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Using the two-point bend technique to determine failure stress of pristine glass fibers

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ABSTRACT

Two-point bending (2pb) is the simplest technique for the measurement of failure strains of glass fibers under a variety of experimental conditions. There is little chance of damage to fibers even when testing in liquid nitrogen, leading to reproducible and precise measurements of failure strain with Weibull moduli of greater than 100 measured routinely. However, a limitation of 2pb is that it measures failure strain not failure stress, and thus the Young's modulus of the sample must be known *at the failure strain* in order to evaluate the failure stress.
In this paper the failure strains, under both inert and ambient conditions, for a number of conventional glasses (commercial silica, soda-lime silicate, and E-glass), as well as a number of simple glasses, including a nepheline glass and a range of binary sodium and potassium silicate glasses are presented. These strain values are converted to failure stresses using known or estimated non-linear modulus parameters and compared with strength values found in the literature. For silica optical fibers, the failure stresses calculated from 2pb failure strains vary from 12.1 ± 0.2 to 14.4 ± 0.3 GPa in inert (liquid nitrogen, 77 K) conditions and 7.0 to 7.3 ± 0.1 GPa in ambient conditions (room temperature, 50% RH), compared to reports of 11–14 GPa for liquid nitrogen and 4–5 GPa ambient tensile strength measurements. For a commercial E-glass, the calculated failure stress from 2pb, is 5.1 to 5.2 ± 0.1 GPa in inert conditions and 3.7 to 3.8 ± 0.1 GPa in ambient conditions, compared to reported tensile strengths of 5.3 GPa and 3.0–3.8 GPa, respectively. The failure stresses for binary alkali silicate glasses calculated from 2pb failure strains are 2–3 times greater than those reported in the literature.

1. Introduction

Strength is one of the most important properties of glass, but also one of the most difficult to measure. Griffith [1] showed that practical strength is greatly reduced from its theoretical value due to flaws that form during processing or handling. Substantial effort has gone into preparing and testing "flaw free" or pristine samples to determine intrinsic strength [2]. The measurement of intrinsic strength of glass is of significance because without the effect of surface flaws, it is determined by glass composition and can be related to the nature of the glass structure and bonding. In addition, glass strength is reduced due to environmental fatigue [3–5], with water being the cause of this degradation [6]. The inert strength of glass is the strength measured under conditions where there is no environmental fatigue [7]. To avoid environmental fatigue, strength measurements have been carried out either in vacuum, where water activity is very low [8], or in liquid nitrogen, where the kinetics of the water degradation reactions are arrested [9].

1.1. Conventional glass strength measurement

Freshly drawn glass fibers are often used in studies of glass strength because they can be prepared and handled in such a way as to avoid damaging their pristine melt surfaces. Conventionally, strength is measured by tensile tests [9–13], three-point bend tests [14] and four-point bend tests [15]. In a tensile test, the sample must be gripped on both ends and this generally damages the sample surfaces causing a decrease in measured strength. Another disadvantage of the tensile test is that the test volume includes the entire fiber, which increases the probability of finding flaws and increases scatter in measured values [7]. Three-point bend and four-point bend tests significantly reduce the probability of encountering a critical surface defect, due to smaller volume that is effectively under tension compared to typical tensile tests, but may create strength-limiting critical flaws where the testing fixtures touch the pristine surfaces.

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1.2. Two-point bend failure strain measurement

Although the two-point bending (2pb) technique was used as early as 1944 [16], extensive use in testing fibers began in 1980 [17]. In a 2pb test, a pristine section of glass fiber, diameter d, is bent into a U-shape between two parallel face plates, one of which travels towards the second at a constant faceplate velocity (v_{fp}), compressing the 'U' until failure (Fig. 1). The gap between face plates at failure (D) is recorded, and the failure strain (ε_f) is then calculated from [18]:

$$\varepsilon_f = \frac{1.198 \times d}{(D-d)}. \quad (1)$$

The 2pb test does not require the special grips needed for conventional tensile tests, and the relatively small gauge length (0.3–0.9 mm) in the region of highest tensile stress minimizes the probability of extrinsic flaws [17]. A more detailed gauge length calculation can be found in [18]. The application and possibilities of the 2pb test are discussed in our previous publications [19–24]. For example, Lower et al. used 2pb to determine the inert intrinsic failure strains for sodium silicate glass fibers [22], sodium aluminosilicate glass fibers [23] and E-glass fibers [24]. A 2pb test has the advantage that it can be conveniently performed in liquid nitrogen. An important limitation of 2pb is that it measures failure strain, not failure stress. However, it should be pointed out that knowledge of the strain dependence of the modulus is valuable in understanding and modeling glass structure.

