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#### EFFECT OF HYDROSTATIC PRESSURE

ON

ANELASTIC STRESS RELAXATION IN A MIXED-ALKALI SILICATE GLASS

BY

YOUNG-GIL KIM, 1939 -



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#### THESIS

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#### ABSTRACT

The effect of hydrostatic pressure on the anelastic stress relaxation of a mixed-alkali silicate glass (0.05 Na<sub>2</sub>O, 0.95 K<sub>2</sub>O, 3 SiO<sub>2</sub>) was studied at 75°C between ambient pressure and 6 kbar. The activation volume of diffusion was found to be - 4.2 cm<sup>3</sup>/mole. It is proposed that a reequilibration of the defect population at pressure gives rise to the negative activation volume of diffusion.

#### ACKNOWLEDGEMENT

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#### I. INTRODUCTION AND LITERATURE REVIEW

# A. ANELASTICITY AND DIFFUSION IN CRYSTALLINE SOLIDS

If a solid is subjected to a stress within the elastic limit, the resultant strain is the sum of an instantaneous elastic strain  $\varepsilon$  and another strain component that develops as time progresses; this latter strain is called an anelastic strain  $\varepsilon_{\rm AN}$  (see Figure 1). While the elastic strain is characteristic for the solid proper, the anelastic strain is the result of its defect structure. All classes of defects can give rise to anelasticity<sup>1</sup>. For example, the thermally activated motion of grain boundaries, dislocations, and point defects have been identified as the cause of anelastic effects<sup>2</sup>.

The anelasticity due to the thermally activated motion of point defects in crystalline solids is the simplest example and the easiest to interpret theoretically<sup>3</sup>. Consider interstitials located in an octahedral void of a bcc crystal as shown in Figure 2. Without an applied stress, the x-, y-, and z-voids are equally populated. If an uniaxial stress is applied in the [100] direction, the equilibrium population will change provided the interstitials are mobile. The reason for the change is the fact that the free energy of the crystal can be lowered if a majority of the interstitials resides in the x-voids, thus relieving part of the applied stress. Basically the same phenomena will occur whenever a stress is applied to a crystalline solid containing point defects provided their symmetry is lower than the symmetry of the host lattice

STRAIN VERSUS TIME FOR AN ANELASTIC CRYSTAL

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TIME



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bcc UNIT CELL WITH THREE ENERGETICALLY AND CRYSTALLO-GRAPHICALLY EQUIVALENT OCTAHEDRAL INTERSTITIAL SITES



and the product of the stress and defect strain tensors is different from  $zero^4$ .

As a change of the point defect population will have to occur by defect motion, i.e. diffusion, the rate at which the anelastic strain develops contains information about the diffusivity of the defects<sup>3</sup>. If the defect travels a distance a in the time interval  $\tau$ , its diffusivity D is given by

$$D = \frac{a^2}{\tau} \cdot g \qquad [1]$$

where: g - geometrical factor, g =  $\frac{1}{24}$  for above mentioned defect

a - distance between neighboring voids Since the defect motion is thermally activated, reaction rate theory<sup>5</sup> can be applied in order to obtain an expression for  $\tau$ . The result is

$$\tau = \tau_0 \text{ EXP } \left(\frac{\Delta G}{kT}\right)$$
 [2]

where:  $\tau_0$  - constant

 $\Delta G$  - activation energy

- k Boltzman constant
- T absolute temperature

Consequently, we may derive an expression for the diffusivity as follows:

$$D = a^{2} \cdot g \cdot \tau_{0}^{-1} \cdot EXP\left(-\frac{\Delta G}{kT}\right)$$
 [3]

In practice,  $\tau$  is obtained from the time dependence of the anelastic strain. It is given by an exponential law as can be expected for a physical process where the rate of change depends on the number present:

$$\varepsilon_{AN \cdot MX} - \varepsilon_{AN} = \varepsilon_{AN \cdot MX} EXP \left(-\frac{t}{\tau}\right)$$
 [4]

A similar expression holds for the stress relaxation:

$$\sigma_{AN \cdot MX} - \sigma_{AN} = \sigma_{AN \cdot MX} EXP \left(-\frac{t}{\tau}\right)$$
 [5]

