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Characterization of Glasses and Ceramics by Continuous Indentation Tests

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Abstract:Continuous indentation tests were performed on soda lime silica glasses and tetragonal zirconia polycrystal ceramic samples containing 10-14 mol% CeO_2 (Ce-TZP ceramics) to determine their Vickers hardness (HV) and fracture toughness (K_{IC}). 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. Using the HV values and the measured length of the cracks the K_{IC} values were calculated. The fracture toughness of Ce-TZP ceramic samples decreases with increasing CeO_2 content as a consequence of the reduced transformability of the tetragonal phase.

1. Introduction

Various types of hardness tests are used for the determination of the mechanical properties of the materials. During the conventional Vickers hardness test a hard, sharp indenter (Vickers pyramid) is pressed into the surface of the sample with constant normal force. The diamond pyramid is a sharp indenter, therefore the pressure below the pyramid is large enough to deform the material plastically. This is the reason for leaving a residual square-shaped impression after unloading.

The Vickers hardness (HV) is calculated from the following formula:

$$HV = \frac{F}{A} = 1.8544 \cdot \frac{F}{d^2}, \tag{1}$$

where F is the applied load, A is the area of the produced indentation after unloading and d is the diagonal of the indentation pattern.

In the case of brittle materials hardness data and the size of the cracks arising around the indentation pattern can be used for the determination of the fracture toughness [1]. Continuous indentation tests are more informative than conventional hardness tests because the elastic recovery can also be investigated [2,3].

In the present paper the results of continuous indentation tests performed on silica glasses and Ce-TZP ceramics are presented.

2. Experimental

Continuous indentation tests were performed on soda lime silica glasses and tetragonal zirconia polycrystal ceramic samples containing 10-14 mol% CeO_2. During the test a Vickers pyramid is pressed into the previously mechanically polished surface of the sample by a computer controlled MTS machine. The measurements were carried out in the macrohardness region at room temperature.

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. The velocity of the indenter and the applied load can be varied in a relatively wide range. In the course of the test the load is registered as a function of the penetration depth. The load - penetration depth curves (a typical one is shown in Fig.2) prove that in the course of the unloading period elastic recovery takes place.

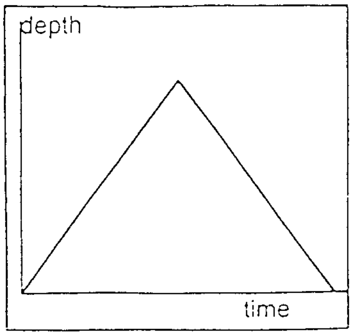

Fig.1. Penetration program of the indenter

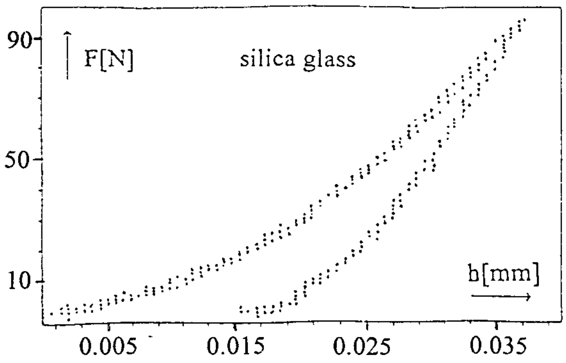

Fig.2. A typical load-penetration depth curve

The fracture toughness can be determined in terms of the Vickers hardness, the length of the cracks arising at the corners of the Vickers pattern and some material parameters. However, the exact form of this relationship depends on the type of the crack initiated [1]. In the glasses and ceramics investigated half-penny and Palmquist cracks were observed, respectively.

3. Results and discussion

According to Bernhardt [4] the load - penetration depth (h) relationship can be described by a power series. 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 [3]:

$$F = a_1 h + a_2 h^2 . \tag{2}$$

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 depth function, the following equation can be obtained:

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

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. The second term can be attributed 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 were obtained in the loading period (Fig.3). It is interesting that the unloading part of the load - indentation depth function gives also a straight line in F/h versus h representation. Taking into account the geometrical relation between the indentation diagonal and depth, d=7h, the Vickers hardness can be obtained from the following formula:

