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The influence of temperature and environment on the slow crack propagation in glass

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Summary

The crack velocity of a straight crack in a plane specimen was measured from 10^{-7} cm/s up to 10 cm/s as a function of crack extension force G from 10^3 erg/cm^2 up to 10^4 erg/cm^2 in three different environments: water, air containing 11·3 gr/m^3 water vapour, and high vacuum of 10^{-6} Torr. The temperature was varied between 20°C and 100°C. The results show that different mechanisms participate in crack propagation. In water and humid air the mechanism appears to be one of stress corrosion, but results in vacuum indicate that much slower propagation can occur in the absence of a corrosive environment.

Introduction

A great deal of experience has been gathered about the rapid propagation of cracks ($v > 1000$ m/s), especially about the maximum crack velocity. However, very little is known about slow crack propagation ($v < 1$ m/s). Only Widerhorn [1] has published detailed investigations on this problem.

Crack propagation is influenced at sufficient small velocities by the environment and the temperature. The effect of water or humidity is particularly striking and is known from the behaviour of strength of many glasses. It may be assumed that a chemical process like stress-water-corrosion is responsible for this [2, 3, 4]. During this work special attention was given to the problem, whether a crack can extend slowly without any corrosion process, i.e. whether velocities smaller than 1 cm/s can be attained without catastrophic failure of the specimen. Therefore tests were also carried out in high vacuum.

During crack propagation energies are transformed at the crack front [5]. Energy is absorbed by forming of new surfaces, by microplastic deformations, by acceleration and probably by chemical and electrical processes. The energy offered at the tip of the crack is composed by the energy of elastic stress concentration at the tip, the thermal energy and the energy due to chemical processes. It is difficult to calculate the quantitative influence of chemical and thermal energies on the crack propagation. However, in simple problems the change in energy of the elastic stress field can be calculated.

One of the most simple problems is the propagation of a straight crack in a plate under homogeneous tensile stress (Fig. 2 (a)). For this case Irwin has stated the crack extension force G [6, 7]:

$$G = \frac{(1-\mu^2)\cdot\pi}{E}\cdot\sigma^2\cdot a\cdot k^2\left(\frac{a}{b}\right) \qquad (1)$$

σ = homogeneous tensile stress at a sufficient distance from the tip of the crack.
E = Young's modulus
a = crack length
b = width of the specimen
$k(a/b)$ = correction factor
μ = Poisson's ratio

Apparatus and experimental measurements

The apparatus is shown in Fig. 1. In order to get an uniaxial tensile stress the specimen hangs vertically. The loading was done by a twisting-free vertical suspension in a cylindrical copper container. (see Fig. 1 and 2 (b). The force was produced either by a weight or by a pneumatical loading system and was measured with strain gauges. The inertia of the loading device was negligible for velocities smaller than 1 m/s. According to the experimental conditions the container could be filled with water or with air of definite humidity, or it could be evacuated up to a pressure of 10^{-6} Torr. The temperature could be regulated between 20 °and 100 °C by a thermostatically controlled liquid pumped through bores in the wall of the container. The temperature was measured with a thermocouple on the specimen near the crack. Through windows the crack could be illuminated and observed by a microscope. The whole apparatus was mounted on a heavy stone platform resting on springs in order to protect it from vibrations.

The crack velocity v was measured in different ways according to its magnitude. For $v < 10^{-5}$ cm/s an automatic microscope camera photographed the crack after every 30 min. The change of the crack length could be measured on the film with the aid of markings on the specimen. For $10^{-5} < v < 10^{-2}$ cm/s the movement of the crack was directly observed with the microscope. When the crack traversed a definite length seen through a micrometer eye-piece, a printer connected to an electronic timer was manually operated. In the range of $10^{-2} < v <$ cm/s, the crack was filmed with a high speed camera (Fastax) in combination with a microscope. With the time markings on the border of the film, the time intervals could be determined.

