

## Some aspects of strength characteristics of glass<sup>1)</sup>

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Strength characteristics of glasses are reviewed with some new aspects, emphasizing their specific features different from those of polycrystalline ceramic materials. The absence of any strength-controlling, structurally inherent defects in glass discriminates between the pristine strength on the GPa order and the practical strength controlled by the presence of surface flaws externally induced. For investigating the nature of the pristine strength, hydrofluoric-acid etching of glass samples having a definite indentation is shown to be quite useful to get consistent experimental results. Some experimental examples clarify the degradation mechanism in glass strength when heated or coated with thin films as well as the fatigue nature of the pristine strength. In evaluating the practical strength of glass, the strength-degrading effect of blisters and strengthening by providing surface compressive stress are discussed. A strength scale of glass and glass ceramics is given.

### Festigkeitskriterien bei Gläsern

Die Festigkeitskriterien bei Gläsern werden an Hand einiger neuer Gesichtspunkte unter Berücksichtigung besonderer Merkmale bewertet, die von denen polykristalliner keramischer Materialien abweichen. Das vollständige Fehlen von festigkeitsbeeinflussenden natürlichen Strukturfehlern in Glas führt zu einer Unterscheidung zwischen der Ausgangsfestigkeit in der Größenordnung von GPa und der praktischen Festigkeit, die durch nachträglich verursachte Oberflächenfehler bestimmt wird. Zur Untersuchung der Ausgangsfestigkeit mit dem Ziel, übereinstimmende Versuchsergebnisse zu erhalten, hat sich die Methode bewährt, Glasproben mit definierten Oberflächenfehlern zunächst in Flußsäure zu ätzen. Einige experimentelle Beispiele erklären sowohl die Ursache für die Abnahme der Glasfestigkeit, wenn das Glas erwärmt oder mit dünnen Schichten versehen wird, als auch die Verringerung der Ausgangsfestigkeit durch Ermüdung. Zur Beurteilung der praktischen Festigkeit von Glas werden der festigkeitsmindernde Einfluß von Blasen und die Festigkeitssteigerung durch Erzeugung einer Oberflächendruckspannung diskutiert. Eine Festigkeitstabelle von Glas und Glaskeramik wird aufgestellt.

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### 1. Specific features of glass strength

Both glasses and ceramics are known to be representative brittle materials, which tend to fail catastrophically by growth of a single crack that originates from a very small defect without showing any ductility in contrast with metallic materials. There is, however, a marked difference in strength characteristics between glasses and ceramics; glass is an amorphous material, isotropic, homogeneous and free of structural strength-controlling defects, such as dislocations or grain boundaries, whereas in polycrystalline ceramic materials the complicated microstructure is an important factor in characterizing their strengths. The absence of these defects gives glass distinctive strength characteristics, which were experimentally well ascertained by the 1960's [1 to 3]. The essential features, which are still commonly accepted, are summarized as:

a) Strength-controlling defects in glass are not structurally intrinsic but are surface flaws externally induced by mechanical damage.

b) The intrinsic strengths of glasses with damage-free surfaces are on the order of GPa and show a very low scatter when carefully controlled specimens are used in measurements.

c) The intrinsic strength still shows fatigue and temperature dependence.

The first consistent strength measurements on glass specimens with damage-free surfaces were reported by Thomas in 1960 [4]. He observed a strength of 3.8 GPa for E-glass fibers independent of diameters with a coefficient of variation of only about 1%. Hillig [5] reported that fused silica rods 0.4 to 0.9 mm in diameters showed strengths as high as 14 GPa at liquid nitrogen temperature. It became accepted that there is no inherent size effect on the strength of damage-free glass samples and the notion of pristine or intrinsic strength was introduced. It must be noted that the concept is only applicable to glass among all solid materials; the intrinsic strength is presumed to be a property of the matter as elastic modulus or expansivity.

