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Duplex Ceramics: II. Strength and Toughness

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Abstract

Strength and toughness of various sintered and HiPed duplex ceramics are investigated. It is found that the strength is reduced and the toughness increased compared with the respective matrix data. The transformability of the ZrO_2 particles in the incorporated zones is examined. The retained strength of duplex ceramics after a mechanically introduced surface damage, i.e. their mechanical damage resistance, is improved when compared to the pure matrix. An attempt is made to relate normalized strength, normalized toughness, and mechanical damage resistance of duplex ceramics to an internal stress intensity factor, K_i, generated around the pressure zone.

Verschiedene gesinterte und heißisostatisch gepresste Duplex-Keramiken werden auf ihre Festigkeit und Bruchzähigkeit hin unterscht. Durch die Einlagerung von Druckzonen nimmt die Festigkeit zwar ab, die Bruchzähigkeit wird hingegen verbessert. Das Umwandlungsverhalten der ZrO_2-Teilchen in den eingelagerten Zonen wird untersucht. Die Widerstandsfähigkeit von Duplex-Keramiken gegenüber von aussen mechanisch zugefügten Oberflächenschäden, d.h. ihre mechanische Schadensresistenz, ist verglichen mit der reinen Matrixkomponente verbessert. Es wird untersucht, ob zwischen Festigkeitsabnahme sowie Bruchzähigkeits- und Schadensresistenzzunahme in Duplex-Keramiken und einem inneren Spannungsintensitätsfaktor, K_i, um die Druckzone ein Zusammenhang besteht.

On a étudié la résistance mécanique et la ténacité de diverses céramiqes duplex, élaborées par frittage naturel ou par pressage isostatique à chaud. On montre que, par rapport aux valeurs obtenues pour la matrice, la résistance mécanique est plus faible et la ténacité plus élevée. La transformabilité des particules de ZrO_2 dans les zones incorporées a été examinée. La résistance des céramiques duplex après introduction mécanique de défauts de surface, c'est à dire leur résistance à l'endommagement mécanique, est accrue par rapport à celle de la matrice seule. On tente de relier la résistance mécanique normalisée, la ténacité normalisée et la résistance à l'endommagement mécanique, à un facteur d'intensité de contrainte interne, K_i, généré autour de la zone en tension.

1 Introduction

Duplex ceramics consist of relatively large pressure zones of a component B dispersed homogeneously in a ceramic matrix A. The duplex concept is described in detail in previous papers.[1–4] The stress distribution within and around the pressure zones can be calculated by modifying a stress model proposed by Lundin.[5] The stress distribution depends on the effective volume expansion of the B zones relative to their environmental matrix A, ε_v, the B zone size, R_B, and the volume fraction of the pressure zone component B, V_f.[1] Assuming that a crack initiates from matrix/zone-interfacial defects and encircles the zone before it extends radially and finally links up with cracks originating from neighboring zones, it is possible to calculate the conditions for spontaneous crack initiation and propagation.[1,3]

The transformability of dispersed ZrO_2 particles depends in general on the volume fraction, grain size, grain size distribution, and shape of the particles, dopants and sintering additives.[6] Following Whitney,[7] the martensite start temperature M_S for the

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transformation of ZrO_2 is reduced with about 0·3°C/MPa pressure. Preliminary studies showed that the fabrication conditions highly influence the transformability of ZrO_2 within the B zones,[1-3] and hence, their effective volume expansion. Incongruent sintering behavior of the components which is observed in most structures causes porous B zones. ZrO_2 particles within such zones transform nearly completely during cooling after fabrication, but the stress field caused by the volume expansion due to the transformation is reduced by the effect of porosity. These structures could be highly densified upon post-HIPing. However, the hydrostatic pressure in the zones seems to counteract the ZrO_2 transformation. The particles transform spontaneously only to a certain degree, but completely in a crack-tip stress field by stress-induction.

Early studies[8,9] on duplex ceramics showed that the incorporation of pressure zones into a matrix of Al_2O_3 and Al_2O_3 + 15 vol.% ZrO_2 always reduced the strength. K_{Ic} of the matrix could not be increased as a result of either too small or too large B zones. The results, however, indicated that a successful K_{Ic} can be achieved under certain preparation conditions.

