INFLUENCE OF INDENTATION RESIDUAL STRESSES ON THE STRENGTH OF CERAMICS

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Abstract. The purpose of this paper is to study the ratio between the bend strengths of specimens with "as indented" and "as annealed" indentation Vickers cracks. This ratio is usually found larger that the value expected in an inert environment and it is often associated to a decrease in the level of indentation residual stresses. This point has been addressed here by studying a 99,7% purity alumina with a dispersion of 5% in volume of zirconia stabilised with 3% molar of yttria. It is shown that environmental crack growth after indentation has an important contribution to reduce the level of indentation residual stresses but it can only explain partly the results. Then, it would be necessary to consider another explanation to this discrepancy, which could be the interaction between applied and residual indentation stress field.

Resumen. En este trabajo se estudia la relación entre las resistencias a flexión de probetas con grietas de indentación Vickers, con y sin tratamiento térmico de recocido. Generalmente se ha encontrado que esta relación es mayor que el valor esperado en un ambiente inerte, lo cual ha estado asociado frecuentemente a una reducción de las tensiones residuales de indentación. Este punto ha sido tratado aquí a través del estudio de alumina con 99.7% de pureza con una dispersión de 5% en volumen de circona estabilizada con 3% molar de itria. El crecimiento de grieta por efecto del ambiente tiene una importante contribución a la reducción de las tensiones residuales de indentación. Sin embargo, esto solo explica parcialmente los resultados. En consecuencia, sería necesario considerar otra posible explicación para esta diferencia, que podría ser la interacción entre el campo de tensiones aplicado y el campo de tensiones residuales de indentación.

1. INTRODUCTION

Indentation methods are widely used in materials science in the study of the mechanical behaviour of ceramics, such as strength and fracture toughness. The contact of a rigid indenter, like a sharp diamond pyramid, on a surface of a ceramic material induces a local irreversible deformation and the appearance of cracks at the imprint corners. On unloading these cracks propagate as a result of the local residual stress field that originates from the mismatch in the elastic and plastic deformation beneath the contact.

Since indentation cracks can be produced very easily and can be located at any place on the surface of a specimen, they are well suited to study fracture and fatigue behaviour, such as the sensitivity of the strength to the presence of relatively small cracks. For this purpose, a number of indentation cracks are located on the tensile face of a four-point bend specimen and perpendicularly to the tensile stress. The strength can be determined in terms of different cracks lengths or other variables that can influence the strength such as rate of stressing, environment, surface residual stresses induced by machining, surface finish, etc.

In particular, indentation residual stresses have a large effect not only in the measured strength, but also in the fact that it makes crack extension stable in bending until the crack reaches a critical length that can be more than twice the original one. From the well-established model of Lawn et al [1] for residual indentation stresses, it comes out that the ratio between the bend strengths of specimens with "as indented" and "as annealed" cracks in an inert environment should be constant and equal to 0.47 for any material. However, it is usually found that this ratio is higher, being very often close to 0.7, either in an inert environment or also in humid air when a high stress rate is used. Then, even in humid air this ratio is hardly different from that corresponding to an inert environment, as reported as early as in the results of Marshall and Lawn [2] for Vickers-indented soda-lime glass in water.

The purpose of this paper is to address this discrepancy in zirconia toughened alumina (ZTA). This will be pursuit by first considering the reduction in the level of indentation residual stresses induced by environmental crack growth after indentation. However, it will be shown that environmental crack growth alone can not explain the results obtained in ZTA. Finally, it will be proposed that the ratio between both bend strengths referred above could be explained by taking into account the interaction between the indentation residual stress and the applied stress.

2. EXPERIMENTAL PROCEDURE

The material used in this investigation was 99.7% purity alumina with a dispersion of 5% in volume of zirconia stabilised with 3% molar of yttria, that shall be referred to as ZTA 5%. Details about material preparation can be found elsewhere [3]. The grain size (spherical diameter) of each phase was evaluated with an image analyser, using scanning electron microscopy (SEM). The zirconia is well distributed in the matrix, primarily at three-point matrix junctions (Fig.1). The average grain sizes of alumina and of zirconia are 1.46 and 0.42 μm respectively.

