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What does the pendulum hardness test measure?

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Various modes of energy absorption are considered as possible explanations for pendulum attenuation in the dynamic hardness testing of soda lime silica glass. It is concluded that the test gives little direct information about fracture processes, the major part of the energy absorption being accounted for by plastic flow in the solid as in the case of static hardness measurements. However, it is proposed that the effects of aqueous environments (which may be produced catalytically at the crack tip) can be observed by their effects on the yield stress and the onset of cracking of the glass.

1. Introduction

Since the introduction of the pendulum hardness test by Kuznetsov [1] in 1929, little effort seems to have been made to understand the meaning of pendulum hardness values although it has generally been assumed that they are related in some way to the fracture behaviour of the test material. Indeed the material removal process in such a test is so similar to that in technologically important comminution processes such as abrasion, drilling and grinding of brittle materials Fig. 1 [2], that it might be supposed that this simple method could be used to study the mechanisms of these notoriously inefficient processes with a view to improving efficiencies. More than this, it has been reported [3, 4] that the test is sensitive to the effects of liquid environments, thus making it a potentially valuable method of monitoring the effectiveness of these environments in facilitating comminution.

Here we consider the interpretation of the pendulum hardness test with a view to assessing the method in the context of comminutive processes.

2. The nature of the test

In this test the attenuation in amplitude of a pendulum pivoted on the specimen, is measured. Since attenuation is large for "soft" materials i.e. those in which damage is extensive, the hardness is defined as [5]

$$H = -\left[\frac{1}{A_0}\left(\frac{A}{t}\right)_{t=0}\right]^{-1}$$

in which A_0 is the initial amplitude of the pendulum. This definition assumes an exponential decay of amplitude with time according to

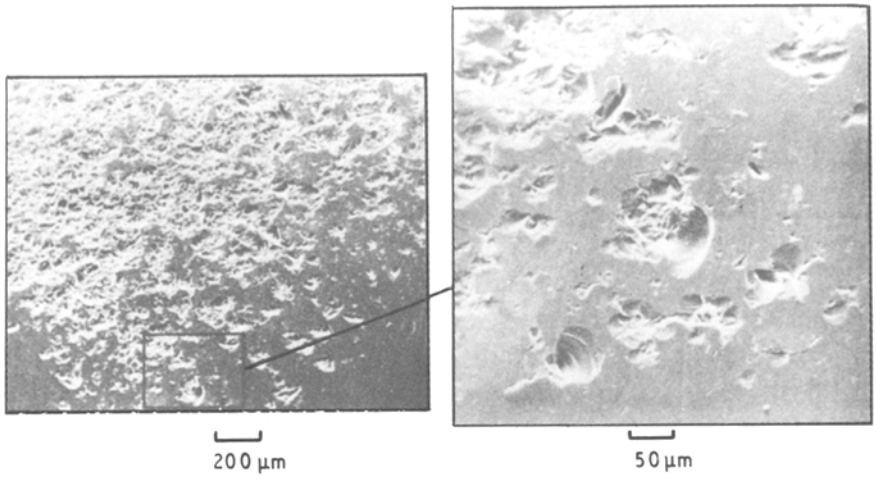
$$A = A_0 \exp(-t/H)$$

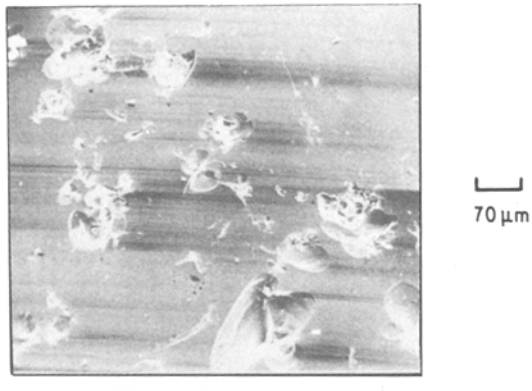
In practice, either the time for a fixed change in amplitude or the amplitude change in a fixed time is measured to determine H. Although the meaning of H is not understood, Terichow and Larsen [6] have shown a simple numerical relationship between it and the static hardness measured with a Vickers identer, for a variety of materials. In the present experiments, pendulum attenuation for 100 swings was typically from ~8.8° to 5.8° of arc for a pendulum of length 0.195 m and mass 0.342 kg. This corresponds to a total loss in energy of 4.3×10^{-3} J.