The aim of this study is to investigate strength and toughness of duplex ceramics consisting of different components A and B. The strength/toughness results are compared with the properties of the pure matrix and used to analyze their damage resistance. The importance of the transformability of the ZrO_2 in the B-zones and its influence on the mechanical properties of the materials are discussed. This will be followed by an attempt to predict their strength on the basis of previous stress calculations.

2 Experimental Procedure

Different duplex ceramics were prepared by spray drying,[3] tumble mixing of the components, cold-isostatically pressing, sintering, and post hot-isostatically pressing (HIPing). The fabrication conditions are described in detail in a previous paper.[1] The specimen dimensions were 3·5–4 × 3·5–4 × 45 mm. Strength and toughness were measured in 4-point-bending (span 24/8 mm) at a cross-head speed of 0·3 mm/min. Fracture toughness was measured by the strength-in-bending method (ISB-method)[10] with a Vickers indentor load of 30 kg. The Vickers hardness was obtained by measuring the indentation diameter. The Young's modulus was determined by the ultrasonic method. The Young's modulus of ceramic mixtures can be estimated by the proportional contributions of their components.[11] The dynamically measured values are compared with the estimated values. The m-phase fraction of polished and fractured surfaces was determined by the X-ray intensity ratio.[12] The ZrO_2 transformation on fractured surfaces depends on the fracture stress. To achieve uniform fracture stress conditions, especially for ceramics of different strength, the samples were notched by a 30 g Vickers indentation to achieve a comparable modulus of rupture.

To avoid complicated and long descriptions a short designation for duplex structures is introduced which reflects their exact composition. The matrix components (A), 2Y-TZP, 3Y-TZP + 20 wt% Al_2O_3, and Al_2O_3 are labeled 2YZ, 3YZ20A, and AO, respectively. The pressure zone components (B), Al_2O_3 + 35, 50, 80 vol.% m-ZrO_2 and pure m-ZrO_2 are labeled az35, az50, az80, and z100, respectively. For small (16–32 μm diameter) and large (45–65 μm diameter) pressure zones the subscripts S and L are used, respectively. The duplex ceramic labeled 2YZ–20az35s, for example, consists of 20 vol.% of small pressure zones composed of Al_2O_3 + 35 vol.% m-ZrO_2 dispersed within a 2Y-TZP matrix.

3 Results

3.1 Transformability of B-ZrO_2

The spontaneous transformation of ZrO_2 particles in B zones dispersed within TZP and TZP-containing matrix materials is nearly complete after sintering. After HIPing, however, the m-ZrO_2 fraction is considerably reduced. This is shown in Fig. 1 for duplex ceramics consisting of a 3YZ20A matrix with 10 and 20 vol.% of small (16–32 μm diameter) zones of az35, az50, az80 and z100. For structures containing large zones (45–65 μm diameter) the content of m-ZrO_2 in the B zones is about 15–30% higher. The transformation completes

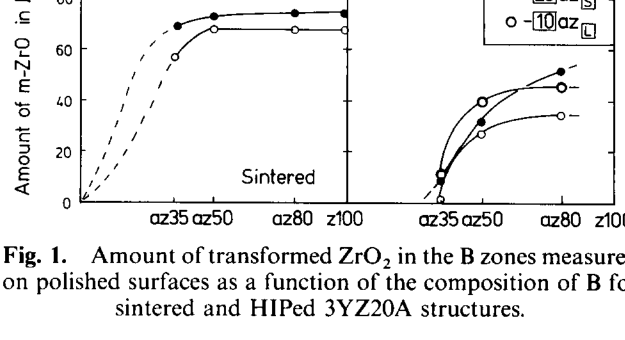

Fig. 1. Amount of transformed ZrO_2 in the B zones measured on polished surfaces as a function of the composition of B for sintered and HIPed 3YZ20A structures.

