

STRENGTH OF GLASS AND GLASS FIBERS

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ABSTRACT

The article provides a selective review on strength of glass and glass fiber, covering effects of surface flaw and surface hydrolysis on the usable strength of glass (USG). Application of Griffith-Inglis-Orowan theory on fracture of solids is demonstrated, elucidating importance of stress-assisted hydrolytic effect on glass USG and associated change in glass surface energy. The fundamental understanding of glass fracture supports critical needs for development of new glasses and new durable and/or resin compatible hydrophobic coatings to significantly improve USG of glass and fiberglass products, respectively.

1. FRACTURE OF GLASS AND GLASS FIBERS

1.1 Fundamental of Solid Fracture

Theoretical tensile strength of solids, according to Orowan [1], is proportional to Young's modulus (E) and surface energy (γ_0) of the material as

$$\sigma_{th} = (E\gamma_0/r_0)^{1/2} \quad (1)$$

where r_0 is the equilibrium distance between atomic centers. Experimental measurements, however, report that glasses typically have tensile strengths much lower than the theoretical values by as much as one order of magnitude. Unlike crystalline materials, for which grain boundaries serve as one type of defect, glass defects mostly come from surface "damage" or surface flaw as one of the key factors of lowering the usable strength of glass (USG) from its expected theoretical level.

Surface flaws of a given size (c) serve as a stress concentrator when glass is subject to an applied tensile load; these weak spots cause glass to fail at a tensile stress level well below the theoretical expectation. By the Griffith energy-balance criterion, apparent or measured strength (σ_m) of a solid is defined by [2, 3]:

$$\sigma_m = (2E\gamma_0/\pi c)^{1/2} \quad (\text{plane tensile stress}) \quad (2a)$$

$$\sigma_m = [2E\gamma_0/\pi(1-\nu^2)c]^{1/2} \quad (\text{plane tensile strain}) \quad (2b)$$

Inglis further demonstrated [4] that tip geometry of the flaw, in terms of its size c and radius, ζ_{tip} , can significantly magnify the stress applied onto the material, which affects σ_m , according to

$$\sigma_m = (E\gamma_0/4r_0)^{1/2} (\zeta_{tip}/c)^{1/2} \quad (3a)$$

$$\text{or} \quad \sigma_m = \sigma_{th}/2 (\zeta_{tip}/c)^{1/2} \quad (3b)$$

Equation 3b implies that the maximum measured strength of "flaw-free" samples will be approximately 50% of its theoretical strength and the same size of a critical surface flaw with a sharper crack tip (or lower radius at the crack tip) will further reduce the material strength [5].

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It becomes clear that experimentally measured glass strength is not an intrinsic property of the material. Besides composition, atomic structures of glass are affected by their thermal history in terms of melting temperature, cooling rate, degree of annealing, degree of aging under conditions under which they are stored before application, and fatigue in terms of test or application conditions, including temperature, humidity, and cleanness of laboratory, and sample strain rate [6-9]. Furthermore, it is expected that the glass “surface defects” can be generated from “contact damage” even from finger contact during sample handling.

When developing new glass and glass fiber compositions, keeping in mind the multiple factors that affect USG, it is critical to test all samples that are made by the same method under the same laboratory conditions in order to screen composition effect on glass strength.

In reporting and comparing glass strength, “pristine” strength refers to testing samples made under controlled humidity, not being “damaged” by any physical contacts in handling, and tested under the same humidity environment within a very short period of time after the samples are made. “Inert” strength means that the samples are tested in liquid nitrogen to minimize any moisture interaction with glass or glass fiber surface under an applied force. In this case, the samples can be tested after aging under specific conditions or as its “pristine” form without any treatment. “Inert” strength of the “pristine” glass is significantly higher than that of “pristine” glass and hence, closer to the glass intrinsic property.