3. Mechanisms of energy dissipation

To account for energy dissipation in the pendulum hardness test, we propose to discuss the following five mechanisms for observations made on soda-lime silica glass. These are:

(1) fracture and the production of new surface area;





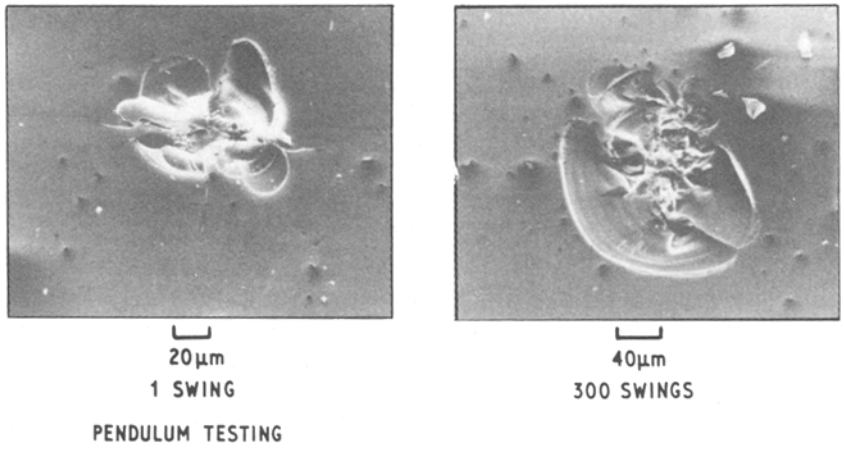


Figure 1 Comparison of the modes of material removal in the percussive drilling, slurry abrasion and dynamic hardness testing of soda-lime silica glass. Note the similarity in each case.

(2) plastic (viscous) flow of the solid under the pendulum pivot;

(3) the energy of viscous flow of the liquid environment/solid slurry produced at the pendulum pivot;

(4) the effect of electric charge on the viscous flow of the liquid through the debris;

(5) catalytic activity of freshly generated fracture surfaces.

Mechanisms 1 and perhaps 5 absorb energy as a direct consequence of the fracture process itself. Mechanism 1 was assumed by Rebinder and his co-workers to be responsible for the effects of certain liquids on the fracture mechanics of brittle materials through the reduction in surface energy resulting in a decrease in fracture stress (Rebinder effect). Mechanism 2 is favoured by Westwood [7] to explain the effect of liquid environments on dislocation mobilities and hence plastic flow in the near surface region of crystalline solids (chemomechanical effect). Mechanisms 3 to 5 have not so far been considered to contribute to energy dissipation in comminution processes in dynamic harness testing.

3.1. The formation of new surfaces

The energy consumption in the test must be at least that required to produce new surface area which, if the fracture is ideally brittle, is equal to the product of the surface energy and the total area produced. The first task, therefore, is the estimation of this new area.

Two types of behaviour are distinguishable in the damage area. The effect of initial loading is to produce a plastic indentation (as in static hardness testing) together with a crack extending from the tip of the pivot (indenter) into the solid. This is known as the median vent. No lateral chipping is observed until the load is removed, when the median vent closes and tensile stresses induced in the surface region of the solid by plastic deformation give rise to shallow chips which characterize the appearance of the area of contact, Fig. 2a.

A second feature which is immediately obvious in such tests is that considerable debris of small size is produced within the plastic indentation, Fig. 2b, and the amount of this material depends upon the liquid environment in which the test is performed. An extreme example of this is shown in Figs. 2c and d. The origin of this debris is not obvious and we must assume that sufficient stress intensification of the otherwise small tensile components of stress around a point indenter occur and may be induced by the presence of surface microflaws or frictional effects arising from the difference in elastic modulus between indenter and specimen [8]. Moreover, it seems that pendulum precession during the test enhances the production of this debris thus exaggerating the influence of the liquid environment on pendulum attenuation.