$$HV = 1.8544 \cdot \frac{F}{49 \cdot h^2} . \tag{4}$$

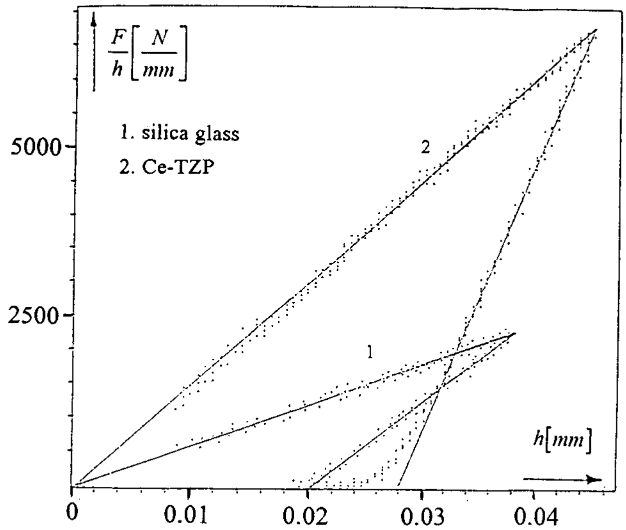

Fig.3. The F/h-h representation of the load-indentation function

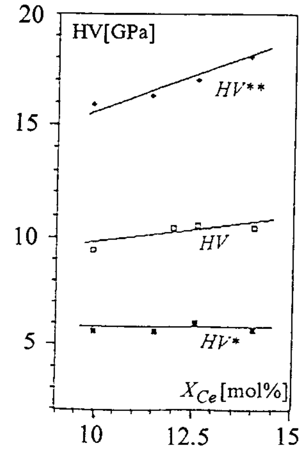

Fig.4. Hardness versus ceria content

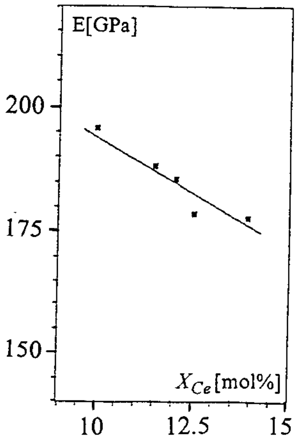

Fig.5. Young's modulus versus ceria content

Two different characteristic values of the indentation depth can be obtained during the continuous test. One of them can be measured at the maximum load, the other one is the depth measured after unloading. Vickers hardness HV* calculated from the maximum load and the maximum indentation depth characterizes the elastic-plastic properties of the materials. The other hardness number HV** calculated from the relaxed indentation depth and the maximum load is characteristic only for the plastic properties of the sample [5].

In Fig.4 the three different hardness values obtained on Ce-TZP ceramics are shown as a function of CeO_2 content (The HV is the optically measured conventional hardness number.). According to our measurements HV** is higher than the conventional hardness number because the optical observation of the Vickers pattern yields somewhat larger indentation diameters in consequence of the distortions around the edges and the corners. HV* which involves both the elastic and the plastic response of the material to the indentation is almost constant. HV** characterizes only the plastic properties of the samples and it is increasing with CeO_2 content. The deviation of the two curves can be attributed to the variation of the elasticity of the samples. Fig.5 shows the Young's modulus obtained by four point bending tests versus the CeO_2 content.

The fracture toughness can be determined by the following formulas for half-penny cracks (in glasses) and Palmquist cracks (in ceramics), respectively:

$$K_{IC} = 0.016\left(\frac{E}{HV}\right)^{\frac{1}{2}} F\, c^{-\frac{3}{2}} \qquad (5)$$

$$K_{IC} = 0.018\left(\frac{2l}{d}\right)^{-\frac{1}{2}} HV \left(\frac{d}{2}\right)^{\frac{1}{2}} \left(\frac{E}{HV}\right)^{\frac{2}{5}}, \qquad (6)$$

where E is the Young's modulus, HV is the Vickers hardness, c is the radius of the half-penny crack and l is the length of the Palmquist crack. The best agreement with the K_{IC} values determined from four point bending tests is obtained if HV** is used as hardness number [5].

For the fracture toughness of soda lime silica glass 0.4 $MPam^{1/2}$ was obtained.

The ceramics containing tetragonal ZrO_2 show high toughness (about 10 $MPam^{1/2}$) because the stress-induced martensitic tetragonal-monoclinic phase transformation at the crack tip makes the material resistant to crack propagation. The crack propagation is caused by the local tensile stress at the crack tip. At the critical stress value the tetragonal ZrO_2 grains near to the crack tip transform to monoclinic structure. The volume-growth (3-5%) occuring in the transformation introduces compressive internal stresses in the neighbourhood of the transformed grains. The compressive stress reduces the applied tensile stress at the crack tip and impedes the propagation of the crack.

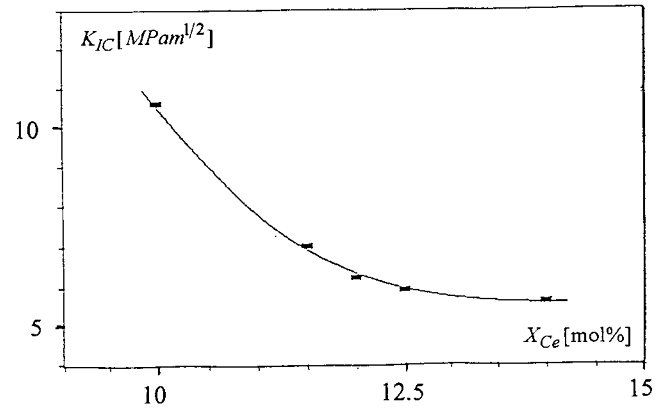

Fig.6. The variation of toughness of Ce-TZP ceramics

Fig.6 shows the variation of the fracture toughness of Ce-TZP ceramics versus CeO_2 content. In the ZrO_2 ceramics CeO_2 stabilizes the metastable tetragonal phase at room temperature. The fracture toughness of the Ce-TZP ceramics decreases with the increase of its CeO_2 content as a consequence of the decreased transformability of the tetragonal phase.

4. Conclusions

-It was shown experimentally that the load-indentation depth relationship can be described in the macrohardness region by a power series containing two-terms. The experimental data prove that the load-indentation depth function using an F/h versus h representation gives straight lines in both the loading and unloading part of the indentation process.

-The fracture toughness of the Ce-TZP ceramics decreases with increasing CeO_2 content as a consequence of the reduced transformability of the tetragonal phase.

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