Bar shaped specimens (dimensions 45x5x8 mm) were cut from the plates and were polished to a mirror-like finish on the upper surface using diamond pastes of 30, 6 and 3 μm. Some specimens were subsequently heat treated at 1200 °C for 20 minutes in order to assess if residual stresses had been introduced by the operations of grinding and polishing. The indentations were performed by a Vickers indenter with loads of 98.1, 196.2 and 294.3 N during 10 seconds at the central region of the tensile face of polished specimens. Five indentations were made with different loads in order to study the growth of cracks during monotonic flexure loading of the indented specimen. Special care was taken in order to obtain indentations cracks well aligned with the specimens edges so that one pair of indentation cracks was perpendicular to the specimen central axis with a maximum angular error of $\pm 3^{o}$.

A fully articulating four-point bend test fixture was used for all tests. The fracture toughness was determined by the indentation method, both in air and in silicone oil, and by the indentation strength bending (ISB) method [4-5]. The fracture strength was measured for specimens containing natural flaws as well as for those indented. These tests were conducted in an electro-mechanical machine Instron 8562 using a loading rate of 200 N/s. The crack length was measured during monotonic loading and plotted in terms of the applied stress.

In a previous study [3] it was shown that the fracture toughness increases only moderately with increasing zirconia content. One of the reasons may lie in the fact that X-ray diffraction measurements revealed that in a ZTA containing larger amounts of zirconia than in the present study, at the end only ~5% of the available zirconia transformed to monoclinic symmetry during fracture. This very limited amount of transformation was believed to be the main reason for the generally small increase in fracture toughness with increasing zirconia content.

The strength is greatly influenced by the void size, and since this it is related to grain size, the addition of zirconia resulted in considerable increase in strength compared to pure alumina. Therefore, the main effect of the addition of zirconia to alumina in the composite studied here is the reduction of the grain size of the alumina matrix, and consequently the reduction of the size of the flaw population.

The values of K_{Ic} measured by indentation were obtained by using the equation proposed by Anstis et al [4],

$$K_{Ic} = A\left(\frac{E}{H}\right)^{m}\left(\frac{P}{c_{inert}^{3/2}}\right) = \chi_0\left(\frac{P}{c_{inert}^{3/2}}\right) \qquad (1)$$

where P is the indentation load, A and m are constants for a given material (for the ZTA studied, in a previous work [6] it has been found that A=0.024 and m=0.5, E is the Young´s modulus, H the Vickers hardness, usually defined as $H=P/2d^2$,where d is the indentation half-diagonal) and c_{inert} is the crack length.

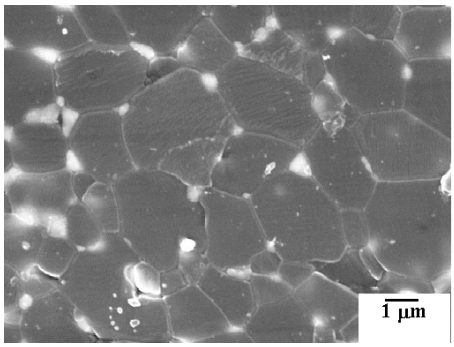


Fig. 5. SEM micrograph of ZTA5% microstructure.

3. EXPERIMENTAL RESULTS AND DISCUSSION

The results for hardness, fracture toughness measured by the indentation method and fracture strengths of "as indented" as well of "as annealed" specimens, are depicted in Table 1. In addition, previous results [3] on static fatigue crack growth exponent, n, is also indicated.

Table 1. Mechanical parameters for ZTA5%

HV15 **(GPa)**	K_{Ic}^{air} **(MPa·m$^{1/2}$)**	K_{Ic} **(MPa·m$^{1/2}$)**	$\sigma_0^{indented}$ **(MPa)**	$\sigma_0^{annealed}$ **(MPa)**	***n***
17,7	3,3	5,01	120	180	30

The fracture toughness measured under inert conditions, K_{Ic}, was always larger than in laboratory air, K_{Ic}^{air}. In fact, the latter is much closer to the threshold for subcritical crack growth than to the actual fracture toughness.

The values of K_{Ic}^{air} that were measured in polished specimens were consistently and sligthly larger than for specimens polished and annealed at 1200 °C. Because of this small effect all the specimens were annealed after final polishing.

Vickers indentation was used in order to form a crack on the tensile surface of four-point bend specimens for studying the growth of cracks under monotonic loading conditions. This configuration was also used to determine the fracture toughness, either with residual stresses or after a suitable heat treatment that relieves the residual stresses generated around the imprint. In the last case, no stable subcritical crack growth was detected before fracture.

From observations by SEM of the fracture surfaces it was noticed that the eccentricity of the crack was very close to 1, so that it will be assumed that the crack has a semicircular shape.

