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Investigation of the mechanical properties of silica glasses by indentation tests

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Abstract: Soda lime silica glasses were investigated by continuous indentation tests. The load indentation depth curves were taken during the loading as well as the unloading period by a computer controlled MTS machine. It was found that the loading force is a quadratic function of the indentation depth during both the loading and unloading stage of the deformation. The validity of the quadratic relationship in the case of the unloading stage seems to be characteristic only for glasses. Taking into account the elastic relaxation of the indentation depth an estimation is given for the size of the hydrostatic core which is necessary to symmetrize the stress field around the indenter. Using the measured length of the radial cracks started from the corners of the Vickers indentation pattern the $K_{IC}$ values were calculated.

1. Introduction

Various types of hardness tests are conventionally used for the determination of the mechanical properties of the materials. The response of solids to indentation provides important information about the elastic-plastic and fracture properties of materials. Although there are well established theoretical models [1-3] for the interpretation of the results of hardness tests and also for the evaluation of the indentation process due to the complexity of the stress response of the solids to the indentation, these processes cannot be treated in a unified way.

In the case of the brittle materials hardness data are for example in close connection with the Young modulus and from the size of the cracks arising around the indentation pattern an estimation of the fracture toughness can be given [4, 5]. However, conventional hardness measurements do not make possible to take into account the elastic recovery of the material, therefore in this case continuous indentation testing during which besides the residual deformation the instant deformation due to the applied load can be also registered are more informative [6]. By the use of continuous indentation measurements carried out with a Vickers type microhardness tester Fröhlich [7] introduced a new hardness number in the microhardness region.

In the present paper the results of continuous indentation tests performed in the macrohardness region are presented. From the load-penetration data an estimation for the size of the hydrostatic core around the indenter is given. It is also shown that in the macrohardness region there is a linear connection between the conventional hardness number and that of proposed by Fröhlich.

2. Experimtinals

The mechanical properties of soda lime silica glasses were investigated by special kind of Vickers-hardness test proposed by Fröhlich [7]. During the tests a Vickers pyramid was pressed into the surface of the glass sample by a computer controlled MTS machine. The measurements were carried out in the macrohardness...
region. Fig. 1 shows schematically the penetration program of the indenter. During the loading period the Vickers-pyramid is penetrated with constant velocity into the surface of the sample and the same velocity is applied in the unloading period when the pyramid moves backwards. In the course of the test the load was registered as a function of the penetration depth. The upper limit of the load was 100 N. The load - penetration depth curves (a typical one is shown in Fig. 2.) prove that in the course of the unloading period pronounced elastic recovery takes place.

3. Results and discussion
From the load-penetration function the indentation work and the energy relaxed during the elastic recovery can be calculated. The difference between the two energies is the plastic work performed during the deformation. The elastic-plastic properties of the materials can be characterized by the ratio of the elastic work to total work and the plastic work to total work, respectively. The former ratio was found to be 0.52 -0.6 for the glasses investigated. (On the contrary, this ratio is less than 0.3 for Al alloys.)

3.1 The estimation of the size of the hydrostatic core
There are both theoretical and experimental evidence that in materials where the elastic deformation is commensurable with the plastic one, the stress and strain field developing under the punch has a spherical symmetry independently from the shape of the indenter [8, 9]. Using this fact, the elastic-plastic deformation process taking place under the indenter are generally interpreted by slightly different versions of the expanded cavity model proposed originally by Hill [10]. According to the model the indentation pressure is transferred to the material investigated through a hydrostatic core where the pressure is equal to the indentation one. The hydrostatic core causes the observed symmetry of the stress field under the punch and it leads to the development of a hemispherical plastic zone around the indenter outside of which the material deforms elastically. However, the exact size and the physical meaning of the hydrostatic core is a crucial point of the model and regarded by some research workers in some sense to be nebulous [11]. Therefore there are various attempts for the more exact interpretation of it. Chiang et al. for example defined its volume to be equal to the residual volume of the indentation after removing the punch [11]. In the following it is shown that using some simple assumptions, a reasonable estimation can be obtained for the size of the hydrostatic core from the measured value of the energy relaxed during the elastic recovery. Let us consider the elastic field developing under the indenter during the loading and regard it as a Boussinesq one [12]. The stress field under the Vickers pyramid can be described as a Bussinesq one only outside of the small hydrostatic core of radius, $R$. Theoretically the Boussinesq stress field is produced by point-like loading. In this case the elastic stress decreases with the reciprocal of the square distance measured from the loading point and the density of the elastic energy in the material is given by
where $F$ is the applied load, $E$ is the elastic modulus of the glass and $r$ is the distance form the loading point. The elastic work, $W$ recovered in the unloading period can be considered in a first approximation to be equivalent to the energy of the elastic stress field integrated from the radius, $R$ to infinity. Comparing the calculated results with the value of the elastic work obtained experimentally, the radius of the core can be estimated. It was obtained that the size of it is equal to that of the Vickers pattern measured after removing the indenter.