Today, the commercial application of E- and S-glass fibers to reinforce plastics as well as the use of silica fibers in optical communication has enriched the information about the nature of the pristine strength of glasses [6 and 7]. The high pristine

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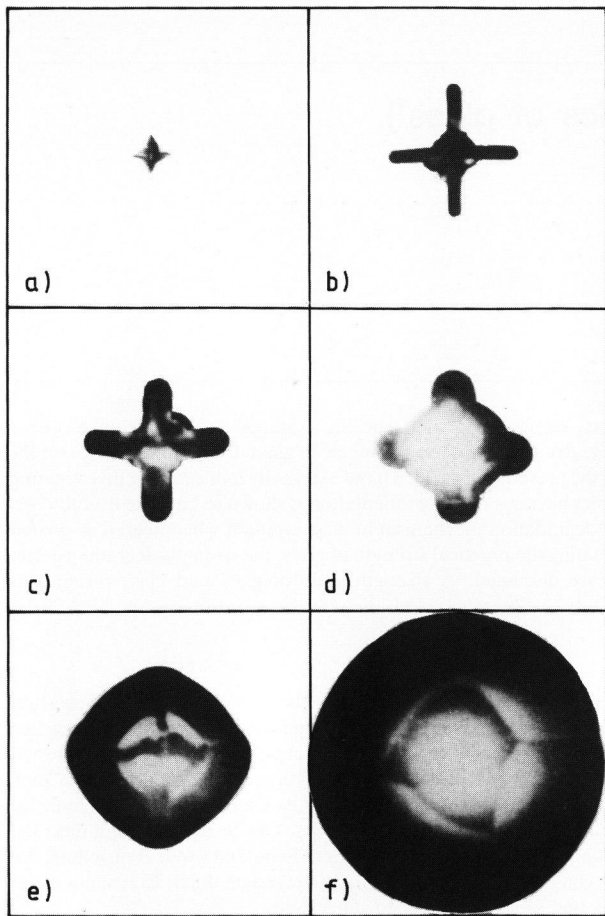
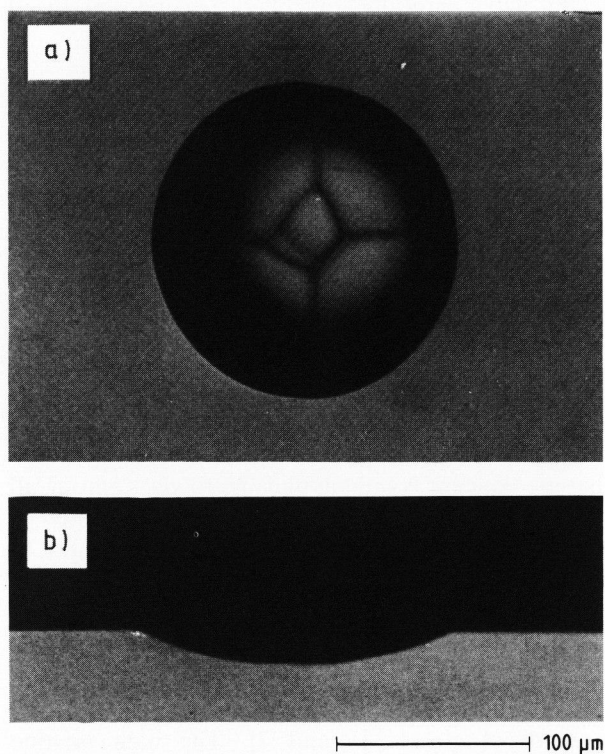


Figure 1. Development of indentation shape with the progress of etching after Hara [13]. Depths removed from surface are: a) 0  $\mu\text{m}$ , b) 4  $\mu\text{m}$ , c) 8  $\mu\text{m}$ , d) 12  $\mu\text{m}$ , e) 32  $\mu\text{m}$ , f) 122  $\mu\text{m}$ .



◀ Figures 2a and b. Different views of an etch pit developed from an initial semicircular crack system, a) dish-like etch pit, b) cross-sectional view after Ishikawa et al. [17].

strengths are practically utilized by drawing fibers in a perfectly dust-free atmosphere and by immediate protection of surfaces with plastic coatings before any mechanical contact. The remarkably homogeneous nature of pristine silica glass surfaces is emphasized recently by Kurkjian and Paek [8], who reported that the apparent variability in strength of silica fibers calculated assuming a constant diameter is the result of the variability in diameter and therefore, the strength is essentially single-valued.

The theoretical strength of perfectly flawless silica glass calculated from the cohesive force of the silicon–oxygen bond is about 24 GPa [9 and 10]. The introduction of alkali and/or alkaline earth oxides, which break the silicon–oxygen bond, may lead to a significant decrease in this ultimate strength limit. For multicomponent glasses, fluctuation of composition [2] or grouping of alkali ions [11] is thought to be a possible source of weakness. However, when one considers observed pristine strengths fairly close to the theoretical limits, the effect of those structural defects on strength is rather limited.

