>
<td>171.1</td>
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</table>

for 24 h, (b) Vendor 1 glass treated at 480°C for 24 h, (c) Vendor 2 glass treated at 470°C, and (d) Vendor 2 glass treated at 480°C for 24 h. Table 1 summarizes the mechanical properties and exchanges profiles of the glasses. K⁺ exchange depth is higher for Vendor 1 that Vendor 2 which would suggest a more open structure for Vendor 1. Deeper exchange depth at 480°C (Fig. 10d) than 470°C (Fig. 10c) for Vendor 2 has resulted in higher strength as can be observed in Weibull plot, Fig. 8. In the first 5 µm, we observe a leveling off of K⁺ exchange, which suggests that Na⁺ ions have replaced K⁺ ions from the surface. This feature is prominent in Fig. 10(a). Higher temperature treatments resulted in an increase in maximum concentration of potassium ions at the sample surface for both glasses.

Conclusion

Our results show that Vendor 1 glass has slightly higher indentation hardness than Vendor 2 for all test loads suggesting a better surface response to localized stress. As the compositions of these glasses are similar, this difference is not significant. Ion exchange tests, as expected, show considerable increase in strength of both vendor glasses without significant decrease in the transmission of light. This shows promise for designing potentially stronger glasses for future neutrino detection experiments. Detailed knowledge of chemistry and thermal history of glasses and chemistry and dynamics of the modified layer on the surface of the glasses is critical to understanding the failure of these glasses and ensure their stability through long neutrino detection experiments.

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