The existence of a core with spherical symmetry can be supported by the investigation of the plastic zone below the indenter. The plastic deformation leads to the development of internal stress field in the plastic zone which can be observed in the case of glasses between crossed polar filters. Analysing the photoelastic stress field pattern it can be proved that the stress field has a spherical symmetry. It supports that the approximation of the stress field by that of a point like loading is a reasonable choice.

### 3.2 The analysis of the load-penetration relationship

According to Bernard [13] the load-penetration depth relationship can be described with a power series:

$$F = a_0 + a_1 h + a_2 h^2 + \ldots + a_n h^n$$

The number of the terms of the series was restricted to the second and third terms by Fröhlich who supported this restriction with physical argumentation [7]. Considering that the indentation work can be determined by the integral of the load with respect to the indentation depth and using the Fröhlich supposition for the load penetration function, the following equation can be obtained:

$$\int_0^h F dh = \frac{a_1}{2} h^2 + \frac{a_2}{3} h^3$$

On the right hand side of this equation the first term can be considered to represent the work which is necessary for the increase of the surface of the material. It is proportional to the square of the penetration depth, and depends on the surface tension of the material. The second term is due to the sum of the plastic and elastic work. This relationship between the load and the penetration depth can be verified by plotting $F/h$ against $h$. As it was expected linear curves shown in Fig. 3 were obtained. It can be seen, that the straight line representing the loading period starts from the origin. This means that the surface energy term in the Fröhlich's formula is negligible in the macro-hardness region. From the diagram the value of $a_2$ can be determined as the slope of the line obtained for the loading. As it was already mentioned this value can be used as a new hardness number which is characteristic for the materials with elastic-plastic properties. Plotting this new hardness number, $a_2$ against the conventional one, HV, a linear connection can be obtained between them (Fig. 4).

It is interesting that the unloading part of the load - indentation depth function gives also a straight line in $F/h$ versus $h$ representation. It seems to be characteristic only for glasses. (For the sake of comparison in Fig. 5, a load - penetration curve in $F/h$ versus $h$ representation is given for a metal sample. It can be seen that only the loading part of the curve can be approximated by a straight line.)
3.3 The fracture toughness

Using the penetration pattern remaining after removing the load and observing the cracks at the corner of the Vickers pattern the fracture toughness, $K_{IC}$, of the glass can be also assessed. Using the Anstis, Chantikul, Lawn, Marshall equation [14]:

$$K_{IC} = 0.016 \left( \frac{E}{HV} \right)^{\frac{1}{2}} F_c^{-\frac{3}{2}}$$

for the glass investigated this value was found to be $0.4 \cdot \text{MPa} \cdot \text{m}^{-\frac{1}{2}}$, where $E$ is the Young modulus, $HV$ is the Vickers hardness, $F$ is the maximum load and $c$ is the length of the radial cracks. Fig. 6 shows the SEM picture of the cracks of the indentation pattern with cracks starting from the corners of the pattern.

4. Conclusions

1. Using energetic argumentation an estimation for the size of the hydrostatic core arising around the indenter was made. It was found to be approximately equal to the radius of the indenter.

2. It was shown experimentally that the load-indentation depth relationship can be described in the macrohardness region by a power law function. Using this a new hardness number can be introduced for the characterization of the elastic-plastic properties of the glasses. However, this new parameter based on the whole indentation process is in linear connection with the conventional hardness number.

3. The experimental data proves that the load-indentation depth function, using an $F/h$ versus $h$ representation gives a straight line equally in the case of glasses and metallic materials for the loading stage of the deformation. On the contrary for the unloading period it seems to be characteristic only in the case of glasses.

Fig. 4. HV versus $a_2$ plot

Fig. 5. $F/h$ - $h$ plot obtained for a metallic sample

Fig. 6. SEM picture of the cracks
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5. References