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Progress Report

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MECHANISM OF MECHANICAL FATIGUE OF SILICA GLASS

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Research results obtained in the current program

Significant Accomplishments

1). Existence of a blunt crack tip.

Strength increase of abraded glasses by thermal, chemical, and hydrothermal treatments was found best explained by crack tip blunting.

2). Mechanical behavior of glasses with blunt cracks.

Fatigue of silica glass with blunt crack tips was found to involve crack initiation in addition to crack propagation, and only water and ammonia were found capable of causing crack initiation from blunt crack tips at sub-critical stresses.

Furthermore, apparent fracture toughness was much higher for silica glasses with blunt cracks than those with sharp cracks. Correspondingly, the mirror constant, which is normally considered to be a material's constant, was found to increase with the estimated crack tip radius.

3). Water entry into glass under stress.

Water was found to enter into silica glass during microhardness indentation as well as during slow crack growth in water-containing atmospheres at room temperature. The effect of applied stresses on water diffusion into silica glasses were re-examined. At low temperatures, tensile stress was found to accelerate the diffusion coefficient.

4). Water diffusion into SiO₂ glass at low temperature.

Water diffusion into silica glass at low temperature showed anomalous features including initial time dependence of solubility and diffusion coefficient. It was found that these anomalous features are related to the time dependent relaxation of glass structure. This was shown by measuring the 1873 cm⁻¹ peak shift, which is an indirect measure of the Si-O-Si bond angle change. The time dependent anomalous diffusion behavior was observed when rate of the structural relaxation lags behind the rate of water diffusion.

5). Mechanical properties of glasses with high water concentration.

Glass containing high concentrations of water showed greater fatigue susceptibility and lower crack initiation stress than dry glass.

6). Cyclic fatigue.

Fatigue of abraded silica glass showed effects of cyclic stress when tested by the rotation bending method while no cyclic stress effect was observed in reciprocal bending.

7). Fatigue resistance improvement by coating.

A SiO coating on abraded silica glass was found to reduce mechanical fatigue.

8). IR peak assignments for the silica-water system.

IR peaks of SiO₂ glasses with H₂O were investigated and assigned. Also, molecular water was found to be present in silica glass after low temperature hydration experiments.

9). Water diffusion and structural changes in silica glasses.

At high temperatures, rates of both structural relaxation of a silica glass and oxygen vacancy disappearance of oxygen-deficient silica glasses exposed to water vapor were faster than the rate of water diffusion itself. This indicates that a minor fraction of water is diffusing faster, unrestricted by the chemical equilibrium, than the majority portion which is.

10). Water diffusion and time dependent mechanical properties.

Water diffusion behaviors of silica glasses at 350°C ~ 650°C and indentation creep at room temperature were found to be correlated, with the glasses having a faster diffusion rate exhibiting the greater indentation creep rate. Dynamic fatigue susceptibility also followed a similar trend.

11). Effect of nitrogen and carbon implantation on devitrification of silica glasses.

Crystal growth rate was decreased when nitrogen was implanted in oxygen deficient silica glasses

but not in stoichiometric silica glass. It was suggested that the oxynitride bonds which formed in the surface layer reduced the water diffusion and diffusion-controlled crystal growth rate.

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20. H. Li, M. Tomozawa and V. Lou "Effect of Nitrogen and Carbon Implantation on

Devitrification of Silica Glasses" to appear in J. Non-Cryst. Solids.

21. H. Li and M. Tomozawa, "Mechanical Strength Increase of Abraded Silica Glass by High Pressure Water Vapor Treatment", to appear in J. Non-Cryst. Solids.

22. M. Tomozawa, H. Li and K.M. Davis, "Water Diffusion, Oxygen Vacancy Annihilation, and Structural Relaxation in Silica Glasses" to appear in the Proceedings (J. Non-Cryst. Solids) of the Robert Weeks Symposium on Defects in SiO_2 and GeO_2 .

23. K.M. Davis and M. Tomozawa, "Water Diffusion into Silica Glass." to appear in the Proceedings of the Conference on Diffusion in Amorphous Materials.

24. M. Tomozawa, K.M. Davis, and H. Li, "Water Diffusion and Time-dependent Mechanical Properties of SiO_2 Glasses," Submitted to J. Non-Crystalline Solids.