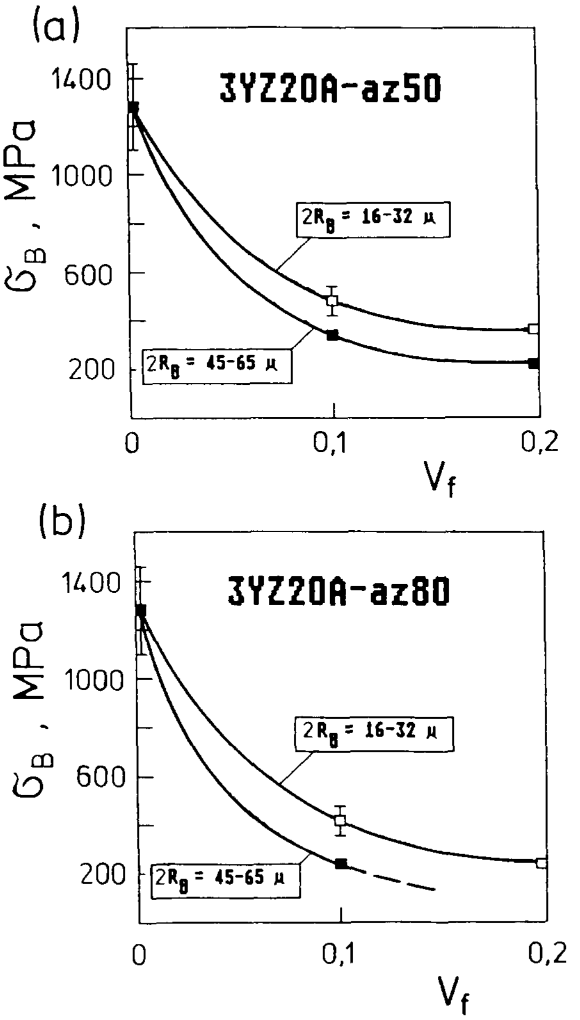

Fig. 2. Strength of duplex ceramics consisting of 3YZ20A with pressure zones of (a) az50 and (b) az80. The open symbols correspond to small (16–32 μm diameter) zones and the solid symbols to large (45–65 μm diameter) zones.

during fracture in both, sintered and HIPed materials.

In 2YZ–az35 systems the transformation is totally suppressed after HIPing. The m/(t + m) ratio on the polished surface of HIPed 2YZ is, at 0·01, the same as on the polished surface of HIPed 2YZ–10az35$_L$ and 2YZ–20az35$_S$. In contrast, the m/(t + m) ratio on fracture surfaces of the pure matrix is 51%, compared with average values of 58% and 63% for 2YZ–10az35$_L$ and 2YZ–20az35$_S$, respectively.

In all tested sintered and HIPed AO structures no t-ZrO$_2$ could be detected on polished and fracture surfaces.

3.2 Strength

The strength of the pure B zone components is very low when compared to the high strength of the matrix components, which lies in the range between 320 MPa for sintered AO and 1780 MPa for HIPed 3YZ20A. The strength of sintered az35, az50 and az80 was 143, 141 and 86 MPa, respectively. The strength of z100 could not be measured because it decays completely after sintering. The strength of duplex ceramics is generally reduced when compared with the strength of the pure matrix material. The following tendencies which confirm the results of previous studies[8,9] are apparent:

The strength decreases with

(i) increase volume fraction of B, V_f (Fig. 2(a) and 2(b));
(ii) increasing B zone size, R_B (Fig. 2(a) and 2(b)); and
(iii) increasing amounts of m-ZrO$_2$ in the zones (Fig. 3).

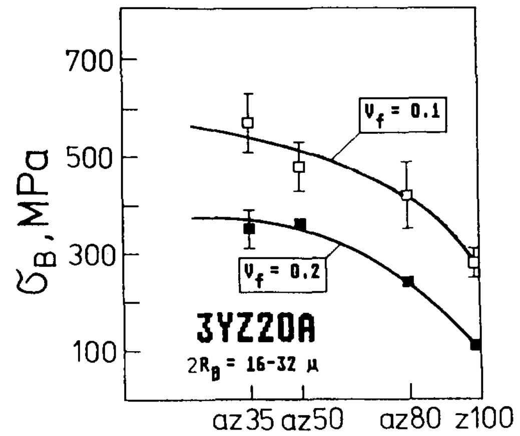

Fig. 3. Strength of duplex ceramics consisting of 3YZ20A with different pressure zone components. The strength decreases with increasing content of m-ZrO$_2$ in the zones as well as with increasing volume fraction, V_f, of the zones as is shown by the curves for $V_f = 0{\cdot}1$ (open squares) and $V_f = 0{\cdot}2$ (solid squares).

The strength of duplex ceramics is in most cases increased by HIPing (Fig. 4). However, the strength of those materials in which spontaneous cracking occurs upon cooling after the sintering step can not be further increased by HIPing.