## 2. Acid etching for investigating intrinsic strength

Preparing damage-free, fire-polished surfaces of glass samples is not so easy, because any tiny dust particles bonded to the surface of glass samples during hot processing (e.g., fiber drawing) become severely strength-degrading sources; the existence of such ill-defined flaws raises a strength variation and hinders a quantitative analysis. An alternative method of raising the strength of glass to a level of the intrinsic strength is hydrofluoric-acid etching [12]. In order to avoid ambiguity and scattering of measured strength values, a technique was developed, where well-defined flaws were provided by using a standard Vickers diamond pyramid indenter at the center of specimen surfaces prior to hydrofluoric-acid etching [13]. A sequence of photographs shows the change in the appearance of the indentation on sheet glass samples with the progress of the etching (figures 1a to f); a dish-like etch pit, as shown in figures 2a and b, was developed from an initial semicircular crack system.

The fracture of the etched specimens in static bending always originated from the place of the indentation even after etching. The severest flaw at the beginning continued to be the severest throughout the etching procedure; no new severer flaws were produced during the etching. (Roach and Cooper [14] reported on the weakening of soda-lime glass by particle impact during hydrofluoric-acid etching.

Such a weakening rarely happened in the present case presumably because of a lower acid concentration.) The strength increased rapidly in the early stage and somewhat slowly in the later stage with increasing depths of the surface layer removed, corresponding to the decrease in the stress concentration factor of the etch pit, and it finally exceeded 2 GPa (figure 3). The variation of the strength values was very small even after etching and indeed, on the order of the accuracy of thickness and width measurements of specimens.

The high consistent strengths thus obtained were unchanged during storage in air at room temperature for several weeks, when any mechanical damage carefully avoided, but showed a further increase after immersion in water for 2 d or longer, which amounted to about 10 %, presumably because of desorption of fluorine ions at the surface [15]. The intrinsic strength of sheet glass can be evaluated from an approximate stress concentration factor of the etch pit, which fell to about 1.5 after prolonged etching. On the basis of the strength values measured after aging in water, the pristine strength of sheet glass was estimated to be 3.4 GPa [15]. The difference of structure between fire-polished and etched glass surfaces is not entirely clear, but this value estimated for sheet glass at room temperature is almost equal to the strength of interior surfaces determined by the oblate-bubble technique for a similar soda-lime glass by Ernsberger [16].

This technique was successfully utilized for investigating the strength deterioration of sheet glass due to metal coating [17]. The thin metal film coating on the acid-etched glass surface caused an appreciable loss of strength, but the fracture was still initiated from the bottom of the dish-like etch pit. The photograph (figure 4) shows a fracture surface of 69 nm thick chromium-coated glass that failed in bending. The fracture mirror is observed just beneath the bottom of the etch pit, showing that newly formed defects are quite dense and homogeneous.

Heating acid-etched glasses to about 500 °C led also to a large drop in strength and to an increase in variation of test results [15]. In this case, the fracture did not start at the etch pit, suggesting that some random defects newly formed become a fracture origin. Re-etching the heat-treated samples led to a rapid return to the previous reproducible high strength. Removal of only a few micrometers from the surface was sufficient to recover the full strength and to cause fracture again at the place of the indentation. Microscopic dirt particles bonded to the surface are supposed to be the origin of fracture as Brearley and Holloway suggested in 1963 [18].

The fatigue property of glass having surface cracks has well been understood by the fracture mechanic approach, which is based on the underlying assumption that fatigue failure occurs by subcritical

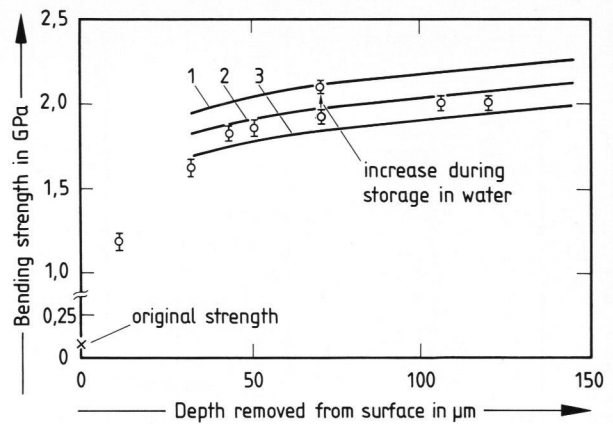


Figure 3. Strength versus depth of surface layer removed after Hara [15] (mean strength and 95 % confidence limit). Theoretical curves under the assumption that the intrinsic strength  $F_0$  is 3.334 GPa (curve 1), 3.138 GPa (curve 2), 2.942 GPa (curve 3).