The specimens were slides of soda lime glass (Deutsche Spiegelglas AG, Grünenplan) with the following dimensions: length 70 mm, width 15 mm, thickness 0·5 mm. The specimens were carefully tempered. With the aid of Vickers indentations on the edge of the specimen (see Fig. 2 (a), original crack lengths of 1 to 2 mm could be produced by cautiously loading the specimen under tension.

The crack extension was investigated in three different environments: In distilled water, in air containing 11·3 gr/m^3 water vapour, and in high vacuum of 10^{-6} Torr. For the experiments in water the temperatures were set at 20, 40, 60, 80 and 98 °C. The experiments in air and in vacuum were carried out at 23 °C and 100 °C.

The crack velocity v was determined by the time interval Δt passed between two crack lengths a_i and a_{i+1} and the difference Δa of these lengths. This velocity was coordinated to the average crack length $(a_i + a_{i+1})/2$. Every test delivers a number of points forming a curve $v = v(a)$ or $v = v(G)$. At least 6, mostly 8 to 14 tests were performed for every setting to get satisfactory averages.

The crack length is one of the parameters determining the crack velocity. It is necessary to know this length as exactly as possible. At first there were doubts, whether the tip of the crack as seen through the microscope is identical with the true tip, i.e. whether the measured crack lengths were smaller than the true lengths or not. To investigate this, small markings were placed near the crack (see Fig. 3). The distance between the markings and the crack tip was measured. Then the specimen was broken by twisting. According to the hypothesis of normal stress [8] the crack in glass always propagates perpendicular to the direction of the principal stress. Breaking by twisting changes the direction of the principal stress. Thus the twisting must cause a spontaneous change of the crack direction, which appears as a sharp bend on the fracture plane. The distance of this bend from the markings was also measured. This distance was found to be the same as the distance of the crack tip from the markings within the measuring accuracy (± 1 μm).

Equation (1) shows that due to the finite width of the specimen the correction $k(a/b)$ of the G-value is necessary. In the literature different correction functions are given from theoretical considerations and from experimental measurements (7, 9, 10, 11, 12). Some of them differ considerably from each other. Experimental values derived from compliance measurements by Srawley [11] agree with theoretical results of Gross [9] only for $0{\cdot}2 \leqslant a/b \leqslant 0{\cdot}5$ with an error of a few precent.

The value of the correction function depends on the length of the crack According to equation (1) the same G-value can be obtained for different crack lengths a by changing the tensile stresses b. The assumption, that identical crack velocities correspond to identical G-values if the experimental conditions did not change, makes it possible to check the correction function. It could be demonstrated that Gross' results agree also with experimental average values for $0{\cdot}07 \leqslant a/b \leqslant 0{\cdot}25$ with 1·5% error.

Results, discussion

In Fig. 4, 6 and 7 the crack velocity v is plotted against the crack extension force G. All the points plotted in the diagrams are averages. For some of these points the mean square error of the average is given. Fig. 4 shows the complete results of the measurements in water, air and in vacuum, all at two different temperatures.

Tests in water

When G increases tenfold, the increase in crack velocity is about 10^7 times. The smallest average velocity was about 5.10^{-7} cm/s, which corresponds to a G value of 10^3 erg/cm². For different values of G the crack velocity does not depend uniformly upon the temperature. With the change from 20°C to 80°C the increase of v is largest at $G = 3000$ erg/cm², viz. 40 times. At $G = 8000$ erg/cm² the increase is only 3 times.

In the range of $5.10^{-5} < v < 5.10^{-2}$ cm/s, the relation between v and G was examined at temperatures between 20°C and 98°C. As Fig. 5 shows, we get straight lines when $\log v$ is plotted over $1/T$ for constant G-values. T is temperature in degree Kelvin. Therefore a relation of the form $\log v = c_1 - (c_2/T)$ exists. Assuming that a corrosion process with alkali- and hydrogen ion-exchange takes place at the crack front (2, 3), a thermal activation can be expected. From the Arrhenius equation the activation energy ΔE can be calculated which amounts to 13·6 kcal/mol at $G = 2500$ erg/cm². As expected, ΔE decreases with increasing G. However, the dependence of the crack velocity on G is not exponential, as seen in Fig. 4.