Due to irregularities in the solid  $\tau$  represents a mean of a distribution of relaxation times. If a Gaussian distribution of the relaxation times is assumed, the stress relaxation is given by  $\frac{6}{2}$ 

$$\sigma_{AN \cdot MX} - \sigma_{AN} = \frac{\sigma_{AN \cdot MX}}{\sqrt{\pi}} \int_{-\infty}^{+\infty} EXP \ (-x^2) \ EXP \ [-\frac{t}{\tau_m} \cdot EXP \ (-\beta x)] dx \ [6]$$

where:  $\sigma_{AN \cdot MX}$  - relaxation strength

The above relations are valid for aperiodic stresses and strains. If a periodic stress is applied to a solid, the anelasticity manifests itself in a lag of the strain behind the stress. This phenomenon is called internal friction. Analogous to electrical power dissipation, its magnitude is given in terms of the tangent of the phase angle between stress and strain which is given by<sup>1</sup>

$$Q^{-1} = \Delta \frac{\omega \tau}{1 + (\omega \tau)^2}$$
 [7]

$$\Delta - relaxation ratio: \frac{G_0 - G_{\infty}}{G_{\infty}}$$

 $\omega - 2\pi f$ 

 $G_0$  - instantaneous shear modulus

G\_ - relaxed shear modulus

f - frequency of measurement

#### B. PRESSURE DEPENDENCE OF THE DIFFUSIVITY

Equation 3 can serve to derive the pressure dependence of the diffusivity. As the Gibbs free energy is given by

$$\Delta G = \Delta E - T\Delta S + P\Delta V$$

it follows that:

$$kT \frac{\partial \ln D}{\partial p} \Big|_{T} = -\Delta V_{act} + \frac{\partial \ln g a^{2} v}{\partial p} \Big|_{T} kT \qquad [8]$$

where: v = vibration frequency $\Delta V_{act} = \frac{\partial \Delta G}{\partial p} \Big|_{T}$ 

It can be shown that the second term in Equation 8 is small and contributes less than 10% to the pressure dependence<sup>7</sup>. Hence, the pressure dependence of the diffusivity is given by:

$$\Delta V_{act} = \frac{\partial \Delta G}{\partial p} |_{T}$$
 [9]

The magnitude of the activation volume of diffusion may be expected to be of the order of the atomic or ionic volume of the diffusing species, whichever applies. It is characteristic of this species. Physically, the activation volume represents the dilatation (or, possibly compression) of the solid as the diffusing species resides in the activated state. If the activated state is an equilibrium state, a thermodynamical argument may be applied in order to rationalize the activation volume in terms of the elastic distortion of the lattice. On the basis of this approach, it was found<sup>8</sup> that the activation volume of diffusion is related to the pressure derivative of the elastic constants through

$$\frac{\Delta V_{act}}{\Delta G^{*}} = \frac{\partial \ln C}{\partial p} \Big|_{T} - X$$
 [10]

C - elastic shear modulus

X - isothermal compressibility

 $\Delta G^*$  - free energy of lattice imperfection The above relation may be rewritten as

$$\frac{\Delta V_{act}}{\Delta G^{\star}} = 2 \left(\gamma - \frac{1}{3}\right) X \qquad [11]$$

γ - Grüneisen constant

It should be noted that Equation 11 yields a positive activation volume of diffusion always.

#### C. ANELASTICITY AND DIFFUSION IN GLASSES

Contrary to crystalline solids, glasses do not possess long range order. Rather, on an atomic scale silicate glasses consist of distorted arrays of SiO<sub>4</sub> tetrahedra which may be slightly distorted, i.e., display deviations from the shape of a regular tetrahedron. Thus, in referring to the SiO<sub>4</sub> tetrahedron, it may be said that glasses display short range order. Furthermore, if the basic SiO<sub>4</sub> tetrahedron is excessively distorted due to the presence of what may be called **an** interstitial or substitutional ion, these perturbations of the basic glass structure represent point defects in the sense that they destroy the short range order. As an example, consider the structure of the glass (1-x) Na<sub>2</sub>O, xK<sub>2</sub>O, 3SiO<sub>2</sub> as shown<sup>9</sup> in Figure 3. In this glass, the short range order is destroyed by a pair of mixed-alkalis with the overall mixing