1.2 Glass Fracture from Microscopic Defects

One of the most detrimental factors impacting glass strength is glass surface attack by corrosive media in the form of liquid or vapor, including water, acid, and base [10-14]. Figure 1 illustrates the effect of fiber surface flaw geometry on silica fiber “inert” tensile strength as the fibers treated in hydrofluoric acid vapor over time [5]. Prediction from the data set suggests that for the silica fibers with very sharp surface flaws, i.e. $\zeta_{tip} \ll c$, its strength is approximately 35 - 40% of its theoretically predicted value of ≥ 17 GPa.

Figure 2 shows fiber failure strain of boron-free E-CR fibers with and without aging up to 270 days at 50°C under 80% relative humidity (RH) [15]. The tests were conducted by using the two-point bending method [16] at room temperature (RT) under 50 %RH and in liquid nitrogen (LN), respectively. Several characteristics can be summarized from the results as follows: First, at semi-logarithmic scale, the two sets of data can be reasonably fit by using linear regression least square method. The total reduction in fiber failure strain is about 12.5% for fibers tested at RT - 50%RH and 13% for fiber tested in liquid nitrogen, respectively. Therefore, it is reasonable to conclude that fiber aging under stress-free conditions results in approximately 13% deterioration in terms of failure strain. Secondly, in terms of absolute failure strain comparing the two test conditions, ϵ_f (LN) is significantly higher than ϵ_f (RT-50%RH); the ratio of the average values for the same aging durations between the two cases is between 2.2 and 2.3, supporting that fiber failure at much higher load or applied stress once moisture of water is minimized or eliminated under which the samples are tested.



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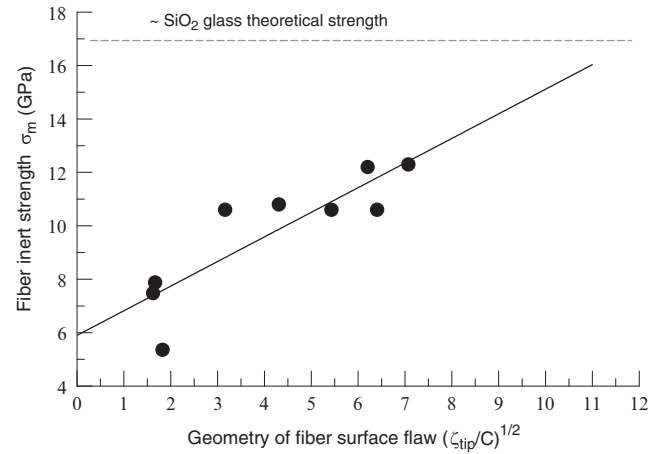


Figure 1. Silica Glass Fiber Tensile Strength as a Function of Fiber Surface Defect Geometry Characterized by the Ratio of Tip Radius (ζ_{tip}) of the Surface Flaw over the Flaw size (c) (solid line is determined by using least square linear regression analysis; the plot is constructed based on [5]).

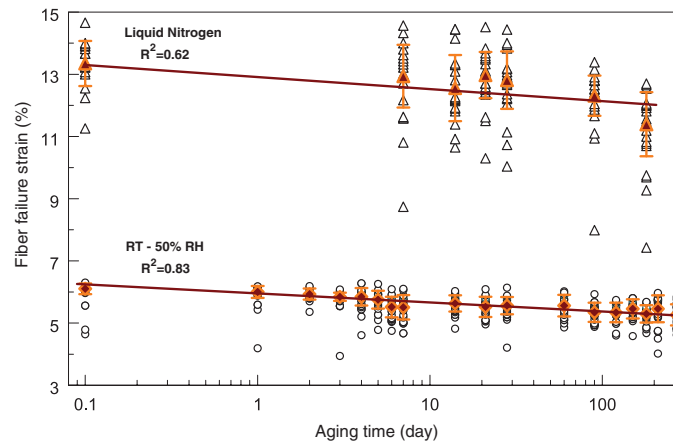


Figure 2. Fiber Failure Strain of E-CR Fibers Measured at Room Temperature under 50% RH and in Liquid Nitrogen as a Function of Fiber Aging under 50°C - 80% RH Conditions (open circle and triangle represent individual measurements; filled diamond and triangle represent average values; error bars represent one standard deviation; solid lines are obtained by using linear regression method fitting average values of the data sets; 20 measurements were performed per data set) [15].