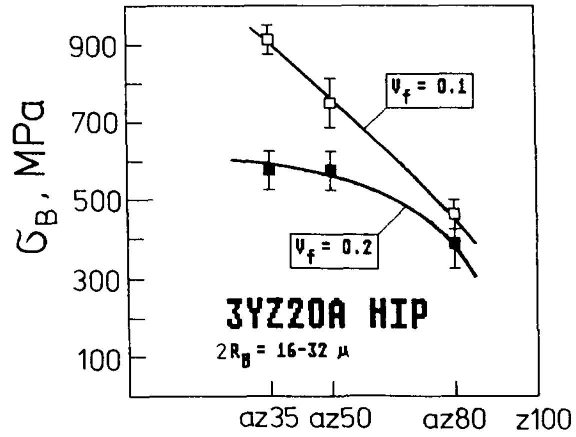

Fig. 4. Strength of HIPed duplex ceramics consisting of 3YZ20A with different pressure zone components. Open and solid squares correspond to materials with 10 and 20 vol.% of pressure zones, respectively.

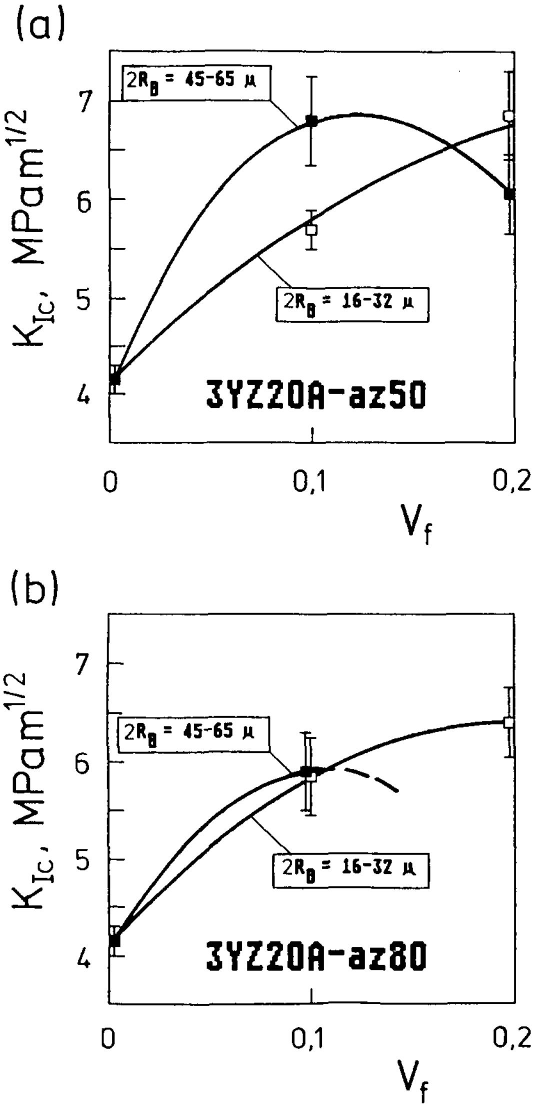

Fig. 5. K_{Ic} of duplex ceramics consisting of 3YZ20A with pressure zones of (a) az50 and (b) az80. The open symbols correspond to small (16–32 μm diameter) zones and the solid symbols to large (45–65 μm diameter) zones.

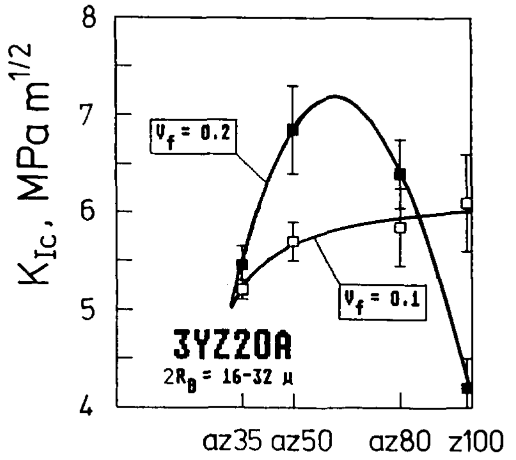

Fig. 6. K_{Ic} of duplex ceramics consisting of 3YZ20A with different pressure zone components. Open and solid squares correspond to materials with 10 and 20 vol.% of pressure zones, respectively.