ATOMIC CONFIGURATIONS OF THE  $(1-x)Na_2O$ ,  $xK_2O$ ,  $3SiO_2$  GLASS

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ratio given by x. As can be seen from Figure 3, there will be four kinds of point defects:

- Oxygen ions which are only bonded to one silicon, so called "nonbridging" oxygen ions
- (2) Na<sup>+</sup> K<sup>+</sup> "pairs"
- (3) Na<sup>+</sup> Na<sup>+</sup> "pairs"

(4) 
$$K^{+} - K^{+}$$
 "pairs"

The relative concentrations of these types of pairs will be given by the ratio x and the respective Gibbs free energies of pair formation. In addition, depending on the differences in the Gibbs free energies of formations of the various kinds of defects, "unpaired" Na<sup>+</sup> and K<sup>+</sup> ions may also exist. On the basis of the discussions in the preceding paragraphs, it can be concluded that a variety of anelastic effects will be observed in silicate glasses. There should be at least two kinds of anelastic effects in simple alkali glasses, and four kinds in mixed-alkali silicate glasses. Internal friction studies in a series of mixed-alkali silicate glasses<sup>10</sup> could indeed separate four distinct internal friction peaks. This observation tends to confirm the crude hypothesis presented above although there is still enough room for different interpretations<sup>11</sup>. For instance, one of the observations which this hypothesis fails to explain is the occurrence of the mixedalkali peak at extremely low concentrations of the second alkali<sup>12</sup>.

As outlined previously, the activation volume of diffusion is a quantity characterizing the defect instrumental in the matter transport. In order to help identify the defect giving rise to the mixed-alkali peak, the activation volume of the diffusion in a mixed-alkali glass was measured. Since the internal friction peak height of the mixed-alkali glass is largest in Na-K glasses, it was used for the present study. In order to facilitate a direct comparision with the previous internal friction data<sup>10</sup>, the aperiodic stress relaxation was measured as a function of hydrostatic pressure.

#### **II.** EXPERIMENTAL PROCEDURE

#### A. MEASUREMENT OF ANELASTIC STRESS RELAXATION

A slightly modified version of the experimental technique described by Lazarus<sup>7</sup> was used. A schematic diagram of the apparatus is shown in Figure 4. In this arrangement, the glass helix is strained by the magnetic field of the solenoid. The solenoid was designed such that the product of the magnetic field times its axial gradient at the position of plunger is constant<sup>13</sup>.

The relaxation of the glass spring was followed by measuring the solenoid current necessary to keep the plunger from contacting the stopper (Figure 4). For this purpose a control circuit was designed which reduced the solenoid current until the plunger broke contact with the stopper. The detailed circuit is shown in Figure 5. In combination with the springs used, it permitted stress relaxation curves characterized by  $\tau^{\geq}$  0.5 min. to be recorded.

In order to determine whether or not the plunger made contact with the stopper, a conducting copper helix was mounted parrallel to and inside of the glass helix. This copper helix has been omitted from Figure 4. The dimension of the inner copper helix was chosen so that its spring constant comprised only .1% of the spring constant of the glass helix. Consequently, no corrections of the glass relaxation data due to the presence of this copper helix were made.

P - Plunger

S - Stopper

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A - Stopper

### SCHEMATIC DIAGRAM OF THE RELAXATION APPARATUS

#### FIGURE 4



FIGURE 4

DETAILED CIRCUIT DIAGRAM OF THE RELAXATION CONTROL CIRCUIT

- A Automatic operation
- M Manual operation
- F Forward rotation of the synchronous motor
- R Reverse rotation of the synchronous motor
- L Signal light
- S Switch (1,2)
- O Off
- All resistors are  $k\Omega$  unless otherwise noted.
- All capacitors are µF unless otherwise noted.



