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DYNAMIC FATIGUE OF SODIUM-SILICATE GLASSES WITH HIGH WATER CONTENT

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Résumé.— On étudie la manière dont dépend la résistance mécanique de la vitesse d'application des contraintes pour un verre de silicate de sodium ayant un contenu en eau élevé (jusqu'à 25% en poids). Les verres ont été préparés par séchage de la solution commerciale de silicate de sodium dans un four ou dans un autoclave. La résistance mécanique a été mesurée par une méthode de flexion en quatre points pour des vitesses d'application des charges différentes. Ces verres ont montré une forte dépendance de la vitesse, même dans l'huile de paraffine sèche; elle augmente avec la teneur en eau. La valeur de n , mesure de cette dépendance est approximativement 5 pour les verres avec ~ 25% en poids d'eau. Parallèlement, le module d'Young décroît. On suggère que cette dépendance accrue résulte du mouvement d'eau sous l'action des contraintes appliquées. De plus, la valeur apparente du module d'Young décroît avec la vitesse de charge. Ce phénomène est expliqué par les propriétés visco-élastiques des verres.

Abstract.— The stress-rate dependency of the mechanical strength of sodium silicate glass with high water content (up to ~25% by weight) was investigated. The glasses were prepared by drying commercial sodium silicate solution in an oven or an autoclave. The mechanical strength of the glasses was measured by a four point bending method at various stress rates. These glasses showed a strong stress rate dependency even in dried paraffin oil and the dependency increased with increasing water content; the value of n , a measure of the stress rate dependency, was approximately 5 for glasses with ~25 wt% water. At the same time Young's modulus decreased with increasing water content. It is suggested that stress-induced motion of water is responsible for the increased stress rate dependency. In addition, the apparent value of Young's modulus decreased with decreasing stress rate. This phenomenon was explained in terms of the visco-elastic property of the glasses.

1. **Introduction.**— Water in the environment is well known to affect the mechanical strength of glass [1,2]; the strength measured in water is about one half of that measured in vacuum [3], and the strength decreases with increasing loading time (static fatigue) and with decreasing stress rate (dynamic fatigue) [4]. On the other hand, very little is known about the effect of water in glass on its mechanical strength. Recently, Wu [5] reported some data showing that the elastic modulus and the strength of hydrated silicate glass decreased with increasing water content, while McMillan et al [6] reported that the bending strength of the soda-lime silica glass with a small amount of water (below 780 ppm) was not influenced by the water content. So far, no research has been reported on details of the effect of the water in glass on the strength, especially on fatigue phenomenon.

In this study, the stress rate dependency of the mechanical strength and the elastic modulus of the sodium silicate glass with high water content were investigated.

2. **Experiment.**— The glasses with high water content were prepared by drying commercial sodium silicate solution (Na_2O ~8.9 wt%, SiO_2 ~28.7 wt%, H_2O ~62.4 wt%; $\text{Na}_2\text{O} \cdot 3.3\text{SiO}_2 \cdot 24\text{H}_2\text{O}$ by mole ratio) in an oven at 50 ~ 80 °C for 5 ~ 7 days in air (for

specimens with H₂O greater than 20 wt%) and then in an autoclave at 80° ~ 150°C for 3 ~ 5 days in ~30 bar N₂ (for specimens with H₂O less than 20 wt%). The water contents of these glasses were determined from weight loss by drying the glass at 400°C for 2h. The dry glass (Na₂O·3.3SiO₂: by mole ratio) was prepared by heating the glass with high water content at 400°C for 2h and subsequently melting the dried powder at 1450°C for 5h. The glass was annealed at 530°C for 2h. The water content of the dry glass was determined approximately from the peak intensity of IR spectra [7]. The glasses were cut into samples ~1.7 mm thick, ~3.5 mm wide, ~25 mm long with a diamond saw. The surface of the specimen was polished with SiC paper (600 grit). Prior to the mechanical strength measurement, the center region of one surface was abraded by a rougher SiC paper (240 grit), in a direction perpendicular to the long axis of the specimen. This surface was placed on the tension side during the mechanical testing. All the polishing and abrading were performed in paraffin oil.