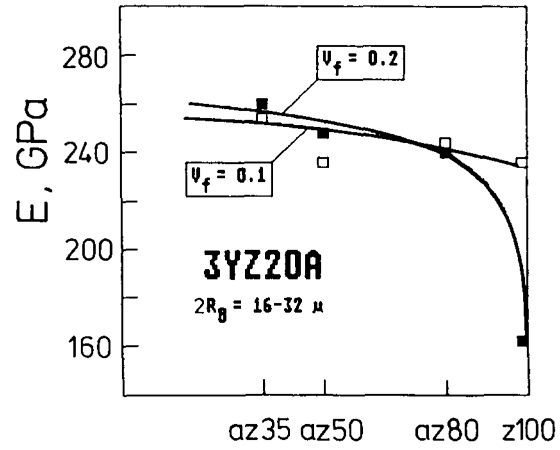

Fig. 7. Young's modulus of duplex ceramics consisting of 3YZ20A with different pressure zone components. Open and solid squares correspond to materials with 10 and 20 vol.% of pressure zones, respectively.

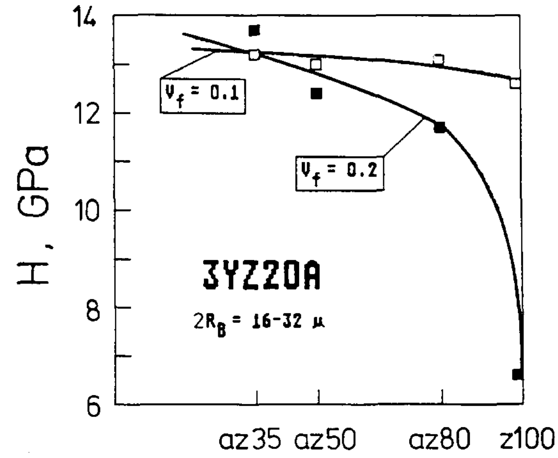

Fig. 8. Vickers hardness of duplex ceramics consisting of 3YZ20A with different pressure zone components. Open and solid squares correspond to materials with 10 and 20 vol.% of pressure zones, respectively.

3.3 Toughness

The fracture toughness of duplex ceramics is increased when compared to the pure matrix. In Figs 5 and 6 the dependence of K_{Ic} on the volume fractions of B, zone size and content of m-ZrO_2 inside B is illustrated for the system 3YZ20A. A similar behavior has been observed in all tested materials. However, the toughness of TZP and TZP-containing matrices can be enhanced by more than 60%, whereas the toughness increase in AO does not exceed 30%.

3.4 Young's modulus and Vickers hardness

The Young's moduli of duplex ceramics agree tolerably well with the estimated values, with the exception of one composite. Figure 7 shows that the dynamic values fall off for 3YZ20A–20z100$_S$. The

Young's modulus is only 161 GPa when compared with an expected value of about 240 GPa. This indicates that the structure is heavily cracked after fabrication.

The Vickers hardness is easier to measure and can be determined more precisely than the Young's modulus. It is reduced for all duplex ceramics when compared with the matrix value indicating stress-induced microcracking during indentation. The dependence of the volume fraction of B and the amount of m-ZrO_2 in B on Young's modulus and Vickers hardness is very similar. This is shown in Fig. 8.

4 Discussion

The X-ray measurements showed that in TZP systems the spontaneous transformation of ZrO_2 particles within the B zones is considerably reduced after HIPing. This stands in direct contrast to AO systems where no stabilization of B-ZrO_2 has been observed.

A part of the t-ZrO_2 is thought to be stabilized by the hydrostatic pressure in B which develops on cooling due to a positive thermal expansion mismatch ($\alpha_A > \alpha_B$) between A and B and the beginning transformation. This presupposes highly densified B zones. Following Whitney,[7] the M_S temperature is reduced with 0·3°C/MPa pressure. The M_S temperatures of the pure B components az35, az50, az80 and z100 are 740, 850, 920 and 1050°C, respectively.[3] Taking the estimated grain size of the B zones of 2–4 μm after sintering and 3–5 μm after HIPing into account[3] the measured M_S temperatures are in good agreement with the M_S temperatures reported by Wagner.[13] Following Whitney[7] a pressure of 2·4, 2·8, 3·0 and 3·4 GPa would be required to stabilize all ZrO_2 particles in B zones of az35, az50, az80 and z100, respectively. Based on the calculations in the accompanying paper[1] the hydrostatic pressure in the B zones during a complete transformation can be estimated to be at maximum 1·7, 2·2, 3·0 and 3·5 GPa, respectively. This indicates that pressure will lead only to a partial stabilization.