25. K.M. Davis and M. Tomozawa, "IR Studies of Molecular Water in Silica Glasses" submitted to J. Non-Cryst. Solids.

26. K. M. Davis and M. Tomozawa, "The Diffusion of Water into Silica Glass: The Effects of Structural Relaxation on Water solubility and Diffusivity." submitted to J. Non-Cryst. Solids.

Discussion of obtained results

1). Existence of a blunt crack tip.

The issue of blunt crack tips was examined first. It is well recognized that the mechanical strength of freshly abraded glass increases upon heat-treatment or hot water-treatment. Two explanations were offered for this phenomenon; the reduction of the stress concentration due to crack tip blunting¹ and the release of the residual tensile stress^{2,3} which existed near the crack tip. It was shown^{4,5} that the strength increase upon heat-treatment at the annealing temperature can not be attributed to residual stress release but can best be explained by crack tip blunting caused by the viscous flow of the glass surface facilitated by water diffused into the glass from the atmosphere. One of the evidences for this conclusion was that annealing in vacuum of abraded glasses did not produce appreciable strengthening even though residual stress can be released by this procedure. It was shown that the strength increase caused by hot water treatment was due to crack tip blunting caused by the dissolution and reprecipitation of glass at the crack tip^{6,7}. This process of strengthening by hot water treatment was extended to higher temperatures (in 200°C water and in 250°C saturated water vapor) using a hydrothermal unit. As much as 200% strengthening was achieved by this process while no change in crack length took place. It is not possible to obtain this much strengthening of abraded and aged samples by residual stress release. Glass samples with sharp macro-cracks also showed similar strengthening even though there can be no residual stress in this case. These observations can be explained by crack tip blunting rather than by the release of residual stress.

An alternative mechanism of strengthening by hot water treatment was suggested for soda-lime glasses. Namely a glass-water reaction product precipitates behind a crack forming a bridge but leaving the original crack tip intact⁸. This mechanism is similar to dissolution and precipitation suggested for silica glass crack tip blunting. The only differences are the location of the precipitate, behind the crack tip vs. at the crack tip, and the precipitate composition. Near the crack tip silica glass is expected to have the lowest solubility in water because of its curvature and precipitation is expected to take place most readily there. Furthermore, the precipitate composition in silica glass is expected to be nearly pure silica as was discussed earlier. In contrast, the composition of precipitate in the crack of soda-lime glass is expected to be different from that of the glass. When a precipitate with the same composition as the glass forms at the crack tip the distinction between crack tip blunting and crack bridging disappears. Also, there are people who prefer to define a crack as having a sharp tip in distinction to all others as flaws. If this definition is followed, then one should say that a crack is turned into a flaw by thermal and hydrothermal treatments. In either case, the important point is that thermal and hot water treatments can make the abraded silica glasses stronger without shortening the length of crack.

2). Mechanical behavior of glasses with flaws or blunt cracks.

Both dynamic fatigue⁹, fatigue under steadily increasing stress, and static fatigue^{5,10,11}, fatigue under a constant stress, of high silica glass and silica glass were measured for freshly abraded glass samples with sharp crack tips and treated glass samples with blunt crack tips in various environments. It was found that the fatigue behavior of the freshly abraded glasses can be explained by the slow crack growth mechanism while that of the treated glasses with blunt cracks requires the assumption that crack initiation plays an important role. In addition, experiments showed that only water and ammonia can cause crack initiation at subcritical stress. Equivalently, the glasses with blunt cracks showed static fatigue in water and in ammonia but not in other chemicals. The mechanical fatigue behavior of these glass samples with blunt cracks is also expected to be closer to that of pristine silica optical fibers than that of freshly abraded silica glasses.

Increases in both fracture toughness and mirror constant were also observed^{5,12} for the hydrothermally treated samples, even though these quantities are considered to be constants for a given material. Fracture toughness is usually obtained by multiplying the fracture strength of a sample and square root of the crack length. Since hydrothermally treated glass samples with blunt crack tips have a higher mechanical strength with unchanged crack length, their fracture toughness was higher than freshly abraded silica glass samples. On a fracture surface of a glass, the crack origin is surrounded by a smooth zone called the mirror region, which in turn bounded by rougher regions called mist and hackle. The size of mirror is a function of fracture strength and the product of the fracture strength and the mirror radius is known as the mirror constant, believed to be a unique constant for a given glass composition. For hydrothermally treated silica glasses, the mirror constant increased with increasing extent of crack tip blunting. Apparently, in order to obtain a constant fracture toughness and mirror constant, the crack tip has to be sharp.

