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Feature article

Fracture toughness of glasses as measured by the SCF and SEPB methods

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ABSTRACT

The fracture toughness, K_{Ic}, of six glasses was measured by the surface crack in flexure (SCF) and single-edged precracked beam (SEPB) methods. Results depended upon the loading rate as well as the test environment. Environmentally-assisted slow crack growth affects the results for tests done in air. Dry nitrogen testing is preferred. Crack healing may be a severe complicating factor with precracked flexure bar type specimens if the specimens are unloaded between the precracking and final fracture test. Success in K_{Ic} testing depends to a large degree on upon the ability to make good precracks.

1. Introduction

In the 1970s–1980s, considerable work was done on measuring the fracture toughness and *K-V* curves (stress intensity–crack velocity) as shown in Fig. 1 for many glasses. Most of this work was done with flat slab-shaped specimens (e.g., glass microscope slides) using methods such as double cantilever beam (DCB)

or double torsion (DT). At the same time, fracture toughness of advanced ceramics was being measured with a variety of methods, many of which were based on flexure bar type specimens. Several of the latter methods were standardized in the 1990s–2000s by CEN [1], ISO [2–5], ASTM [6], and JIS [7]. The flexure bar methods should in principle be suitable for glasses, but there are some important nuances and problems. Some of these problems were identified our preliminary report on this work in early 2016 which focused on the surface crack in flexure (SCF) method [8].

This paper presents new SCF data and single-edge precracked beam (SEPB) results on six glasses. Problems in obtaining accurate and reliable SCF results that were identified in the preliminary work [8] have been solved. Questions that have lingered for many years are addressed. Just what is the fracture toughness, K_{Ic}, of glass? Can consistent results be obtained from different methods? How do the results from flexure bars specimens compare to those from larger traditional configurations such as DCB or DT? Does testing have to be done in an inert environment? Does testing have to be done at a fast rate? Does crack healing complicate matters?

Fracture toughness is often defined as "the resistance to crack extension" or "the resistance to unstable crack extension." (It is not a condition of crack arrest such as occurs in Vickers indentation crack length methods.) Many of the standards cited above use definitions like this. George Irwin, the father of modern fracture mechanics, was quite clear about what fracture toughness is [9–11]:

> "the critical condition for the onset of rapid crack extension" or "the onset of rapid extension"

At this point, it is appropriate to discuss whether there is in fact a fracture toughness, $K_{Ic,}$ for a glass. Most glasses are susceptible to environmentally-assisted slow crack extension (Regions I and II in Fig. 1) over a range of K_I values. Stable crack extension can even occur in inert or vacuum environments (Region III). Unless carefully done, fracture toughness experiments may simply measure a point on a K-V curve. The usual remedy is to test specimens in an inert environment, or as fast as possible to minimize the interference of slow crack growth. Even so, with practical specimen sizes, one

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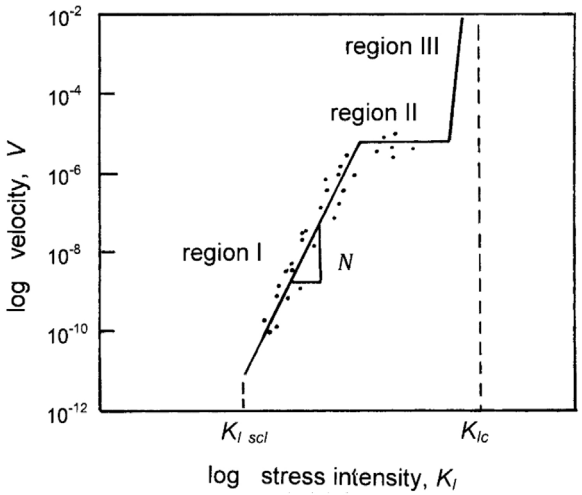

Fig. 1. Schematic of a *K-V* curve for glasses.

may merely be measuring some point in region III. Some authors show versions of Fig. 1 with "K_{Ic}" off to the right, and some show it converging with or intersecting the region III line. There is no consistency in the literature about this. It is not surprising therefore, that some people argue that there really is no set, specific value for K_{Ic} for glasses. They argue that the measured K_{Ic} depends upon the test specimen type and the rate of loading and the environments. This is a pessimistic perspective, however. Rather than dwell on the complications, perhaps it is wisest to remember what Wiederhorn wrote forty-two years ago [12]:

> "Of the glasses studied, the fused silica and low-alkali borosilicate glasses exhibited no subcritical crack growth before failure {in vacuum}. Instead, fracture was abrupt, occurring at a critical value of the stress intensity factor."