The mechanical strength was measured by a four point bending method at five different crosshead speeds (1.27 ~ 0.00254 cm/min.) at room temperature. The testing was done in paraffin oil which had been dried using P₂O₅ for one week, to eliminate the effect of environmental water. At least 7 specimens were used to obtain one data point.

The Knoop hardness number was measured with a Kentron microhardness tester at room temperature. The glass surface was polished, using diamond paste. Within 15 min. after polishing, the indenter was brought into contact with the glass surface for 30 s. with a 200 g load. The length of the long diagonal of the indentation was measured immediately after removing the load.

3. Result and Discussion.- Fracture strength vs. stress rate as a function of water content in the glass is shown in Fig. 1. The glasses with high water content showed a strong stress rate dependency of the fracture strength, even though the strength was measured in paraffin oil. The dependency increased with increasing water content; the value of n [8], which was calculated from the slope of the lines in Fig. 1, decreased with increasing water content. On the other hand, the dry glass showed little stress rate dependency.

In Figure 1, the strength of the abraded dry glass was seen to be lower than that of the glass with 15.9 wt% H₂O, while the general trend appears to be lower strength for higher water containing glasses. This apparent discrepancy is probably due to the difference in the surface microcrack geometry caused by the different hardness. Figure 2 shows the Knoop hardness vs. water content. The hardness decreases with increasing water content of the glass. Thus it is

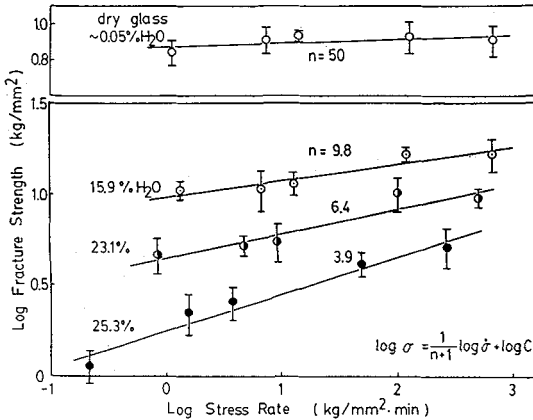


FIG. 1. Fracture strength vs. stress rate for glasses with various water contents. (Measured in paraffin oil at room temperature; ± standard deviation)

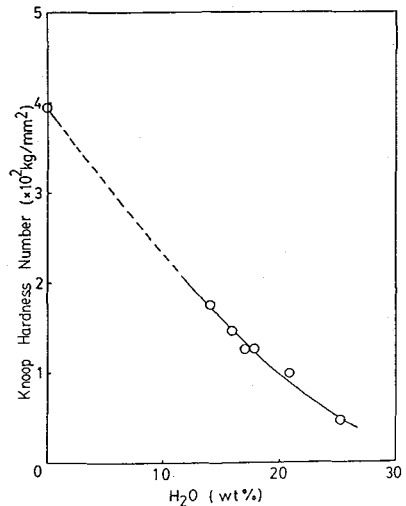


FIG. 2. Knoop hardness number vs. water content in glass.

expected that a glass with high water content would deform easily and a microcrack on its surface would have a more rounded tip than that of a dry glass. In fact it was observed that the abraded surface of the specimens with high water content became smooth indicating the healing effect. If the crack geometry is the same, the strength of the dry glass would probably be higher than that of the glass with 15.9 wt% H₂O.

Some glasses used in this study showed an apparent Young's modulus which varies with crosshead speed as shown in Fig. 3. For the highest water content (~25 wt% H₂O), and the low crosshead speed, apparently non-brittle fracture was observed as shown in the lower curves in Fig. 3.

The apparent Young's modulus value was calculated from the load and the deflection of the specimen [9] and is shown in Fig. 4 as a function of stress rate. From the figure, it is seen that the modulus decreases with increasing water content and the moduli of the glasses containing 23.1% and 25.3% water were markedly dependent on the stress rate, while the moduli of the dry glass and the glass containing 15.9 wt% water were independent of the stress rate.

These results suggest that the glass with high water content shows visco-elastic behavior. Using two parallel Maxwell models [10], the viscoelastic behavior was analyzed and the elastic modulus at infinite stress rate was calculated and is shown in Fig. 5. The modulus decreased gradually up to ~20 wt%, and then rapidly with increasing water content. This can be attributed to the gradual loosening of the glass structure due to the increasing number of broken $\equiv\text{Si-O}$ bonds by water. Beyond 20 wt% H₂O, structural loosening becomes accelerated since the glass transition temperature approaches room temperature [11] where all the measurements were made.