#### B. HIGH PRESSURE SYSTEM

A commercial high pressure booster, High Pressure Equipment Company Model # PS-150, was used to generate pressures up to 6 kbar. The pressure was determined with a manganin guage, and the relaxation apparatus was put into a Cu-Be vessel designed for disaccomodation measurements<sup>14</sup>. A schematic diagram of the high pressure system is shown in Figure 6. A mixture of 1 part Kerosene and 2 parts Diala-AX was used as pressure fluid. The Cu-Be vessel had a built-in heater controlled by a proportional-rate-repeat controller. This arrangement allowed the temperature to be controlled to within ± .05°C.

#### C. SAMPLE PREPARATION

The glass fibers used were from the stock used by Shelby and Day<sup>10</sup> for their internal friction measurements. The glass helix was wound on a tapered and heated graphite cylinder. The completed helix was given the same heat treatment as the sample used in Reference 10. All measurements reported in this thesis were made on one glass sample of the nominal composition of 0.05 Na<sub>2</sub>O, 0.95 K<sub>2</sub>O, 3 SiO<sub>2</sub>.

#### HIGH PRESSURE SYSTEM

A -	Reservoir	J - Pressure Guage
в -	Hydraulic Filter	K - Intensifier
c -	Pump	L - Check Valve
D -	Air Line Filter	M - Vent Valve
Е -	Air Line Regulator	N - High Pressure Outlet Valve
F -	Air Inlet Valve	0 - Manganin Gauge
G -	Air Line Filter	P - High Pressure Vessel
н –	Valve	S - Air Supply
I -	Valve	



#### III. EXPERIMENTAL RESULTS

The stress relaxation at constant strain of the 0.05 Na<sub>2</sub>O, 0.95 K<sub>2</sub>O, 3 SiO<sub>2</sub> glass was measured at 75°C in the pressure range of 1 bar to 6 kbar. The temperature of 75°C was chosen because the majority of the relaxation occurred between 1 and 10 minutes after application of the stress at this temperature (see Figure 7). All relaxation measurements were made at a strain amplitude of about  $5 \times 10^{-2}$  as determined by the respective positions of the stopper and plunger. Before a run was started, the helix was left unstrained *at pressure* for at least twice the time of the total length of the past relaxation run. If the glass helix was left unstrained at ambient pressure just prior to the run, a transient stress relaxation was observed. This observation will be significant in the discussion and will be described more fully below.

Figures 8 through 11 contain all data taken. A typical stress relaxation run is shown in Figure 8. The relaxation data points were taken from the recorder chart mentioned previously. In Figure 8 the solid line represents the least square fit<sup>15</sup> of Equation 6 to the data. The parameters fitted are  $\beta$ ,  $\sigma_{AN}$ , and  $\tau$ . The value of  $\beta$  obtained in this way is 3.0 which agrees fairly well with other observations<sup>16</sup>. It can be seen from Figure 8 that the least square line fits the data to within 5%. The largest deviation occurs at the long time end of the fit which is due to the uncertainity of the value of the completely relaxed stress. In addition, there

MAGNITUDE OF STRESS RELAXATION AT AMBIENT PRESSURE VERSUS TEMPERATURE

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# OBSERVED AND CALCULATED STRESS

# RELAXATION OF 0.05 Na<sub>2</sub>O, 0.95 K<sub>2</sub>O, 3 SiO<sub>2</sub> GLASS

## AT 1 kbar AND 75°C

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was no logical way to obtain information about the background relaxation which undoubtedly exists<sup>17</sup>. This lack of background correction is thought to contribute to the deviations between the measured and fitted curves. Figure 9 shows the relaxation time of the 0.05  $Na_2O$ , 0.95  $K_2O$ , 3  $SiO_2$  glass as a function of hydrostatic pressure as obtained from the least square fits. The error bars correspond to the 95% confidence limit. As can be seen from this Figure, a decrease of the relaxation time with increasing pressure was found, contrary to the expectations. The value of the activation volume of diffusion to be calculated from this plot would be  $-4.2 \text{ cm}^3/\text{mole}$ . The relative relaxation amplitude, the ratio of the relaxation amplitude and the initial stress, increased at pressures above 3 kbar as is shown in Figure 10. The amount of the stress relaxation at ambient pressure agrees well with the relaxation of the shear modulus observed in the internal friction measurements<sup>10</sup> on the same glasses.