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Combining the aging test results, for E-CR glass with low alkali contents and free from boron and fluoride, the study demonstrated that moisture water interaction with the surfaces of fibers being under tensile strain or tensile stress plays a dominant role on its failure over hydration or aging treatment of the fibers without being stressed. It follows that the kinetics of stress-assisted hydrolysis on the fiber surfaces should not be significantly affected by the preexisting “layer” of hydration created from the aging treatment. In turn, one can reason that the hydroxyl groups (Si-OH) formed on the fiber surfaces during aging should be immobile during the growth of crack under the applied stress or strain and hence, newly generated Si-OH groups at the front of surface flaws, i.e., stress-assisted hydrolysis, should dominate the fiber failure strain or failure stress. The mechanism of glass fatigue in a humid environment was proposed and experimentally demonstrated by Hillig & Charles [17] and Wiederhorn [18, 19].

The stress-assisted hydrolysis of the glass near the tip of surface flaws can result in significant glass surface energy (γ) reduction; literature data shows that quartz crystals change in surface energy with and without hydration by as much as 10 times [20-23]. Structure of crystalline quartz and fused quartz glass is very different; in dry liquid nitrogen their perspective ratio is about 0.43 (2.0 J/m² for crystalline quartz over 4.6 J/m² for fused quartz glass) [24]. However, their perspective changes in surface energy to hydration are expected to follow the same trend [25]. Therefore, the glass fibers tested should become much weaker under ambient conditions over liquid nitrogen. Our estimation on the surface energy ratio, $\gamma(\text{LN})/\gamma(\text{RT-50\%RH})$, derived from the study [15] was close to 3.4 ± 0.2 for fibers aged up to 180 days. The surface energy ratio can be derived from Eq. 3a, in which fiber modulus is considered as a strain-dependent variable, i.e., Secant Modulus, according to Gupta and Kurkjian [26].

1.3 Glass Fracture from Macroscopic Defects

As the size of glass surface flaws becomes larger, glasses fail at lower applied stresses, i.e., lower USG, as illustrated in Figure 3 [27]. Within each flaw size range, instantaneous strength represents the samples without any aging effect, and endurance limit represents the samples experienced some levels of aging event before or during the mechanical tests. In product design, one should consider the use of the endurance limit of the glass that has been tested under a relevant application environment to ensure the maximum safety of the products to be used.

Fracture of glass and glass fibers under an applied tensile load initiates at a point of its weakest point according to the Weibull statistical theory [28], which has been widely used to study distribution of glass stress at breakage in relationship to change of glass chemistry or glass thermal history or test conditions. According to the Weibull method, an accumulative probability of failure (P_f) of a solid at an applied tensile stress, σ_f , follows

$$P_f = 1 - \exp[-(\sigma_f/\sigma_0)^\beta] \quad (4a)$$

$$\text{or } \ln[-\ln(1-P_f)] = \beta \ln(\sigma_f) - \beta \ln(\sigma_0) \quad (4b)$$

where β and σ_0 are the statistical linear regression fitting parameters, which are often called Weibull modulus (or shape parameter) and characteristic stress, respectively.