The stress rate dependency of the strength, namely, dynamic fatigue, has been usually explained by a corrosion reaction [1] of the glass with atmospheric water or a surface energy reduction [12,13] due to water adsorption. However, in this study, the effect of the water in the environment is considered negligible, since the strength was measured in dried paraffin oil and in fact, the n value of the dry glass, 50, was much higher than the usual n value [4] measured in the presence of water. This observation suggests that the dynamic fatigue of the glass containing water is induced by the water in the glass.

It is not clear whether or not the observed viscoelastic behavior has an influence on the stress-rate dependency of the strength. However, as shown in Fig. 1, the glass containing 15.9 wt% water shows dynamic fatigue, even though it shows elastic behavior (in Fig. 4), indicating that the fatigue tendency is accelerated by water in glass even when viscoelastic behavior is absent.

Generally, stress is known to cause diffusion [14], and alter the local concentration [15,16] of the solute in the solid. Therefore, when a load is applied to the glass with high

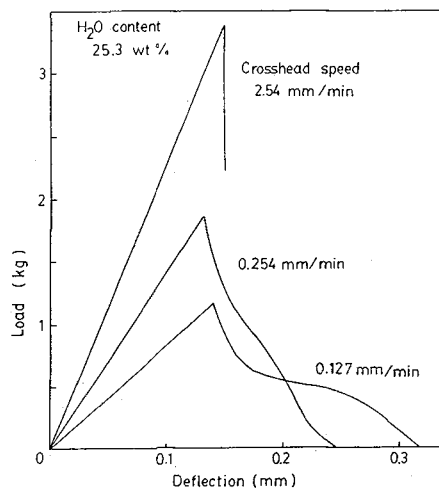


FIG. 3. Load vs. deflection at various crosshead speeds for glass with 25.3 wt% water.

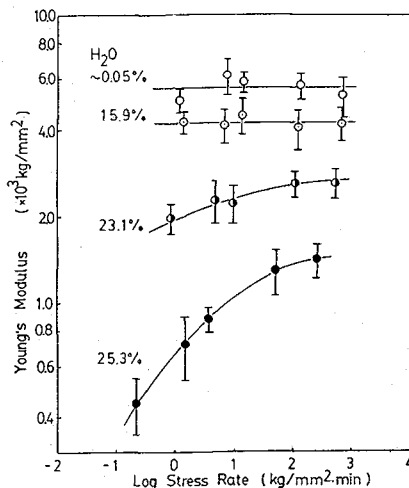


FIG. 4. Apparent Young's Modulus vs. stress rate for glasses with various water contents. (\pm standard deviation)

water content, the water in the glass is expected to diffuse to a crack tip, where a large tensile stress exists because of the stress concentration. It is possible that the water diffused to the crack tip reduces the strength of the glass causing the dynamic fatigue phenomenon. For example, there are indications [17,18] that the fracture toughness decreases with decreasing Young's modulus, at least for homogeneous glasses. Since Young's modulus was found to decrease with increasing water content, the strength of the glass would decrease with increasing amount of diffused water. The diffusion coefficient of water is expected to increase with water content. This would make the strength of the glasses with higher water content more stress rate susceptible, as was observed here.

4. Conclusion.- The water in the glass promotes the stress rate dependency (dynamic fatigue) of the mechanical strength and the visco-elastic phenomena, which makes the apparent Young's modulus value stress rate dependent.

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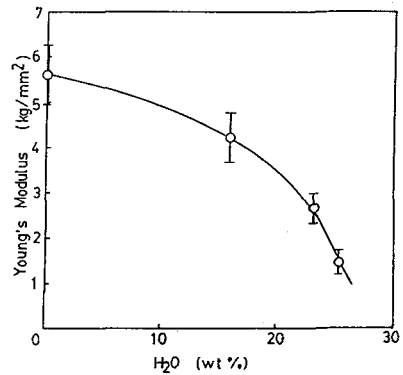


FIG. 5. Young's modulus at infinite stress rate vs. water content in glass (\pm standard deviation).