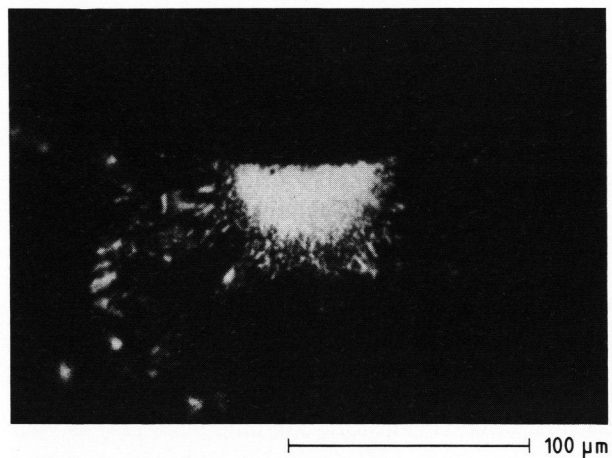


Figure 4. Fracture surface of a 69 nm thick chromium-coated glass failed in bending after Ishikawa et al. [17].

slow extension of a dominant preexisting crack to an instability configuration [19 and 20]. The fatigue parameter  $n$  has been determined for various glass composition/environment systems by one of three experimental procedures: from static fatigue, dynamic fatigue or direct crack growth [21]. For a soda-lime glass the crack velocity measurement conducted by Richter [22] gave  $n = 16.0$  in water,  $n = 18.1$  in air with about 50 % relative humidity. Systematic discrepancy between crack velocity/dynamic fatigue determination of  $n$  was explained by Marshall and Lawn [23 and 24] with particular reference to the role of residual contact stresses around flaws. Their dynamic fatigue tests conducted on Vickers-indented soda-lime glass in water showed  $n = 13.7 \pm 0.2$  for as-indented surfaces,  $n = 17.9 \pm 0.5$  for indented and annealed surfaces; the latter was expected to correspond to the value determined by the crack velocity measurement.

In contrast with that, the fatigue behavior of pristine strength, now important in strong fiber applications, is far from being understood. The large

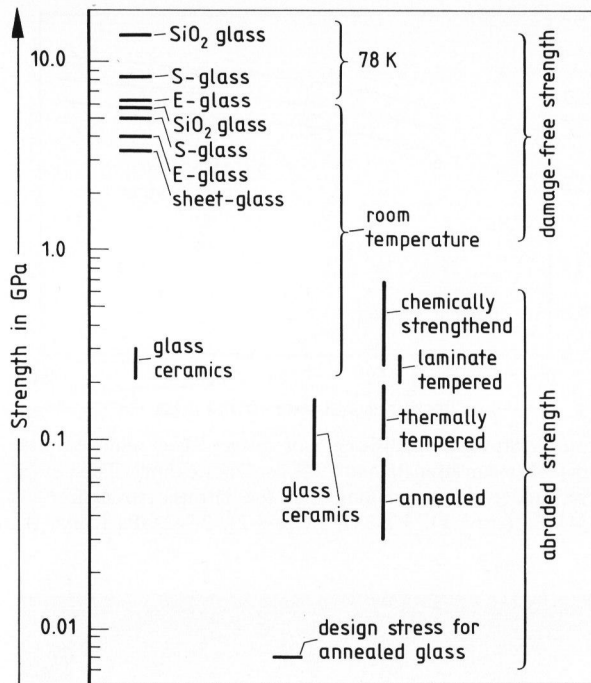


Figure 5. Strength scale of glass and glass ceramics.

experimental scatter stemming from difficulties in preparing homogeneous pristine samples prevents an accurate fatigue study. Dabbs and Lawn [25 to 27] studied fatigue properties in the high-strength region using subthreshold indentation flaws (flaws without radial cracks). They found a higher fatigue susceptibility in the subthreshold region than in the postthreshold region for soda-lime glass [25] as well as for silica glass [27].