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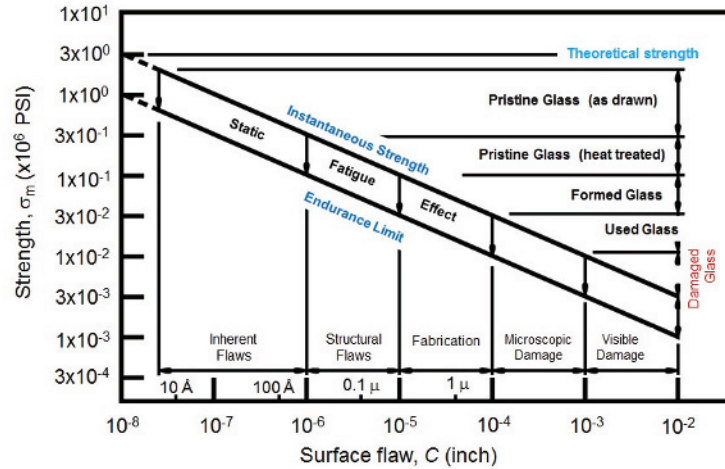


Figure 3. Effect of Surface Flaw Size on Glass Tensile Strength (redraw after Mould [27])

Figure 4 illustrates a Weibull plot of pristine tensile strength distributions of S-Glass, R-Glass, and E-Glass fibers. Under the same sample preparation and test conditions, the average fiber tensile strength ranks in an order S-Glass (5500 ± 133 MPa) > R-Glass (4135 ± 280 MPa) > E-Glass (3215 ± 198 MPa). The Weibull modulus (β -value) of the S-Glass is substantially higher than both R-Glass and E-Glass [29]. In this case, to minimize the size effect on the fiber strength [6,7], the fiber gage length of all samples was kept the same (1 inch) and the diameter of the fibers was controlled at 10 ± 0.5 μm ; the size can be attributed to the change in the defect population as fiber gage length and/or diameter varies.

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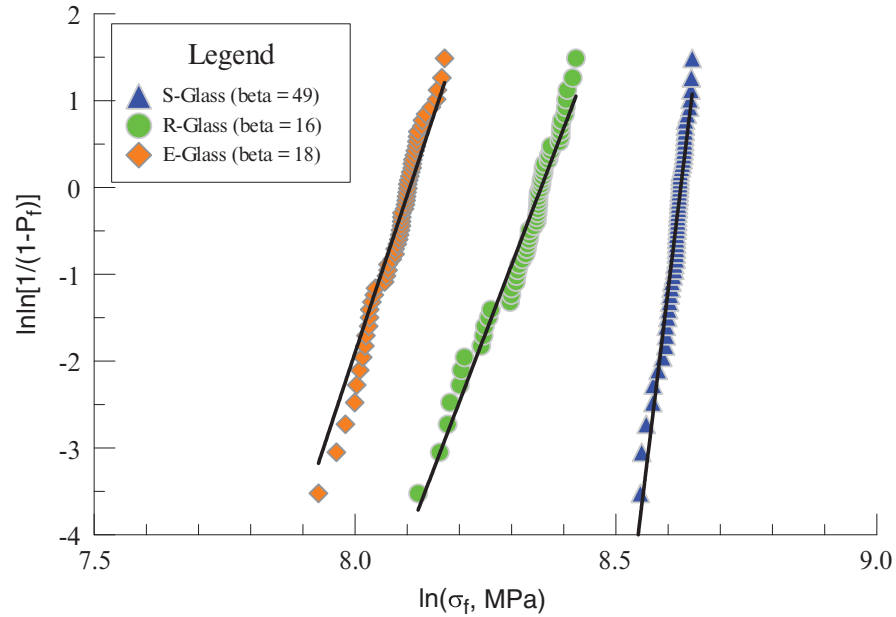


Figure 4. Weibull Plot of Representative Glass Fibers used for Plastic Reinforcement, E-Glass, R-Glass, and S-Glass [29].

2. YOUNG'S MODULUS OF GLASS AND GLASS FIBERS

The strength of glass is a function of Young's modulus (Eq.1 & 2). For oxide glasses, Sun's binding energy approach [30] has been adopted to calculate Young's modulus by Makishima and Mackenzie [31]. The model was later modified by Zou and Toratani [32]. In both models Young's modulus of glass is approximated by a linear combination of contributions from individual glass constituents. Similar approaches to predicting the Young's modulus of complex glass systems can be also found elsewhere [33, 34]. A general presentation of a linear composition model is illustrated in Figure 5, which provides a simplified view of the listed oxide contributions to glass Young's modulus.