Tests in air containing 11·3 gr/m³ water vapour

The two middle curves of Fig. 4 show the characteristic behaviour during loading in air (23°C and 100°C). In Fig. 6 the results are plotted in a linear scale. Three regions can be differentiated in which three different functions of the crack velocity from G are observed. In the range of 4000 to 8000 erg/cm², v increases almost linearly with G, i.e. $\Delta v/\Delta G$ is constant (see Fig. 6). Above 8000 erg/cm² the velocity increases exponentially i.e. $\Delta v/\Delta G$ increases monotonically whereat the relative change $1/v \cdot (\Delta v/\Delta G)$ is constant. In the range of small crack velocities (v 5.10^{-3} cm/s), v and $\Delta v/\Delta G$ increase monotonically with G, and the relative change which is large here diminishes monotonically with increasing velocity.

Obviously, in these three regions, different mechanisms are responsible for the crack propagation. Observation of the crack front profile for velocities between 10^{-3} cm/sec and 5.10^{-2} cm/sec leads to following conclusions (Fig. 6). With increasing crack velocity the inner part of the crack front lags behind the outer parts adjacent to the specimen surface. From this observation it can be concluded that transport processes are important in this range. With increasing crack velocity, the number of water molecules per unit time reaching the crack front by diffusion decreases. Ultimately the corrosive action becomes negligibly small, but the stress concentration at the crack tip becomes sufficiently great to extend the crack further.

Crack propagation without corrosion mechanism can be assumed from these observations for crack velocities above 5.10^{-2} cm/sec. If this is correct, the v-G-curves measured in vacuum should join the upper curves measured in air.

Tests in high vacuum

The results of tests in vacuum (Fig. 7) confirm the above assumption: the values measured at 20°C as well as at 100°C lie well on the corresponding ‘air curve’. Plotted logarithmically they also are straight lines.

Further investigation was made into the effect of an increase in pressure up to 10^{-4} Torr on the results. No significant difference was found (Fig. 7), i.e. the influence of the residual moisture in the evacuated container is negligible. Fundamentally it is possible, that absorbed water molecules spread out from the surface to the crack tip and influence the crack propagation. But the spreading velocity is smaller by some powers of ten than the diffusion velocity. This appears to rule out this type of mechanism.

Until now it was unknown, whether slow crack propagation without corrosion exists, and how v depends upon G and temperature in this case. The measurements appear to confirm that this type of crack propagation exists.

Reliable results of measurements in vacuum have been hitherto available only for temperatures of 23° and 100°C. Thus it has not been possible to confirm the validity of the Arrhenius equation. However, the strict linearity between the logarithm of the crack velocity and the crack extension force is striking, and therefore an equation of the form

$$v = v_0(T) \exp\left(-\frac{U - \alpha G}{RT}\right) \tag{2}$$

may apply. For high crack velocities, this equation can not be valid because the crack velocity is limited by a maximum velocity. To describe the crack propagation, different authors have stated theoretical equations [13, 14, 15]. Gibbs and Cutler [16] give the following equation according to a model of Eyring [17].

$$v = \lambda\left[\frac{kT}{h}\exp\left(\frac{-\Delta F + L_1(a)(\sigma - \sigma_0)}{RT}\right) - \frac{kT}{h}\exp\left(\frac{-\Delta F - L_2(a)(\sigma - \sigma_0)}{RT}\right)\right] \tag{3}$$

where

λ = average increase of crack length during the separation of a pair of atoms.
k = Boltzmann's constant ΔF = Gibbs' free energy
h = Planck's constant a = crack length
$L_1(a)$ and $L_2(a)$ = a kind of stress concentration factors.
σ = applied stress
σ_0 is the smallest stress at which the crack propagation begins.

ΔF has the dimension of an energy, therefore σ should be quadratic in the exponent. It follows, that the L-functions are not only dependent on the crack length but also on the stress σ.