3.1.1. Estimation of total surface area generated during the test by lateral vent cracking

The new area of surface produced by lateral vent cracking was measured as a function of pendulum load for a large number of pendulum hardness tests. The results are shown in Fig. 3. The slope of the line yields a value of $3.2 \times 10^{-8}\,m^2\,kg^{-1}$ of pendulum for the area of new surface generated in this mode of cracking. Thus, using a value of surface energy of $4.0\,J\,m^{-2}$ [9] and pendulum weight of 0.34 kg, we obtain for the total energy dissipation by lateral vent cracking

$$E_{LV} = 2 \times 0.34 \times 3.2 \times 10^{-8} \times 4.0 \simeq 9 \times 10^{-8}\,J$$

3.1.2. Crushed debris

As already mentioned much of the damage produced by the pivot of the swinging pendulum is in the form of fine particles which were estimated by scanning electron microscopic examination to occupy approximately 10 to 20% ($\sim 5 \times 10^{-14}\,m^3$) of the total damage volume. Assuming these particles to be spherical and of diameter ~ 100 nm, then their total number, N, lies in the range

$$1.2 \times 10^7 \leqslant N \leqslant 2.4 \times 10^7$$

which would account for a total energy absorption of

$$1.5 \times 10^{-7} \leqslant E_{particles} \leqslant 3 \times 10^{-7}\,J$$

again assuming the value for fracture surface energy of $4.0\,J\,m^{-2}$.

3.2. Energy absorbed by viscous flow of the solid

In this section we consider that viscous flow or plastic deformation of the material under the pendulum pivot may absorb energy and attenuate the pendulum. For this calculation, we assume