The stabilization of B-ZrO_2 in TZP systems which is not observed in AO systems may be a result of the diffusion of Y^{3+} cations from the TZP matrix into the B zones. In a two-phase system consisting of Al_2O_3 and ZrO_2 such a process is controlled by the diffusion speed through the ZrO_2 lattice.[14] Taking the heating and cooling times during fabrication into consideration it can be estimated that Y^{3+} cations can penetrate into B to a maximum of 0·3 and 0·7 μm during sintering and HIPing, respectively. With respect to an average zone diameter of 24 μm for small and 55 μm for large zones a volume fraction of 7·3 and 3·2% of the zone volume could be infiltrated by Y^{3+} after sintering and 16·5 and 7·4% after HIPing, respectively. Although diffusion is apparently not an important process it is probably responsible for the appearance of two other effects. The transformability of ZrO_2 in small B zones is obviously lower than in large zones which may be a result of the larger infiltrated volume fraction in small zones. In HIPed 2YZ–az35 structures the increase of the m/(t + m) ratio during fracture is much larger than in the matrix. This is thought to be due to a stress-induced transformation of material A around the B zones in addition to the complete transformation within B. The transformation of matrix material can be a result of both the stresses around the zones and a diminution of Y^{3+}.

The influence of the pores in the B zones in incongruently sintered structures is not yet clarified. Pores may influence the stress distribution within the zones, the stress transfer to the matrix and the diffusion process in a complex manner.

From the measured data it is apparent that the mechanical properties of duplex ceramics depend on many different parameters. Strength and toughness depend first of all on the volume fraction of B, the amount of ZrO_2 within B, and the size of B. Although K_{Ic}, as measured by the ISB technique, has not been observed for all cases to pass through a maximum within the tested range of composition, it can be expected that a maximum is reached before spontaneous cracking occurs. The amount and size of B zones and their volume expansion necessary to trigger spontaneous cracking can be estimated using the calculation proposed in the accompanying paper.[1]

Of similar importance are the mechanical properties of the components. The lower toughness increase in AO when compared to TZP, with both having comparable toughness, is thought to be due to the high stiffness of AO. As can be deduced from theoretical considerations, matrices of high Young's modulus are very sensitive with respect to cracking.[1]

In addition to the mechanical properties of the components, the strength is affected considerably by the fabrication conditions. Duplex ceramics consisting of components which sinter congruently can be almost fully densified by sintering.[1,3] The strength of these materials could not be improved considerably by HIPing. Incongruent sintering behavior of the components, however, always leads to more porous

B zones.[1,3] The high transformability of the unconstrained ZrO_2 particles in the B zones[1,3] and the pores resulted in low strength values, which could be increased considerably upon further densification during HIPing.

In contrast to the decrease in strength, the increase in toughness does not necessarily depend on the fabrication conditions, and hence, not on the amount of spontaneously transformed ZrO_2 in the B zones, nor on the volume expansion of the B zones on cooling after fabrication. This behavior can be observed especially in materials consisting of incongruently sintered components, such as $3YZ20A\text{–}20az50_S$ and $3YZ20A\text{–}10az50_L$, which exhibit the same toughness increase whether only sintered or post-densified by HIPing. After sintering the porosity of the B zones in the above structures was about 10 and 3%, respectively.[3] The ZrO_2 particles in the B zones have transformed to more than 70% after fabrication[1-3] (Fig. 1). Cracks around Vickers indentations are affected severely by the presence of the pressure zones. They are deflected towards the zones (Figs 9 and 10). New cracks are initiated at neighboring zones. Using a special dyeing techniques it could also be observed that stress-induced microcracking within the zones occurs in a more or less extended process zone around Vickers indentations.[3,15] After HIPing a large fraction (more than 60%) of ZrO_2 in the B zones remains tetragonal due to the high hydrostatic pressure in the zones. No microcracking and deflection occur around indentations; the cracks are shorter than in sintered duplex ceramics with respect to the same indentation load. After fracture, however, the transformation is complete on the fracture surface.