The crack length (c) measured in monotonic bending tests are given in Fig.2. The same data is represented in Fig. 3 where the maximum applied stress in flexure multiplied by c^2 has been plotted in terms of $c^{3/2}$. From the slope of the straight line, K_{Ic}^{air} can be obtained by using a value for ψ given by Newman and Ragu [6] for semicircular cracks.

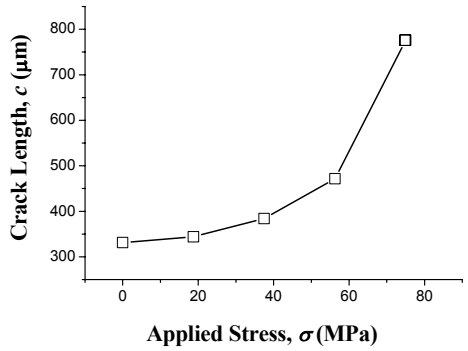


Fig. 2. *In Situ* measured crack size as a function of applied stress for 196 N indentation load.

3.1. Indentation residual stresses

When indentation cracks are of radial type and with a semicircular shape, the stress intensity factor (SIF) caused by the indentation residual stresses, K_r, is assumed to be proportional to $c^{-3/2}$. Then, under inert conditions fracture toughness can be obtained from equation (1).

From the measurement of indentation crack length in humid air, in fact an apparent fracture toughness, K_{Ic}^{air}, is obtained and whose value shall be a function of the time elapsed after indentation. It is equal to the stress intensity factor (SIF) induced by the residual stresses and it can be written as,

$$K_r(t) = K_{Ic}^{air} = K_{Ic}\left(\frac{c_{inert}}{c}\right)^{3/2} \qquad (2)$$

If all testing is done in air, it is well known that ceramics are susceptible to enhanced crack growth in the presence of certain chemical environments, mainly water. Then crack growth in air might occur under the combined action of residual stresses and water molecules present in the air. The dependence between crack velocity, dc/dt and SIF, K, is usually represented by a power law with a very high exponent, n, that is,

$$\frac{dc}{dt} = v_0\left(\frac{K}{K_{Ic}}\right)^n \qquad (3)$$

where v_0 is a constant. This equation is obeyed in stage

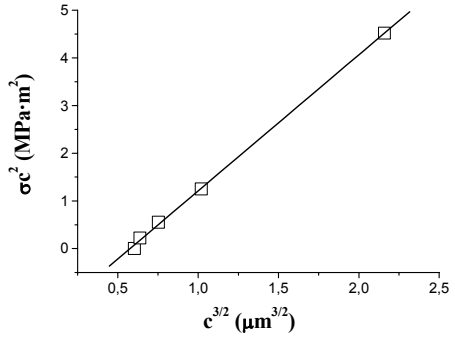


Fig. 3. Typical plot of σc^2 vs. $c^{3/2}$ giving K_{Ic}/ψ as the slope of the plot.

I of subcritical crack growth, but it is not clear whether the same equation reproduces the actual behaviour for K close to K_{Ic}. Nevertheless, since no data is available from direct crack growth measurements in this range, it shall be assumed that equation (3) is satisfied in all the range of K studied.

Accordingly, crack velocity may be very high at SIF´s very close to K_{Ic}, and it decreases very rapidly as soon as K is lower than K_{Ic}. The crack stops when the threshold value of K for crack propagation, K_{Ith}, is reached. Then if the measurement is carried out some time after indentation, the fracture toughness measured in air, K_{Ic}^{air}, may be rather closer to K_{Ith} than to K_{Ic} . Fig. 4 shows the crack length as a function of time after indentation in air. It can be noticed that the crack extension that takes place after indentation is far from negligible.

3.2. Crack extension after indentation in air

In laboratory air, the crack length increases when removing the indentation load and its final length, c_0, is larger than c_{inert}. In fact, by integration of equation (3) with $K=Kr$, it can be shown that, after indentation in air, the time spent in reaching K_{th}, assuming that n>>1, is given by,

$$t_{th} \approx \frac{1}{3n+1}\left(\frac{2c_{inert}}{v_{th}}\right) \quad (4)$$

where

$$v_{th} = v_0\left(\frac{K_{th}}{K_{Ic}}\right)^n \quad (5)$$

In principle, from equation (4) it would be possible to estimate the time during which an indentation crack extends after indentation before stopping in an given humid air environment. However, the results are very sensitive to the values selected for v_0 and n. Defining the threshold for crack growth as the K value that produces a crack velocity, v_{th}, equal or smaller than