The first term in the brackets gives the rate of breaking bonds per unit time, the second term gives the rate of closing bonds per unit time. Neglecting the second exponential term in the bracket, which is significant only for very small stresses, equation (3) can be identified with equation (2) by substituting $v_0(T) = c \, . \, T = \lambda kT/h$ and setting the crack extension force for the unknown function L_1 (a) $(\sigma - \sigma_0)$.

$$v = \frac{\lambda kT}{h} \exp - \left(\frac{U - \alpha G}{RT}\right) \tag{4}$$

For a given G and for $t_1 = 23°C$ and $t_2 = 100°C$, $\Delta E = U - \alpha G$ and c or λ can be calculated from the measurements. For G = 8500 erg/cm² we get $\Delta E = 14{\cdot}8$ kcal/mol, $\lambda = 374$ Å. The average interatomic distance, however, for the used glass is about 2 Å, i.e. λ is 187 times greater.

Calculated from the measurements, ΔE depends for constant G on the ratio of the crack velocity at 100°C to that at 23°C. For G = 8500 erg/cm² we get $v_{100}/v_{23} = 226$. For a given $\lambda = 2$ Å and for a constant t (23°C), ΔE^* can be calculated from equation (4). $\Delta E^* = 11{\cdot}6$ kcal/mol. With the values of λ and ΔE^* we can also calculate a crack velocity for 100°C and get $\overset{*}{v}_{100} = 2{\cdot}74 \, . \, 10^{-2}$ cm/s for G = 8500 erg/cm² and a ratio $v^*_{100}/v_{23} = 88$. This ratio is 2·56 times smaller than the previous one. This can be possibly due to the scattering of the measurements (Fig. 4). Thus the accuracy of the measurements is not sufficient to check equation (4).

Conclusions

Slow crack propagation ($v < 1$ m/s) in glass is dependent not only on the state of stress but also on the environment and the temperature. The crack velocity of a straight crack in a plate was measured as a function of crack extension force G in three different environments: water, air containing 11·3 gr/m³ water vapour, and high vacuum of 10^{-6} Torr. The temperature was varied between 20°C and 100°C.

The smallest measured velocities were 10^{-7} cm/s and the highest 20 cm/s, corresponding to the range of G from 10^3 erg/cm² to 10^4 erg/cm².

The results show that different mechanisms participate in crack propagation. In water and in humid air the crack propagation can be interpreted as stress-water-corrosion, wherein transport phenomena are also involved. The results in high vacuum show that also without corrosion a slow crack propagation occurs.

In all cases crack propagation was observed to be dependent on the temperature. The activation energy for the measurements in water is $\Delta E = 13{\cdot}6$ kcal/mol for G = 2500 erg/cm² and in vacuum $\Delta E = 14{\cdot}8$ kcal/mol for G = 8500 erg/cm².

The results of measurements in vacuum can be described by an exponential function. This function is compared with the equation of Gibbs and Cutler.

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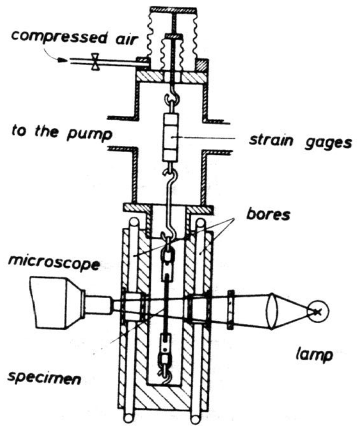

Fig. 1. The apparatus, schematic.

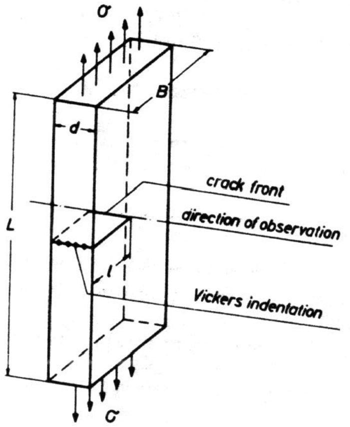

Fig. 2a. Specimen schematic.

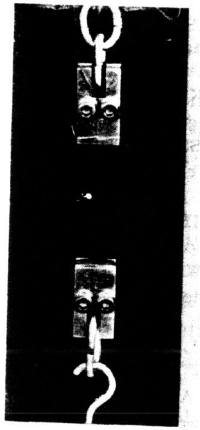

Fig. 2b. Specimen with loading device.