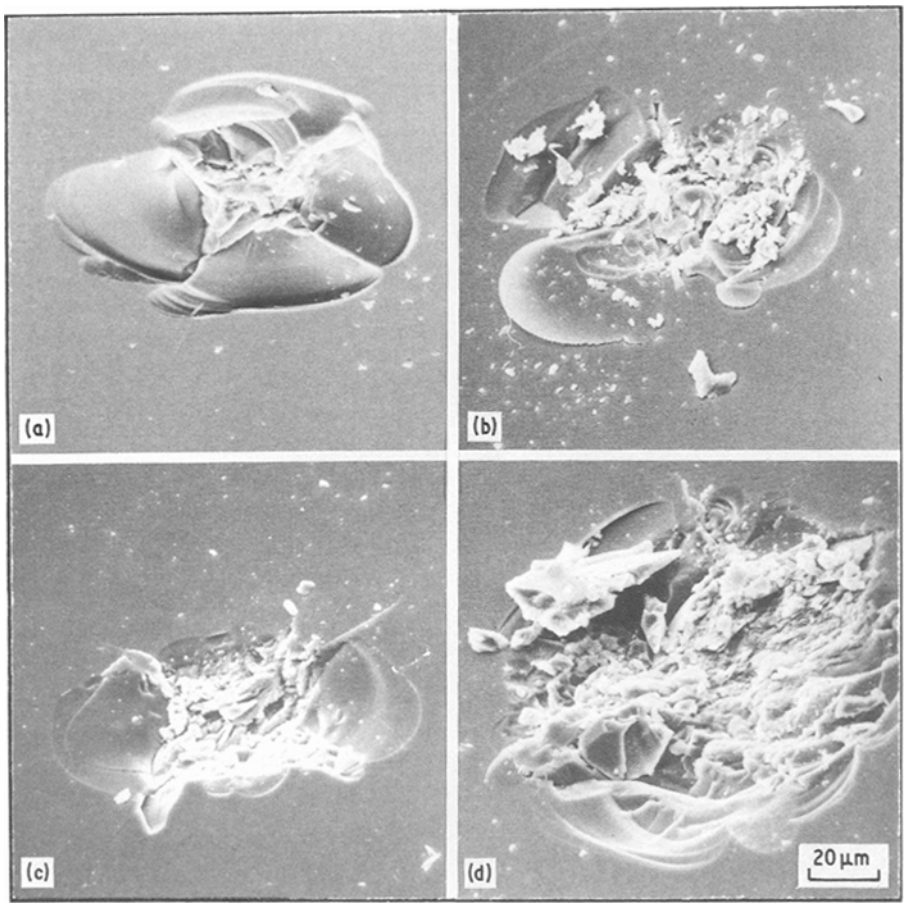


Figure 2 Detail of damage produced by the diamond pivot of the pendulum hardness tester. (a) Lateral chipping after removal of load. (b) Debris within the plastic indentation. Effect of (c) water and (d) phosphoric acid on the amount of debris produced in the pendulum test.

that the solid behaves as a viscous liquid which is squeezed and made to flow by the rocking action of the pendulum. The geometrical situation is shown in Fig. 4 and is complicated in practice by the shape of the indenter which for the Vickers pyramid rocking with the plane of a diagonal in the plane of the pendulum amounts to two surfaces inclined at an angle $\sim 150^\circ$ and shearing varying amounts of material depending upon the angle θ (and hence ϕ) and the instantaneous angular velocity of the pendulum $\dot{\theta}$. We anticipate that in this calculation we shall be concerned only with orders of magnitude estimates of energies so that we make the simplifying assumptions that (i) the average fluid velocity is half the maximum velocity and occurs at $\theta = 0$ in the plane of $(\phi/2)|_{\theta=0}$, (ii) the velocity vector is unchanged over the whole cycle of swing which will be true if θ is small and (iii) the average velocity over the area of contact of indenter and sheared layer in the specimen can be used in the calculation. The velocity varies from zero at the point of contact of the indenter and specimen, reaches its maximum value at the fluid–indenter interface in the direction $(\phi/2)|_{\theta=0}$, and is given by

$$v_i = \frac{r_i \dot{\theta}_{max}}{\sin \phi}$$

which for the pendulum used in the present experiments has the value

$$v_i = 1.6 \times 10^{-4} \text{ m sec}^{-1}$$

assuming $r_i = 25\,\mu\text{m}$ and $\dot{\theta}_{max} = 1.1 \text{ rad sec}^{-1}$.

The energy absorbed per swing of the pendulum can now be estimated as

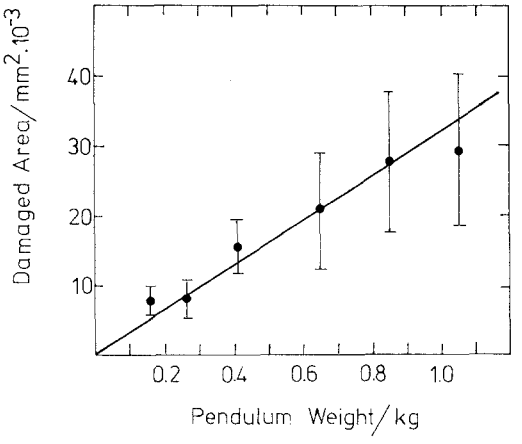


Figure 3 Dependence of absolute areas of damage in soda-lime glass on pendulum weight. Measurements were made from scanning electron micrographs of the specimen surface.

$$E_{vis} = f \times \tfrac{1}{2} r_i \phi = \tfrac{1}{4} \eta v_i \times \text{area}$$

in which f is the shear force per unit area. The area has a value $6.25 \times 10^{-10}\,m^2$, hence $E_{vis} = 2.5 \times 10^{-14}\,\eta$ J per swing. The value of η appropriate to these experiments is a matter for some conjecture. It has been shown by Schultze that sodium silicate glasses of composition $Na_2SiO_3\,3H_2O$ in the presence of sufficient water can have a viscosity $\sim 10^8\,N\,m^{-1}\,sec^{-1}$ [10] at room temperature giving the value 2.5×10^{-6} J for the energy pendulum hardness test of 100 swings. This value is much closer than that required for purely ductile fracture of the glass.