So there are some glasses with finite, specific values of fracture toughness. Although most glasses are susceptible to slow crack growth, the basic concept is sound. Wiederhorn even suggested a definition of fracture toughness in a footnote in that paper [12]:

> "The critical stress intensity factor, K_{Ic}, is commonly defined as the value of the stress intensity, K_I, required for crack growth in an inert environment. K_{Ic} is a well-defined quantity for materials that fail abruptly because the crack accelerates rapidly at a well-defined value of K_I. Materials that exhibit slow crack growth (referred to as subcritical crack growth herein) do not easily fit this definition because the lower limit of K_I for the initiation of crack growth is not well defined. For these materials, K_{Ic} is defined herein as the value of K_I (measured experimentally) required for cracks to move at velocities $\geq 10^{-1}$ m/s. Using this definition, values of K_{Ic} were reported. . . . for glasses that exhibit subcritical crack growth in vacuum."

Wiederhorn chose a velocity of 10^{-1} m/s since it was a practical value. It was the fastest crack that he could monitor when he watched cracks propagating in his DCB specimens. The exact velocity is not important since many glasses have very steep region III trends.

In the present study, fracture toughness experiments were done in an inert environment and at a variety of loading rates and the resistance to unstable crack propagation was measured. Matching experiments in laboratory ambient conditions were done for comparison. There are three key ingredients to a good fracture toughness test: (1) a good crack, (2) a good fracture, and (3) a good stress intensity shape factor analysis. All three were important in this study. Many illustrations of good and bad precracks are included in this paper since this work was presented at a conference on fractography. The illustrations will aid others who will likely encounter similar complications and oddities.

2. Materials

Table 1 lists the six glasses evaluated.[1] The two borofloat grades and the two soda lime silica grades are used in transparent armor applications. The borosilicate crown glass has a different composition than the other two borosilicates and is used in many optical window applications. It has a much higher alkali content. It has been used in many mechanical property studies in the past. Fused silica was included since it is an interesting contrast to the other five glasses. Several of these glasses were evaluated in the late 1960s and 1970s and reported in the seminal works of Wiederhorn et al. on fracture toughness of glasses [12,13]. Wiederhorn et al. used DCB and carefully-precracked three-point flexure specimens in a variety of environments including lab air, dry nitrogen, and vacuum.

3. Methods

Flexure bar methods were used since flexure bars are convenient to make from thick glass slabs. The surface crack in flexure (SCF) and single-edged precracked beam methods (SEPB) were used. The chevron notched beam (CNB) method was not used since slow loading rates must be used to obtain stable crack extension and there was concern that environmentally-assisted slow crack growth might affect the outcomes. Single-edged V-notched beam was not used since the notches are not sharp in glasses.

Flexure specimens, nominally 3 mm × 4 mm × 50 mm, were cut from large glass plates in accordance with slicing and grinding procedures in the ceramic standards [2,3,6]. A fine longitudinal finish was given to each bar. Polishing was not necessary. The four long edges were beveled to 45°, but unfortunately, many were oversized at between 0.15 mm to 0.22 mm. The broken halves of these full-length specimens were long enough that they could be used for additional experiments.

In the SCF method, a Knoop indenter created a small semi-elliptical precrack or "controlled flaw" in a flexure bar specimen. The indentation and damage zone were removed by polishing. The bar was then broken in a flexure strength fixture. Indentations were made with a conventional hardness machine with a force 24.5 N (2.5 kgf). This force was large enough to make good sized precracks, but not so large as to create extreme lateral cracks or semielliptical cracks that required excessive polishing to remove the initial damage zone. Earlier work on the indentation size effect (ISE) of Knoop hardness in glasses showed that 24.5 N was large enough to create obvious cracking for a range of glasses [14]. The glass specimens were not tilted slightly during this step, unlike the procedure used for ceramics. After indentation, the specimens were put in a desiccator. The latter was used between all steps up until fracture. The specimens then were hand ground with 400 grit SiC papers to remove the indentation, its residual stress damage zone, and all traces of lateral cracks. Amounts removed ranged from 8 to 10 times the Knoop indentation depth. Knoop diagonal lengths ranged from 270 μm to 310 μm, and since the indentation depth is 1/30th of the diagonal length, 80 μm to 120 μm was ground off. The hand grinding was done with two specimens at a time mounted on an aluminum block for easy handling as shown in Fig. 2. Grinding was done with a small force applied by hand and done dry to eliminate

[1] Commercial products and equipment are identified only to specify adequately the experimental procedures and does not imply endorsement by the authors, institutions, or organizations supporting this work, nor does it imply that they are necessarily the best for the purpose.

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