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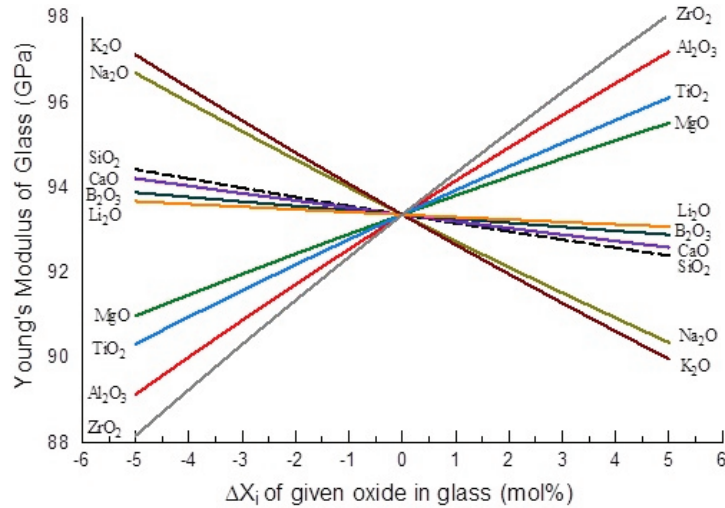


Figure 5. Oxide Effects on Silicate Glass Young's Modulus [29]

In practice, significant deviations between the measured and the model-derived values have been reported, especially in complex multi-component glass systems [35]. There are many key factors contributing to the discrepancies reported. First, local structure or surrounding oxygen environments of glass network formers (SiO_2 , B_2O_3) and conditional network formers (Al_2O_3) vary depending on concentrations of alkalis (Li_2O , Na_2O , K_2O), alkaline earth (MgO , CaO , SrO), and their relative proportions [36-41]. The linear composition models cannot account for the structural variations or speciation of the network formers, such as SiO_2 , B_2O_3 , and Al_2O_3 .

Secondly, glass density or molar volume is affected by fictive temperature or thermal history of the samples in terms of glass structure relaxation [42-44]. In turn, annealed glass has lower fictive temperature, higher density, and hence, higher Young's modulus as compared to the quenched form of the same glass composition. Figure 6 compares the measured fiber glass modulus as obtained by using a sonic method comparing with the calculated modulus. In general, a parallel downshift correlation line can be drawn relative to the ideal 1:1 line, which likely results primarily from thermal effect. The glass models of Young's modulus found in literature are built from experimental data generated from testing annealed bulk glass samples [32-34] and the measured values shown in Figure 6 were collected from measuring fast quenching fibers without annealing. The thermally induced change of glass Young's modulus has been reported to vary between 10% and 20% [43, 44].

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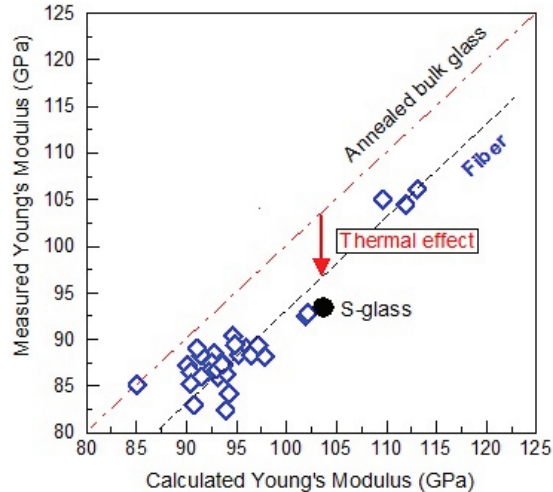


Figure 6. Comparison between Measured Young's Modulus of Glass Fiber and Model Calculated Modulus, Suggesting Thermal Effect.