A further interesting observation is the increase of the stress at zero time with increasing pressure shown in Figure 11. It represents an increase in the elastic modulus. The reason for this modulus increase is presently obscure.

All data presented so far represent equilibrium data in the sense that they could be reproduced provided the sample was kept at the measuring pressure for more than 10 minutes. After this period the characteristics of the stress relaxation no longer changed (see Figure 12). This transient behavior was not thoroughly investigated and recorded. Thus only its qualitative features can be reported in this thesis. They are

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# RELAXATION TIME OF A 0.05 Na<sub>2</sub>O, 0.95 K<sub>2</sub>O, 3 SiO<sub>2</sub> GLASS VERSUS PRESSURE MEASURED AT 75°C



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# RELATIVE STRESS RELAXATION AMPLITUDE OF A 0.05 Na<sub>2</sub>O, 0.95 K<sub>2</sub>O, 3 SiO<sub>2</sub> GLASS VERSUS PRESSURE MEASURED AT 75°C

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INITIAL STRESS OF A 0.05 Na<sub>2</sub>O, 0.95 K<sub>2</sub>O, 3 SiO<sub>2</sub> GLASS VERSUS PRESSURE MEASURED AT 75°C



TRANSIENT BEHAVIOR OF THE AFTER-PRESSURIZATION OF A 0.05 Na $_2O$ , 0.95 K $_2O$ , 3 SiO $_2$  GLASS AT 75° C, SCHEMATIC

- A Immediately after-pressurization
- B About three minutes after-pressurization
- C About five minutes after-pressurization
- D About ten minutes after-pressurization



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#### IV. DISCUSSION

The main question arising from this work is whether or not the reported activation volume of diffusion of  $-4.2 \text{ cm}^3/$ mole is representative for a single point defect or not. While no data exist to which this value may be compared directly, it is in contradiction with the activation volume reported for unmixed glass of the same alkali-oxide to silica ratio<sup>18</sup>. It is also in contradiction with thermodynamical arguments (see Paragraph I.-B) if it is assumed that only one defect species is responsible for the anelasticity at all pressures. However, a negative activation volume of diffusion can be rationalized if a two defect model is assumed; a concept which has been successfully applied to silver halides<sup>19</sup>. This is proposed in this thesis.

It has been shown that in crystalline solids a relaxation under hydrostatic pressure can occur only if a reaction between defects takes place as the solid is pressurized<sup>4</sup>. This result holds for noncrystalline solids as well. Therefore, the transient anelasticity shown in Figure 12 is indicative of a reaction between defects. In terms of the schematic model presented in the previous paragraph, it might be envisioned that the partial molar volume of, say a sodium ion is different when paired with another sodium as when paired with a potassium ion. Thus, if the glass is subjected to a hydrostatic pressure the defect population changes. This change will be diffusioncontrolled. The finite time which it takes to reach the

equilibrium defect population after pressurization observed in this study (see Figure 12) confirms this point of view.

In summary, it is proposed that the observed negative activation volume of diffusion is the result of a reequilibration of the defect population under pressure and is not characteristic for any one defect or diffusion mechanism.

#### V. CONCLUSIONS

Measurements of the pressure dependence of the stress relaxation of a mixed-alkali silicate glass  $(0.05Na_2O,$  $0.95K_2O, 3SiO_2)$  yielded a negative activation volume of diffusion. It is concluded that this unusual pressure dependence is the result of a reequilibration of the defect population in the glass under pressure.

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#### VII. VITA

Young-Gil Kim (Gilbert Y. Kim) was born on October 3, 1939 in Andong, Korea. He attended elementary and junior high school in Andong and received senior high school education in Seoul. He received a Bachelor of Science in Metallurgical Engineering from the College of Engineering, Seoul National University in February 1964. Afterwards, he served in the Army as an Ordnance Officer for about two years.

He has been enrolled in the Graduate School of the University of Missouri-Rolla since September 1967 and has been appointed a Graduate Research Assistant in June 1968.

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