1.3. Nonlinear elastic modulus of glass

To convert the 2pb failure strains to failure stresses, the elastic modulus at failure must be known. For glass samples with failure strain of less than about 1%, failure stress ($\sigma_f(0)$) can be calculated from Hooke's Law:

$$\sigma_f(0) = E_0 \times \varepsilon_f \quad (2)$$

where E_0 is the zero-strain (or linear) Young's modulus. However, pristine glass fibers typically fail at significantly greater strains (5–25%), where the use of the zero-strain Young's modulus is no longer adequate. For example, the Young's modulus of 10 μm E-glass fibers tested in tension drops from 74 to 60 GPa for a strain of 4% [25]. The nonlinear Young's elastic modulus may be approximated by the polynomial [26]:

$$E = E_0 + E_1\varepsilon + \frac{1}{2}E_2\varepsilon^2 \quad (3)$$

where E_1 is the third-order non-linear Young's modulus, and E_2 is the fourth-order non-linear Young's modulus. These higher order elastic moduli can be measured using static techniques [27], ultrasonic techniques [28,29] or by Brillouin scattering [30,31]. Values of E_0 and E_1 for a variety of glasses have been reported, but very few values of E_2 for glasses are available in the literature, due to the difficulty of making these measurements at high strains. Values of E_0, E_1 and E_2 for fused silica have been reported from studies in ambient conditions [32]. There are other reported values of E_0 and E_1 for silica in ambient conditions [29] and in liquid nitrogen [33], but these latter studies did not report values for E_2. Values of E_0, E_1 and E_2 for E-glass have been obtained under ambient conditions [25]. There are also reported values of E_0 and E_1 for soda-lime silicate glass [31] and a nepheline sodium aluminosilicate glass [34]. Manghnani [35] measured the pressure dependence of elastic modulus for Na-silicate and K-silicate glasses in air, and values of E_0 and E_1 were obtained using a method discussed by Gupta and Kurkjian [6]. Using Eq. (3), a stress–strain relation can be described as [6]:

$$\sigma = E_0\varepsilon + \frac{1}{2}E_1\varepsilon^2 + \frac{1}{6}E_2\varepsilon^3. \quad (4)$$

Assuming that the temperature dependence of these moduli is negligible, Gupta and Kurkjian noted that when pristine glasses fail under intrinsic and inert conditions, indicated by an asterisk (*), the stress is maximum with regard to strain and so the differential of stress to strain, the effective Young's modulus, $d\sigma/d\varepsilon$, goes to zero. Using this condition, a value for E_2 can be derived at the intrinsic failure from Eq. (4):

$$E_2 = -2\frac{E_0 + E_1\varepsilon_f^*}{\varepsilon_f^{*2}}. \quad (5)$$

Substituting Eq. (5) into Eq. (4) provides an equation for intrinsic failure stress that can be used when E_0, E_1 and ε_f^* are known but E_2 is not known:

$$\sigma_f = E_0\varepsilon_f + \frac{1}{2}E_1\varepsilon_f{}^2 - \frac{1}{3}\frac{E_0 + E_1\varepsilon_f^*}{\varepsilon_f^{*2}}\varepsilon_f{}^3. \quad (6)$$

Under inert intrinsic conditions, when $\varepsilon_f = \varepsilon_f^*$, Eq. (6) is simplified to:

$$\sigma_f^* = \frac{2}{3}E_0\varepsilon_f^* + \frac{1}{6}E_1\varepsilon_f^{*2}. \quad (7)$$

In this paper, Eq. (6) is used to calculate failure stress under ambient conditions, and Eq. (7) is used to calculate the inert intrinsic failure stress under liquid nitrogen. Values of E_0 and E_1 used to calculate failure stresses are gathered from the literature.

2. Experimental procedures

2.1. Sample preparation

Materials used in this study include fused silica (AT&T, Amersil TO8 fused natural quartz), a commercial calcium aluminoborosilicate glass (PPG, E-glass), a commercial soda lime silicate glass (Owens-Illinois, flint container glass), a nepheline sodium aluminosilicate glass ($25Na_2O{\cdot}25Al_2O_3{\cdot}50SiO_2$, in mol%) from reference [23], a series of sodium silicate glasses, $xNa_2O{\cdot}(100\text{-}x)SiO_2$ ($10 \le x \le 35$), in mol%, described in references [19] and [22], and several potassium silicate glasses, $y\,K_2O{\cdot}(100\text{-}y)SiO_2$, $y = 15$–25, in mol%, also described in reference [19].

Commercial E-glass has a nominal composition in the ranges $(20–25)CaO{\cdot}(10–15)Al_2O_3{\cdot}(5–10)B_2O_3{\cdot}(50–55)SiO_2$, in wt%. E-glass marbles were remelted in platinum crucibles in air at 1550 °C for at least 4 h prior to fiber pulling. The melts were then transferred to a second furnace set at a fiber pulling temperature of 1300 °C. The second furnace was located below a custom fiber drawing system which pulled fiber from the surface of the melt. Fibers were drawn onto a rotating cage which was designed to prevent fiber overlap and damage. Fiber diameter was controlled by the fiber pulling temperature and the drawing speed. All fibers are drawn to a diameter in the range 125 ± 20 μm. After testing, each individual fiber diameter is measured at the broken ends to

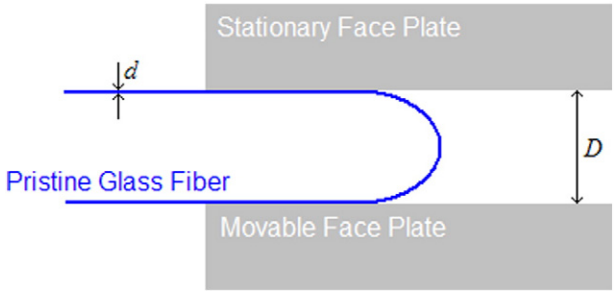

Fig. 1. Schematic diagram of the 2pb test.