A dynamic fatigue test on acid-etched specimens with a definite dish-like etch pit was conducted in air with 43 % relative humidity at 24 °C [17]. The results yielded a fatigue parameter  $n$  of 24.6, being higher than that observed in samples having sharp flaws under the same conditions, indicating that pristine glass is less susceptible to fatigue. Ritter and Sherburne [28] found also the same tendency for an acid-etched soda-lime glass, though their strength values somewhat lower and the variation of test results was not clear. Further tests with this method will afford a deeper insight into the fatigue mechanism of pristine strength.

### 3. Practical strength of glass and glass ceramics

A strength scale of glass and glass ceramics is shown in figure 5. The practical strength of glass is limited by the almost inevitable presence of surface flaws, which arise during usage as well as during fabrication, and it distributes in a wide range due to a variation of flaw sizes. As these flaws extend slowly under tensile stress particularly in a water-containing atmosphere, the design strength of glass articles under sustained

loading is assumed to be as low as about 7 MPa [29]. It must be noted that this value is also taken as an allowable limit of residual tensile stress in annealing or in glass-to-metal sealing procedures.

Glass ceramics are dense fine-grained aggregates produced by controlled nucleation and devitrification of glasses. Their practical strength is higher than that of glasses, because they have higher fracture toughness and are more resistant to mechanical damage. The damage-free strength of glass ceramics, which can be estimated by the strength versus indentation load relationship [30], is, however, far less than that of glasses as a result of their inhomogeneous structure (figure 5). Fractures originating from the inside are often observed in the case of glass ceramics as reported by Lewis III and Spann [31].

In polycrystalline ceramics, voids are regarded as an important strength-degrading source [32], but blisters in glass have generally no influence on practical strength because their stress concentration factor is only about 2. However, when they are located close to or broken at the external surface, their stress concentration factor becomes enormously large and comparable to that of sharp cracks. The stress concentration factors are given graphically in figures 6a and b as a function of bubble location [33]. Those near-surface or broken blisters are frequently generated during hot forming (e.g., glass-to-glass sealing) and are dangerous fracture sources of glass articles. In this case, the fracture originated from the point on the blister surface nearest to the external surface (point B in figure 6a) or from the edge of the broken blister (point C in figure 6b). A fracture step appears at the opposite site of the blister (point A in figures 6a and 6b).

### 4. Strengthening of glass

Since glass has no strength-controlling defects structurally, glass never fails from the inside except in the cases where crystalline inclusions exist in the glass matrix. (Blauel and Richter [34] reported that fracture of glass rods started from the inside by thermally induced stress. Dust particles lie possibly along the central axis of the rods, because during downward rod-drawing procedures the central part is exposed to open air.) The inclusions are never of a structural origin but arise from insufficient melting or from devitrification [35]. The present-day melting technology can well reduce the amount of the defects to under 1 to 2 grains per cubic meter [36]. In this respect, providing surface compressive stress is especially useful in strengthening glass articles, possibly to a level of the intrinsic strength. The applied stress must overcome this compressive stress for surface flaws to extend, so that minimum strength is raised by the amount of the surface stress; e.g., a compressive stress of 100 MPa can raise a design

Figures 6a and b. Stress concentration factors  $\alpha_A$ ,  $\alpha_B$  and  $\alpha_C$  of point A, B and C, respectively, ( $\nu$ : Poisson's ratio) of a) near-surface blisters, b) broken blisters after Nishida and Kim [33].