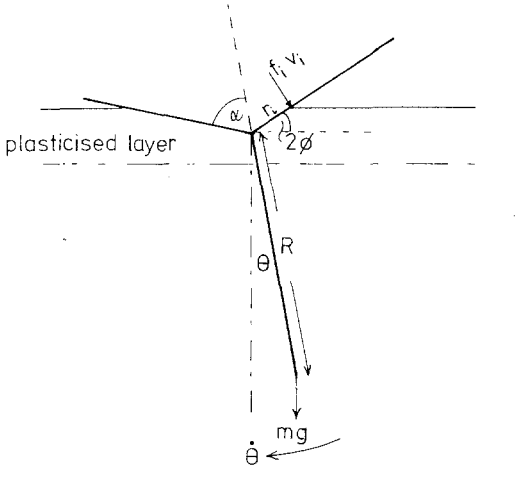


Figure 4 Pendulum geometry used in calculating the amount of work absorbed during plastic deformation of the surface region of the glass.

3.3. Energy absorbed by the viscous slurry of crushed debris

This mode of energy absorption would be applicable only in the presence of a liquid environment and results from the viscous flow of the liquid/debris slurry at the interface between pendulum pivot and specimen. The increase in viscosity due to a volume fraction of particulate debris in a slurry is given by [11]

$$\eta = \eta_0(1 + \tfrac{5}{2}c)$$

for spherical particles from which it can be seen that even for the situation in which the particles are most closely packed ($c = 0.74$) the viscosity and hence the energy absorbed are not increased by more than a factor of ~2 hence for liquids of moderate viscosity, this mode of energy absorption can make no significant contribution to pendulum damping. However, the effect of the build up of fine debris around the indenter is easily seen, Fig. 5, from which it is possible to draw the apparently erroneous conclusion that the presence of this material is important in pendulum damping.

3.4. Energy absorbed by electrokinetic effects in the porous bed formed by crushed debris at the pendulum pivot

Deraguin [12] showed that the viscosity of liquids appeared to increase if flowed through a porous bed of particles which are electrically

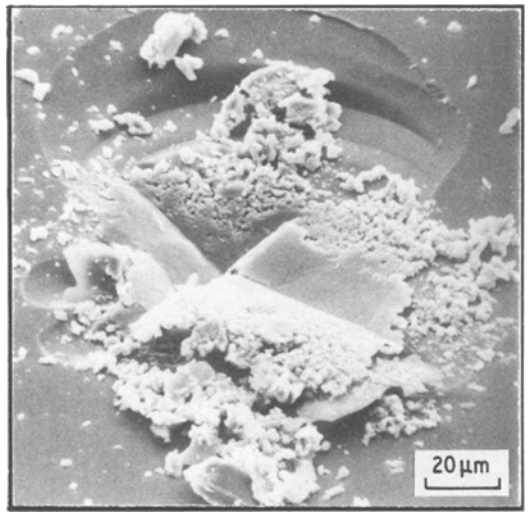


Figure 5 Build up of very fine compacted debris around the pivot of the pendulum.

charged. Since we expect the particles generated in neutral aqueous environments used in the pendulum hardness tests on glass to be negatively charged, the question arises as to whether this additional viscosity can account for an appreciable part of the total energy absorbed in pendulum damping. Levine *et al.* [13] have recently given the detailed theory of electroviscous flow through channels of width $2h$ for which the ratio of apparent to normal viscosity due to the electrical double layer of thickness $1/\kappa$ is written as

$$\frac{\eta_A}{\eta} = \left[1 - \frac{3c}{(1+H)}\rho^* \frac{(1-G)}{h}\right]^{-1}$$

in which $\rho^* = e_0/kT$, where e_0 is the proton charge and ρ the streaming potential. $G \simeq \tanh(\kappa h)/h$, and c and H are constants depending upon the temperature, nature and concentration of the electrolyte. Assuming constant channel spacings of ~ 50 nm for close packed spheres of debris of 100 nm diameter a change in viscosity of much less than 1% is calculated for ρ values up to 100 mV. Thus for liquids of moderate viscosity such as those used here (1 to 30 $N m^{-1} sec^{-1}$), electroviscous flow makes no significant contribution to pendulum attenuation.