3). Water entry into glass under stress.

In order to understand the crack initiation process in glass, the behavior of glass under stress before a crack initiates was investigated using a microhardness indenter^{13,14}. It was found that the crack initiation behavior correlates well with the hardness of the glass. In addition, both crack initiation load and hardness were time dependent in water, while both quantities were time independent in liquids such as formamide and hydrazine. Thus, crack initiation at a sub-critical stress appears to be related to the time dependent hardness. It was found that the time dependent hardness observed in air and water was caused by the entry of water into glass under stress^{13,14}. In related research¹⁵ on defects in silica glass, it was found that point defects are produced below the surface of silica glass powder produced in a water-free environment, while no defects were detected in powder produced in a water-containing atmosphere. This difference appears to originate from the water entry into silica glass during fracture in water-containing atmospheres. Extending these works, it was found¹⁶ that water enters into silica glass during slow crack growth in water-containing atmospheres. This was demonstrated by measuring the hydrogen depth profile of a fractured surface by a nuclear reaction technique. Hydrogen (from water) was observed to exist both on the glass surface as an adsorption layer and also within a few hundred angstrom thick surface layer when the fracture surface was produced by slow crack growth in water-containing atmospheres.

4). Water Diffusion into SiO₂ glass at low temperature.

In order to establish the reference value for water entry into silica glass under stress, a diffusion study of water into SiO₂ glass was extended to lower temperatures^{17,18} than most published works using an FTIR. Above ~850°C, many different silica glasses exhibited almost identical diffusion behavior, while below 850°C, clear difference was observed for different silica glasses. In addition, several anomalous phenomena were found at temperatures lower than ~850°C such as the surface concentration increasing and apparent diffusion coefficient decreasing with time. The infra-red absorption spectra of water in glass were also different; the absorption produced at lower temperatures exhibited additional lower wave number peaks indicating the presence of hydrogen bonded hydroxyl and molecular water. In addition, water entry into silica glass promotes local structural relaxation as indicated by a shift of the IR peak at 1873 cm⁻¹ due to Si-O-Si vibrations. Different water diffusion behavior above and below ~850°C appears to be related to the relative rate of water diffusion and structural relaxation: Above ~850°C, the rate of this structural relaxation is faster than the rate of water diffusion and water is diffusing in a relaxed structure, while below ~850°C, the rate of this structural relaxation was slower than water entry and water is diffusing in a relaxing structure. The observed time dependent phenomena at lower temperatures were attributed to the slow rate of relaxation which affects kinetics of reaction between water in glass

and the glass network as well as solubility.

5). Mechanical properties of glasses with high water concentrations.

In order to learn the role of water which entered into the glass under stress, mechanical properties of glasses containing water were investigated. Earlier, it was shown that fatigue susceptibility increased with increasing water content for sodium silicate glasses¹⁹. Further, microhardness indentation of sodium silicate glasses with various water contents was investigated²⁰. It was found that glasses containing water showed indentation creep even in toluene, while dry glasses did not. This is consistent with the earlier finding that the indentation creep of dry glasses in water-containing atmospheres is due to water entry into the glass under stress. It is possible that the time dependent reaction between molecular water in the glass and the glass network under stress is causing the time dependent hardness observed in toluene. Under high indentation load, all glasses including dry glasses showed time dependent hardness in toluene²¹. This time dependent hardness was attributed to inhomogeneous non-Newtonian flow. This is phenomenologically similar to plastic deformation. Water in glass appears to decrease the onset stress for the inhomogeneous non-Newtonian flow or yield stress. Silica glasses which were heat-treated at different temperatures after hydrothermal treatment showed different fatigue behavior²². This was attributed to different crack initiation stresses as a result of different amounts of water remaining in the glasses. These observations, together with work²³ by other investigators, show clearly that water in glass increases the fatigue susceptibility of glass by promoting crack initiation and crack growth.

6). Cyclic Fatigue.