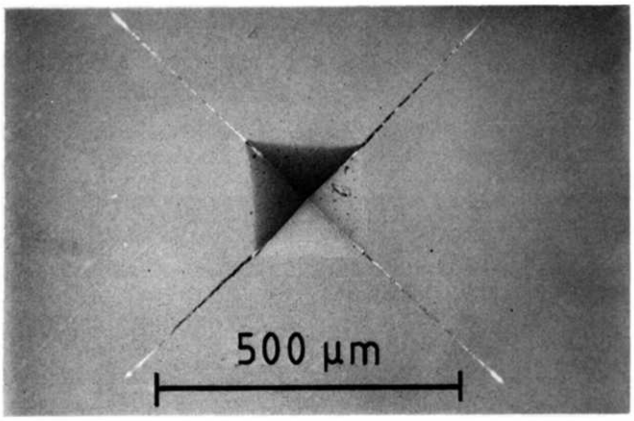

Fig. 9. Crack pattern around a Vickers indentation of 30 kg load on the polished suface of 3YZ20A.

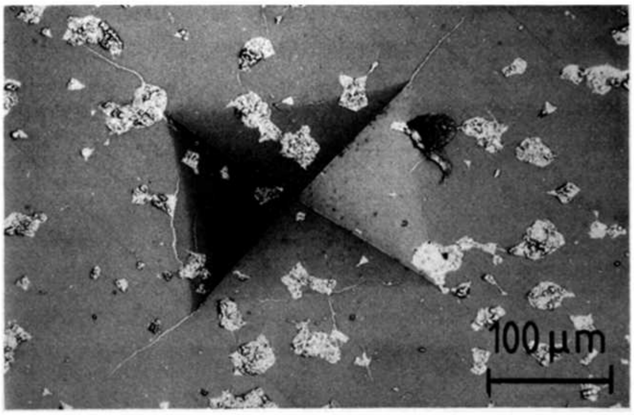

Fig. 10. Crack pattern around a Vickers indentation of 30 kg load in the polished surface of $3YZ20A\text{–}20az50_S$.

In the accompanying paper[1] it has been shown that fracture initiation processes in duplex ceramics can be predicted reasonably well. The attempt to use those calculations as a basis to predict their strength failed. This may indicate that the strength of duplex ceramics is not only controlled by the crack initiation event. A prediction of the change in strength and toughness, however, may be possible by using a measure which only characterizes the qualitative influence of the pressure zones on the mechanical behavior of the matrix. Such a measure should combine the most important structural features of duplex ceramics simultaneously. The effect of pressure zones increases with increasing volume fraction of B zones, V_f, increasing zone radius, R_B, and increasing effective volume expansion of the zones, ε^v. In the previous paper,[1] the Lundin stress model[5] has been slightly modified to calculate the stress distribution in duplex structures. Based on the tangential tensile stress which is maximum on the matrix/zone-interface, $p^A_{tg}(R_B)$, the influence of the pressure zones on the mechanical properties of the matrix may be described by an 'internal stress intensity factor, K_i' around pressure zones which is defined as

$$K_i = p^A_{tg}(R_B) R_B^{1/2} \tag{1}$$

Since p^A_{tg} depends on the volume expansion of the zones, ε^v, and the volume fraction of zones, V_f,[1]

$$K_i = 1/2 c_4 (1 + 2{\cdot}7 V_f) R_B^{1/2} \tag{2}$$

and therefore

$$K_i \sim \varepsilon^v (1 + 2{\cdot}7 V_f) R_B^{1/2} \tag{3}$$

K_i has been calculated for each composition assuming a complete transformation of ZrO_2 causing a 4% volume expansion. The thermal expansion mismatch between matrix and pressure zones has also been taken into account.

To enable a better comparison of the results for strength and toughness of duplex ceramics, σ_B^{AB} and K_{Ic}^{AB}, are related to the respective values of their pure matrices, σ_B^A and K_{Ic}^A, which leads to the relative

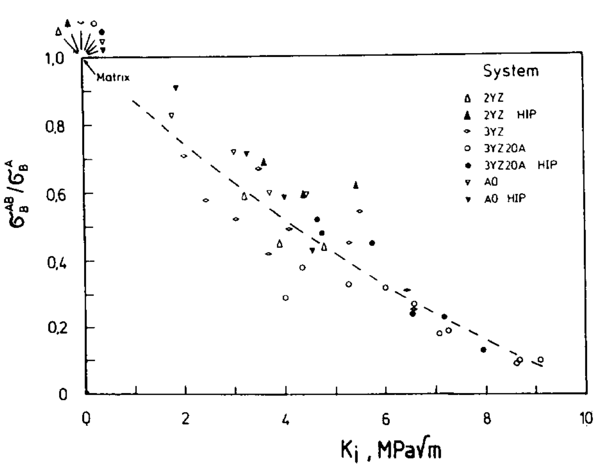

Fig. 11. Plot of the relative strength loss of duplex ceramics, σ_B^{AB}/σ_B^A, versus the calculated internal stress-intensity factor, K_i.