3.5. Energy absorbed by catalytic activity

Newly formed fracture surfaces are known to be highly chemically reactive. Indeed, this reactivity is reflected in the tendency for gases and liquids to become firmly bound to the surface resulting in the lowering of surface free energy [14]. The energy exchange during adsorption will not itself contribute to the attenuation of the pendulum in a hardness test. However, if a catalysed reaction takes place at the freshly formed fracture surface, the products of the reaction may exert an environmental influence on the mechanical properties of the solid.

For example, silicate glass surfaces newly formed by abrasion have been shown to react with water and a variety of organic liquids resulting in hydroxylation of the silica network [15, 16].

$$-\overset{|}{\underset{|}{Si}}-O-\overset{|}{\underset{|}{Si}}- \xrightarrow{\text{grinding}} -\overset{|}{\underset{|}{Si}}-O^- {}^+O-\overset{|}{\underset{|}{Si}}- \xrightarrow{H_2O} 2-\overset{|}{\underset{|}{Si}}-OH$$

Moreover, it is also known that one of the effects of such a reaction is to lower the crack velocity by an amount which depends upon the activation energy, ΔE, of the chemical reaction [17]. In soda lime silica glass, ΔE changes from 605 kJ mol^{-1} for fracture in a vacuum to 109 kJ mol^{-1} in water. The effect of water in this case will be to plasticize the material at the fracture surfaces and its influence on pendulum attenuation will depend upon the availability of the environment at the crack tip and the rate of reaction there. No information is available on the kinetics of such catalysed reactions, although the maximum energy consumption by the catalytic modification of the crack tip structure cannot exceed that given in Section 3.2 in which it was assumed that water was freely available to reduce the viscosity (plasticity) of the glass.

4. Discussion

Table I summarizes the estimated energy losses by each of the mechanisms considered. These are to be compared with that observed experimentally. It is clear that only one of the mechanisms considered can account for the observed values and that this is associated with viscous flow of the glass. It follows therefore that although much debris is produced, the test in no way gives information which is directly relevant to the fracture processes which produced such debris, but, as in static hardness testing, is indicative of the plasticity of the solid. If on the other hand, an environment capable of modifying the plastic behaviour of the specimen is present, then this is reflected in the pendulum hardness value.

Recent discussions [18, 19] on the influence of *n*-aliphatic alcohol environments on the fracture behaviour of glasses has centred on the influence of water in the alcohols. As already indicated, since newly formed fracture surfaces of silicates are likely to be good dehydration catalysts for the alcohols, water is likely to be produced at the crack tip where it will exercise a plasticizing effect on the glass. The interpretation of the

TABLE I

Mechanism	Energy absorption (J)
Brittle fracture	1 to 3×10^{-7}
Plastic flow of solid	2.5×10^{-4}
Liquid slurry flow	v. small
Electrokinetic effects	negligible
Catalysis at crack tip	$\gg 2.5 \times 10^{-4}$

Energy lost by pendulum in typical test = 4.3×10^{-3} J.