Cyclic fatigue tests of abraded dry silica glass were conducted by two different methods; a reciprocal bending method and a rotational bending method. It was found that by the former method, cyclic fatigue tests did not show any detrimental effect compared with static fatigue tests, while by the latter method, the life time was much shorter compared with static fatigue. This finding is qualitatively similar to the preliminary work by Proctor²⁴. The shorter fatigue life by rotation bending was attributed to the widening, in addition to deepening of the flaw, due to the time dependent stressing condition during rotation and increased stress intensity at various locations of the crack tip. The absence of the normal (reciprocal bending) cyclic effect on crack growth is consistent with the earlier analysis by Evans²⁵.

7). Fatigue Resistance Improvement by Coating.

Our study on water diffusion into silica glass at low temperatures suggested an interesting possibility for reducing water diffusion into and mechanical fatigue of silica glass. At low temperatures, the rate of reaction between molecular water and silica glass appears to be slow, and molecular water can diffuse farther as it is trapped (converted to immobile hydroxyl) less frequently than in the case at high temperatures where the local reaction equilibrium is established instantaneously. If one could coat silica glass with a material which can trap molecular water more effectively than silica glass itself, this coating material would work as a water diffusion barrier. Coating materials for silica glass optical waveguides^{26,27} recently developed by industry, carbon and titania-silicate, appear to have this desired high trapping efficiency. Using this idea SiO₂ coating was applied to the surface of abraded silica glass and was found²⁸ to be effective in reducing static fatigue.

8). IR peak assignments for the silica-water system.

IR peaks of wet and dry SiO₂ glasses were investigated^{18,29} and assigned. In particular, molecular water bands were analyzed in detail. This made it possible to determine relative amounts of molecular water in silica glasses hydrated at low temperatures. It was found^{18,29} that under a constant water vapor pressure, the amount of molecular water relative to hydroxyl water increased with decreasing treatment temperatures.

9). Water diffusion and structural changes in silica glasses.

At high temperatures, above $\sim 850^{\circ}\text{C}$, rates of both structural relaxation indicated by the shift of an IR absorption peak at 1873 cm^{-1} due to Si-O-Si vibrations and oxygen vacancy disappearance of oxygen-deficient silica glasses exposed to water vapor were faster³⁰ than the rate of water diffusion itself. At high temperatures, it is usually assumed that the water-glass network reaction is fast and that a local equilibrium is established between the reactants and product instantaneously. The present observation indicates that a minor fraction of water is diffusing faster, unrestricted by the chemical equilibrium, than the majority fraction, which is.

10). Water diffusion and time dependent mechanical properties.

Relations between water diffusion behaviors of silica glasses and time-dependent mechanical properties such as indentation creep and dynamic fatigue were investigated³¹. Water diffusion was measured at $350^{\circ}\text{C} \sim 650^{\circ}\text{C}$ where different behavior for different silica glasses is clearly observed, while indentation creep and dynamic fatigue were measured at room temperature. Comparison at these two different temperatures was considered acceptable since water diffusion at room temperature can be accelerated by applied stress. Three pairs of silica glasses were examined. (A) Suprasil W (type IV) vs. Infrasil (type I) with oxygen vacancies, (B) Corning silica glass (Corning Code 7940) vs. TiO_2 -doped silica glass (Corning Code 7971) and (C) silica glass with different fictive temperatures 1100°C vs. 1300°C . It was found that water diffusion was faster in (A) the silica glass without oxygen vacancy than that with oxygen vacancies; (B) the TiO_2 -doped glass than the un-doped glass and (C) the low fictive temperature sample than the high fictive temperature. In all three cases, the sample with a higher diffusion coefficient had the greater indentation creep rate. Also, for cases where data are available, the glass with the higher water diffusion coefficient had the greater susceptibility for dynamic fatigue (smaller n-value). [A part of these results were shown as Figure 1 of introduction of the proposal.]

11). Effect of nitrogen and carbon implantation on devitrification of silica glasses.

The crystal growth rate decreased³² when nitrogen was implanted in oxygen deficient silica glasses, while it was unaffected by ion-implantation in stoichiometric silica glass. It was suggested that the oxynitride bonding which formed in the surface layer reduced the water diffusion and diffusion-controlled crystal growth rates.

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