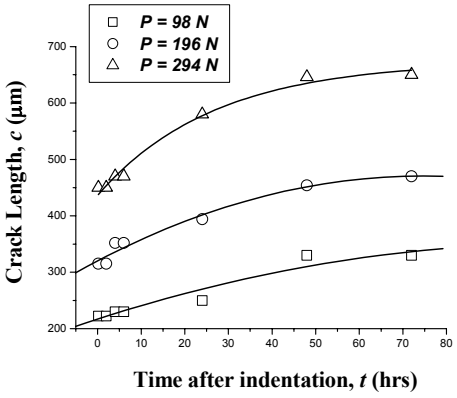


Fig. 4. Indentation crack length as a function of time in air.

10^{-11} m/s, n = 30, c_{inert}=163 μm and using equation (4) we obtain $t_{th} \approx$ 100 hours. Then, it should be possible to detect crack extension in air in the optical microscope if the observation is carried out after a relatively short time just after taking the indentation, confirming the results of Fig. 4.

3.3. Ratio of strengths

Now, it is examined the expected ratio between flexure strengths of bars with indentation cracks, but with and without indentation residual stresses.

If a bar indented in air is annealed so that the indentation residual stresses are removed, the strength of the bar in an inert environment, or in air at a high stress rate so that slow crack growth during the test can be neglected, can be estimated by:

$$\sigma_0^{annealed} = \frac{K_{Ic}}{Y\sqrt{c_0}} \quad (6)$$

If a bar "as indented" is subjected to flexure in air some time after indentation, the initial crack length for bending is c_0 with $c_0 > c_{inert}$, and the corresponding residual indentation SIF during bending is given by,

$$K_r = K_{Ic}^{air}\left(\frac{c_0}{c}\right)^{3/2} \quad (7)$$

Therefore, for those ceramic materials with slow crack growth in humid air, the initial indentation residual SIF in a cracked four-point bending specimen is lower, by a factor μ, with respect to the level that would had reached in an inert environment. If the initial crack length is c_0, the indentation residual SIF can be written as,

$$K_r = \mu K_{Ic}\left(\frac{c_0}{c}\right)^{3/2} \quad (8)$$

where

$$\mu = \left(\frac{c_{inert}}{c_0}\right)^{3/2} \quad (9)$$

If enough time has elapsed between indentation and flexure bending in air, we may have that μK_{Ic} has reached K_{Ith}, but, in general,

$$\mu K_{Ic} \geq K_{Ith} \quad (10)$$

In a bending test the total SFI, K_t, is given by the sum of the residual SIF (equation 8) and the applied SIF, K_a,

$$K_a = K_a^0\left(\frac{c}{c_0}\right)^{1/2} \quad (11)$$

where K_a^0 is given by

$$K_a^0 = Y\sigma\sqrt{c_0} \quad (12)$$

Then, the ratio between the total SIF and K_{Ic} is given by:

$$\frac{K_t}{K_{Ic}} = \frac{\mu}{z^3} + R_0 z \quad (13)$$

where:

$$z = \sqrt{\frac{c}{c_0}} \quad (14)$$

and

$$R_0 = \frac{K_a^0}{K_{Ic}} = \frac{\sigma^{indented}}{\sigma_0^{annealed}} \quad (15)$$

is equal to the ratio between the applied stress in "as

indented" specimens and the strength of "as annealed" specimens.

By imposing the conditions for unstable fracture, that is,

$$\left(\frac{K_a}{K_{Ic}}\right)=1 \qquad \left(\frac{\partial\left(K_t/K_{Ic}\right)}{\partial z}\right)=0 \qquad (16)$$

the critical values of R_0, $(R_0)_{crit}$, and z, z_{crit}, can be obtained. $(R_0)_{crit}$ is equal to the ratio between the strengths of as indented and annealed specimens. Then, by applying the conditions given by equation (16) to equation (13), we obtain

$$(R_0)_{crit}=\frac{(K_a^0)_{crit}}{K_{Ic}}=\frac{\sigma_0^{indented}}{\sigma_0^{annealed}}=\frac{3\mu}{z_{crit}^4} \qquad (17)$$

and

$$z_{crit}=4^{1/3}\mu^{1/3} \qquad (18)$$

where $\sigma_0^{indented}$ is the flexure strength of the as indented specimen. Notice that when no reduction in indentation residual stresses occurs, $\mu = 1$, and $(R_0)_{crit}$ is equal to 0.47, while z_{crit} reaches 1.587, that is, the critical value of c is equal to $2.52c_0$.