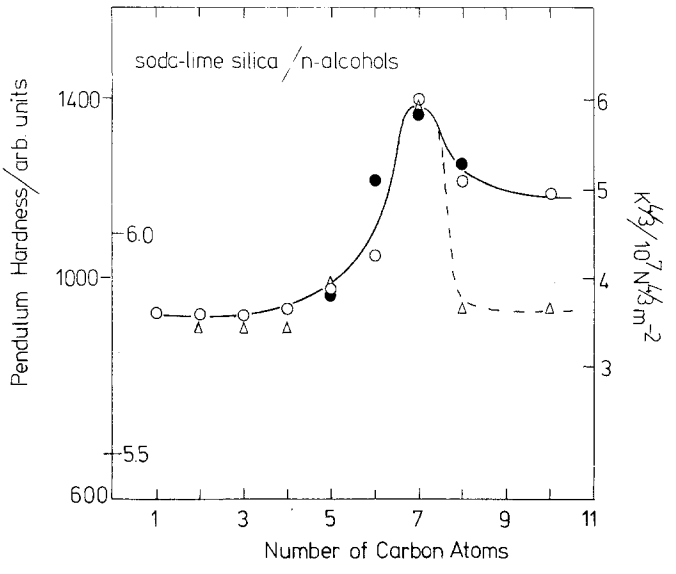


Figure 6 Variation of pendulum hardness with alcohol chain length for the primary alcohols on soda-lime glass. Notice the close reproducibility between the two independent sets of results. ○ – Westwood and Huntingdon. ● – Present data. △ represents the values predicted assuming that water either present in the alcohols or produced by the catalytic action of the new silica surfaces produced is responsible for the pendulum hardness variation shown.

seemingly anomalous variation of hardness with alcohol chain length, Fig. 6, can be understood from fracture mechanics studies reported by Freiman [20], who measured stress intensity as a function of crack velocity in several glasses. If one assumes that crack velocity is controlled by the amount of water at the crack tip, which in turn depends upon the alcohol present, then from Freiman's results relating relative humidities to alcohol chain lengths, corresponding stress intensity values can be deduced. These values are then related to the volume of material removed using an expression of Evans and Wilshaw [21] of the form

$$\text{Volume} \propto \left(\frac{1}{K}\right)^{3/4} \quad \text{for a fixed load}$$

where K is the stress intensity factor. If it is assumed that this volume is directly proportional to pendulum hardness, then the corresponding K values can be scaled to the pendulum hardness results shown in Fig. 6. The similarity between the curves suggests that catalytically produced water may be responsible for increased plastic flow at the crack tip.

The effect of load on pendulum hardness, Fig. 7, is more difficult to explain although similar effects have been observed in the static hardness testing of ceramic and polymeric materials. It is observed experimentally that the pendulum hardness reaches a maximum at a certain value of the load [2] and this depends upon the material [22]. If the glass is assumed to behave as an elastic/plastic material, then the effect of the indenter will be to induce increasing amounts of plastic flow until a critical load (stress) is reached at which fracturing occurs. Perrott [23] has shown recently that the limiting condition for the onset of lateral cracking in such a material is given by

$$\frac{H}{Y} - 1.155 > \frac{3F}{Y} > 0$$

where H is the static hardness of the material, Y the yield stress and F the fracture stress. On this basis the effects of environment would be expected to show up in modifying the yield stress. Marsh has shown [24] that the ratio H/Y has a value ~1.5 for many glasses compared with $H/Y = 3$ for ideally plastic materials. If the environment plasticizes the glass as suggested here for the influence of water, then the onset of cracking in the pendulum test should be controlled

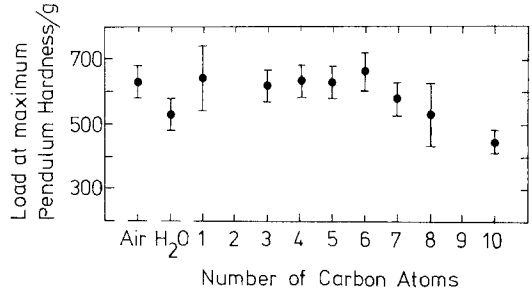


Figure 7 Variation of load at which maximum pendulum hardness is observed in a series of straight chain aliphatic alcohols.

by the variation of Y with water content of the glass and the amount of debris observed should be sensitive to environment (Figs. 2c and d).

5. Conclusions

From a consideration of several possible modes of energy absorption in the pendulum hardness testing of glass, it is concluded that the amplitude attenuation results from energy absorbed by the plastic deformation of the glass rather than its fracture. However, the influence of an environment on the yield stress of the material under test does make it possible to observe an environmental effect in soda-lime silica glass whose fracture properties are known to be sensitive to the water content of the glass surface.

Acknowledgement

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