strength loss, σ_B^{AB}/σ_B^A, and toughness increase, K_{Ic}^{AB}/K_{Ic}^A, for each duplex composite when compared to its matrix. The toughness data can be also evaluated in another way to obtain the mechanical damage resistance which is calculated by relating the retained strength of a material, σ_R, after a 30 kg identation versus its initial strength, σ_B (before indentation).

In Fig. 11 the normalized strength of all tested duplex ceramics is plotted versus K_i. It is apparent that the plots lie within a band commencing with $\sigma_B^{AB}/\sigma_B^A = 1$ and $K_i = 0$ for the pure matrix and decreasing in a monotonic manner with increasing K_i. Sintered duplex ceramics with porous B zones are obviously lying beneath the trend, whereas HIPed materials with partially transformed zones are found above the trend. In Fig. 12 both the toughness increase and the damage resistance are plotted versus K_i for various duplex ceramics of the AO and 3YZ20A systems. The toughness passes through a maximum with increasing K_i, while the damage resistance increases continuously.

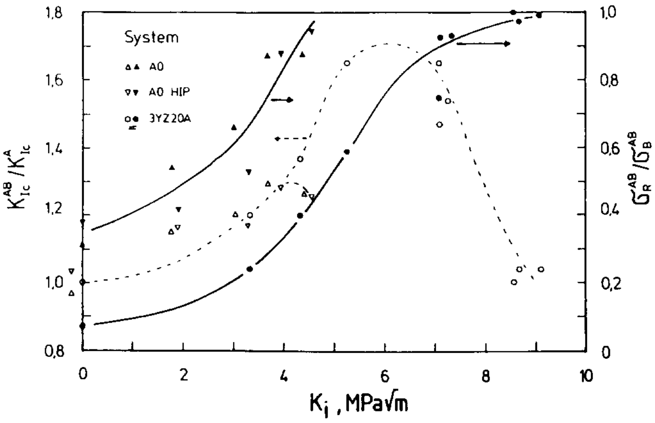

Fig. 12. Plot of the relative toughness increase in duplex ceramics, K_{Ic}^{AB}/K_{Ic}^A, and their damage resistance, $\sigma_R^{AB}/\sigma_B^{AB}$, versus the calculated internal stress-intensity factor, K_i.

With the internal stress-intensity factor the strength of duplex ceramics becomes predictable following a trend which can be described by

$$\sigma_B^{AB}/\sigma_B^A = 1 - \frac{0{\cdot}1K_i}{\mathrm{MPam}^{1/2}} \tag{4}$$

With the knowledge of the mechanical properties of the matrix the toughness increase in duplex ceramics may also be reasonably predicted.

There is some doubt about the reliability of toughness measurements using indentation crack length or the indentation strength-in-bending method. It is, in addition, questionable, if the crack-resistance behavior of ceramics is sufficiently described by a single toughness value. The presence of different toughening mechanisms would have a tremendous effect on the crack growth resistance behavior (*R*- or K^R-curve behavior) of a material. A single toughness value, however, could at best summarize the toughening contributions. In complementary papers the K^R-curve behavior of duplex ceramics and its significance to the thermal shock resistance are discussed.

5 Conclusions

Duplex ceramics show a reduced strength but an enhanced toughness and damage resistance when compared to their pure matrix. The transformability of ZrO_2 in the B zones depends on the fabrication conditions. Pressure and diffusion of Y^{3+} cations from the matrix into the zones are thought to be mainly responsible for the stabilization of B-ZrO_2 in TZP matrices. The influence of the pressure zones on the mechanical behavior of the matrix can be characterized by an internal stress-intensity factor around the zones, K_i, which increases with increasing volume fraction of B, increasing zone size, and increasing volume expansion of the zones. The strength of duplex ceramics when compared to the strength of their matrix decreases continuously with increasing K_i values. The toughness of duplex materials when compared with the toughness of its pure matrix increases for low K_i values, passes through a maximum and decreases again for higher K_i values. In contrast to the strength, the damage resistance increases steadily with increasing K_i.

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