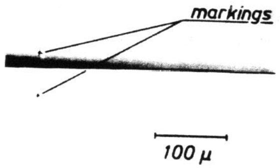

Fig. 3. Microscopic photograph of the tip of the crack with markings.

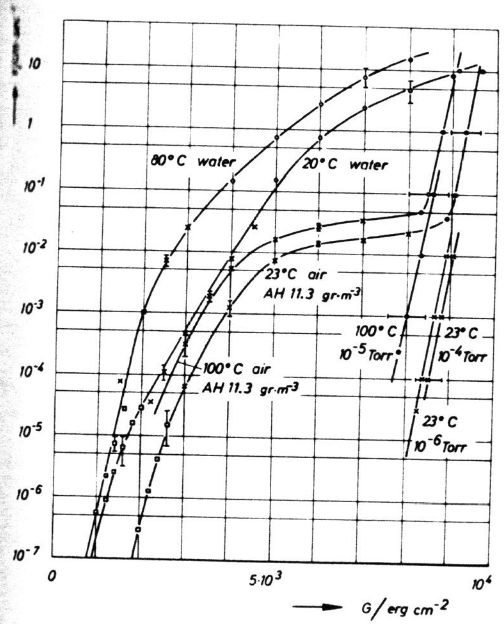

Fig. 4. Crack velocity as a function of crack extension force. Loading in water, in air and in vacuum.

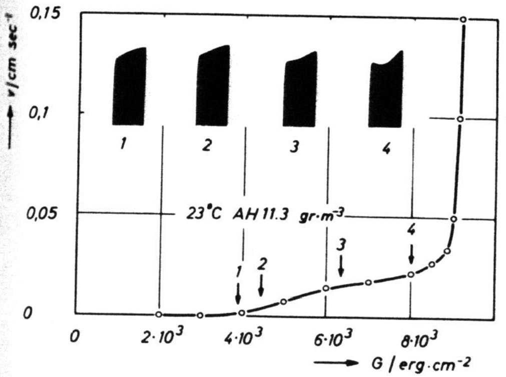

Fig. 6. Crack velocity as a function of crack extension force, loading in air, (linear plotting) and profile of crack front in transition range.

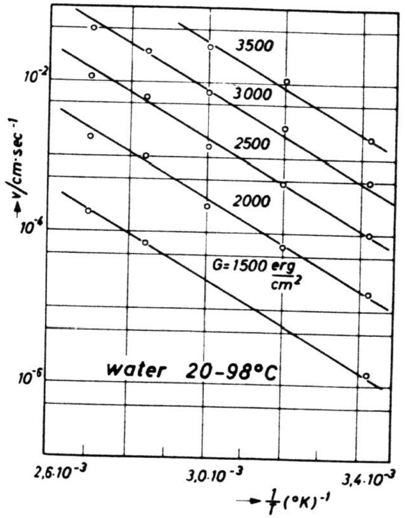

Fig. 5. Crack velocity as a function of temperature. Loading in water.

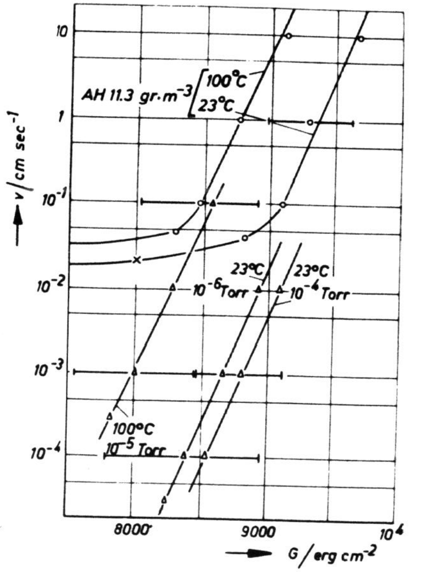

Fig. 7. Crack velocity as a function of crack extension force. Loading in vacuum (together with the upper part of 'air curve').