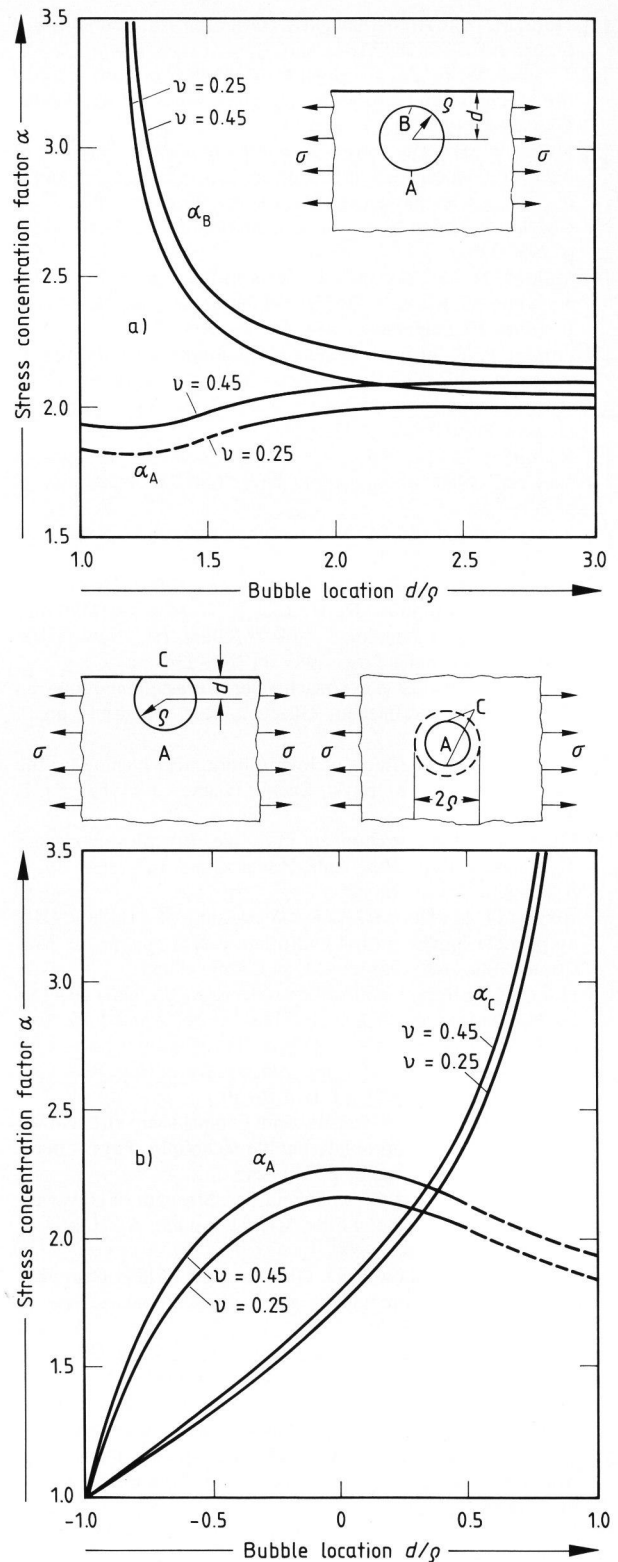
strength of 7 MPa about fifteenfold. There are some trials to strengthen polycrystalline ceramics by the same method [37], but the danger of failure from the inside may limit the effectiveness of strengthening.

It was often reported that the surface compressive stress was also effective in reducing contact damage so that the strength increase by strengthening was higher than expected from the value of compressive stress [38]. An experiment conducted on tempered and annealed sheet glass indented by Vickers diamond showed, however, that the increase in strength was just as large as the compressive stress, though the depth of the cracks was far smaller in tempered glass than in annealed glass [39]. This result agrees with a theoretical expectation based on indentation fracture mechanics, where the residual crack-opening force is assumed to act steadily on a fracture-initiating crack [40]. The discrepancy in experimental results can presumably be attributed to the difference in the aging of fracture-originating cracks, that is still a major concern in fracture studies of glasses [41 and 42].

There is experimental evidence that elastic modulus [43] or hardness [44] of tempered glass is somewhat lower than that of annealed glass because of an open structure of rapidly cooled glass. It is at present not clear how fracture toughness changes with rapid cooling. In an old experiment on sheet glass cutting [45], a correlation between crack depth beneath cut lines and density of glass samples was found to suggest that fracture toughness becomes higher when glass is more rapidly cooled. A recent study by Toninato et al. [46], however, showed only higher threshold for crack nucleation but no variation in crack depth in quenched glasses. More systematic studies are needed to clarify the effect of structure change by rapid cooling on fracture toughness as well as on intrinsic strength, which are important factors in characterizing the strength of flawless glasses.

## 5. Conclusion

It has been pointed out that the amorphous structure gives glass excellent strength properties which can not be found in polycrystalline ceramic materials, such as high intrinsic strength and the possibility of strengthening to the level of the intrinsic strength. Minimizing strength-controlling defects externally induced, such as surface flaws, crystalline inclusions or near-surface blisters, in fabrication procedures as well as developing new methods for providing surface compressive stress will afford a wider application of glasses as a strong material.



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