3. IMPROVEMENT OF USABLE STRENGTH OF GLASS AND GLASS FIBERS

The detrimental effect of surface contact damage on USG has been well understood as reviewed previously, surface protection on glass and glass fibers are important to raise USG. For example, by applying protective sizing on glass fiber, the fiber surface flaw distributions can be altered, resulting in USG improvement [7]. The second critical damage mechanism is glass surface hydrolysis under the applied load. The hydrolysis involves interaction of moisture water or other corrosive media with surfaces of glass or glass fibers. For alkali-free glasses, the adverse effect of hydrolysis on USG is mostly pronounced when the glass object is stressed as comparing with the glass object aged under the stress-free conditions (cf. Fig 2).

Realizing the importance of stress-assisted glass hydrolysis effect, it becomes clear that it will be advantageous by applying a hydrophobic coating on bulk glass [45] or more hydrophobic, resin compatible sizing on glass fibers [48, 49], which will prevent water molecules access to surface defects or surfaces of glass or glass fibers while the objects are under applied load. Prevention and/or slowing down molecular water reaction with glass surface defects in application can be significant in boosting USG according to the glass fatigue mechanism [17-19]. For example, hermetic coating has been long recognized as an important technology and applied to optical silica fibers in telecommunication, which prevents the optical fibers from aging in field applications [48].

Besides thermal tempering [49], improvement of USG can be realized by using various chemical tempering processes, introducing a compressive layer on surfaces of glass and/or glass fibers depending on specific compositions of the glasses commonly containing appreciable amount of sodium. Two methods have been commonly used in commercial glass productions are glass

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surface de-alkalization and ion exchange [50-53]. Table 1 summarizes improvement of glass strength after surface treatments with various chemical methods [50].

It should be noted that most of the aforementioned methods, besides the use of sizing, are technically feasible on bulk glasses in form of plate, tubing, and containers; but not feasible in production of continuous fibers.

Table 1. Improvement of Average Impact Resistance (N·cm) of Glasses Treated by using Various Chemical Tempering Processes (De-alkalization, Ion-exchange, and Combined Methods) [50]

De-alkalization Agent	As-received	De-alkalization	Ion-Exchange*	Combined [#]
SO ₂	76	93 (22% ↑)	105.5 (39% ↑)	144 (89% ↑)
NH ₄ Cl	76	96 (26% ↑)	105.5 (39% ↑)	152 (100% ↑)
(NH ₄) ₂ SO ₄	76	92 (21% ↑)	105.5 (39% ↑)	135 (78% ↑)
AlCl ₃	76	88 (16% ↑)	105.5 (39% ↑)	120 (58% ↑)
(NH ₄) ₂ SO ₄ +AlCl ₃ (10:1)	76	88 (16% ↑)	105.5 (39% ↑)	130 (71% ↑)
NH ₄ Cl+AlCl ₃ (10:1)	76	96 (26% ↑)	105.5 (39% ↑)	129 (70% ↑)
NH ₄ Cl+(NH ₄) ₂ SO ₄ (1:1)	76	98 (29% ↑)	105.5 (39% ↑)	132 (74% ↑)

* Ion-exchange process: soak container in 70°C - 200 ml water solution containing 34g KNO₃ - 69g KCl - 8.5g K₂SO₄ and followed by heat-treatment at 500°C. [#] De-alkalization first and followed by ion-exchange treatment

4. SUMMARY

A selective literature review on strength of glass and glass fiber was made covering effects of surface flaw and surface hydrolysis on USG. Based on fracture mechanism of Griffith-Inglis-Orowan theory, specific examples are provided to elucidate importance of stress-assist hydrolytic effect on USG, which highlights more pronounced detrimental impact of stress-assisted glass surface hydrolysis over the effect of stress-free hydrolysis. Although it is important to develop new glass chemistry for greater pristine strength as shown in the review, in commercial applications, development of new coating materials for bulk glass or sizing for fiber glass is equally important to raise USG. Especially the latter offers the improvement of USG for existing glass or fiber glass products with minimum or without changing of existing processes, i.e., glass melting and product forming.

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