In a first step, it would be reasonable to associate the difference between the theoretical and experimental strength ratio for as indented and annealed specimens to the enhanced crack growth that takes place in air during and after indentation and also during testing in flexure.

It should be noticed that the presence of air in the stable crack growth during flexure could increase the value of z_{crit}, but it would not hardly have any effect on the ratio between the strength of "as indented" and "as annealed" specimens. This is supported by the calculation and experimental results of Lawn on dynamic fatigue in glass (Fig. 3 of reference [4]), where it is shown that for usual high stress rates, the ratio in strength between "as indented" and annealed specimens is nearly constant and very similar to that obtained in an inert environment. This is justified by the weaker effect of the environment at high stressing rates, so that the ratio between strengths shall not be significantly affected by the presence of humidity. It has also been verified here by using specimens indented and tested in silicone oil and comparing the results with those for specimens with cracks produced by indentation in air and tested also in air. Since the strength was very similar in both cases, it is assumed that, at the loading rate used in this work, the effect of environment in the strength is negligible

Since from the experiment the strength ratio given in equation (17) is found to be close to 0.70, the value of μ to account for this would be about 0.30. It would mean that the initial maximum residual SIF of indentation cracks in air is much smaller than under an inert environment. However, values as low as 0.3 are incompatible with some observations, which can be summarised as follows.

Firstly, ZTA ceramics, as well as many other oxide ceramics like alumina, have static fatigue stress intensity thresholds in laboratory air environment, which are usually higher than $0.5K_{IC}$. For example, in the case of ZTA5%, K_{Ith} is about 60% K_{Ic}, so that no enhanced slow crack growth may take place at values of K below $0.6K_{Ic}$. Then the smaller possible value of μ compatible with this fact should be at least larger than 0.6.

Secondly, although indentation cracks do not extend noticeably immediately upon application of the stress in bending, which is consistent with some reduction in K_r, the applied SIF needed to observe crack growth in the present experiments is only about $0.2K_{Ic}$. This means that the initial residual SIF should be already larger than about $0.4K_{Ic}$ in order to reach, together with the applied SIF in bending, the threshold value, that is, $0.6K_{Ic}$. Then μ should be at least higher than 0.4.

Thirdly, if the residual SIF after indentation dropped to $0.30K_{Ic}$, indentation cracks should extend after indentation much further in air than under inert conditions. Actually it would mean that c_0 should be about $2.2c_{inert}$. Such large differences between indentation cracks lengths in air and in inert atmospheres are not observed in most oxide ceramics.

Finally, another reason in support of the above argument is that if μ =0.3, no significant stable crack growth should occur during bending, which is also contrary to observations. In fact it can be shown that stable crack growth only can take place for $\mu > 0.25$. In the present case, the extension of crack during four point bending was still significant, about 44 % of its initial length.

Therefore, it is concluded that in order to explain the results it appears that a reduction of residual indentation stresses because of environmental crack growth does not give a plausible explanation to the experimental observations. In other words, in order to explain the results it is not possible to base the arguments in only a reduction in the value of μ. .

3.4. Interaction between applied and residual stress

The ratio between the strength of "as indented" and "as annealed" specimens could be calculated taking into consideration the influence of the applied stress on the residual stress by adopting a model developed by Fett [8]. In this model, the residual stresses are reduced

considerably if an applied stress is superimposed on the residual stress field produced by the indentation. In other words, the plastic deformation zone that is in a state of compression and maintains the crack open can expand freely as soon as load is applied and hence the residual stresses are reduced. The effect of this interaction can then account for the observations and it is the subject of future work.

4. CONCLUSIONS

The ratio between the bend strengths of specimens with "as indented" and "as annealed" indentation Vickers cracks has been studied in a 99,7% purity alumina with a dispersion of 5% in volume stabilised with 3% molar of yttria. This ratio is found to be larger than the value expected in an inert environment

It is shown that a reduction of residual indentation stresses because of environmental crack growth can not explain the experimental observations. Then, it is proposed that a reduction of indentation residual stresses because of the interaction between applied and residual indentation stress field could play an important role in order to explain the results.

5. ACKNOWLEDGEMENTS

The authors gratefully acknowledge the Spanish MCYT for its financial support under grant No. MAT-99-0781 and to the Generalitat de Catalunya under DURSI grant No.2001SGR-00229.

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