

## MECHANICAL PROPERTIES OF DISPERSION CONTAINING CERAMICS

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Australia**Abstract:**

The variously developed approaches to determine the strength and toughness of dispersion containing ceramics are reviewed. They are then compared with observations of model duplex ceramic materials and ceramic–diamond composites. A simple fracture mechanics basis is developed to rationalise the strength data. This relationship provides a sounder basis for explaining the strength of dispersion containing ceramics than a recently proposed empirical expression.

**Introduction:**

It is well known that the presence of impurities or inclusions in glasses or ceramics may or may not influence the strength significantly.<sup>1-7</sup> It has been observed that mismatch of thermal expansion coefficients of inclusions and matrices results in intense stress fields about and within the inclusions which build up during cooling after fabrication.<sup>1-7</sup> The presence of such stress fields severely affects the mechanical behaviour of a homogeneous brittle matrix material. These stresses, particularly when the inclusion is under compressive stress resulting in tensile circumferential hoop stresses are often the source of crack initiation. Subsequent growth of such cracks are influenced by the spatially varying stress fields in a dispersion containing matrix. These stresses may deflect, attract and even trap the embryonic crack depending upon whether the hydrostatic stress within such inclusions is tensile or compressive.<sup>1,4-7</sup> Such crack deflection and trapping in the case of homogeneously dispersed inclusions leads to an increase in fracture toughness of the composite material.

A considerable debate on the mechanisms responsible for the influence of dispersed inclusions on the strength and toughness of brittle materials has developed over the last few decades. Frey and Mackenzie<sup>8</sup> explored the influence of dispersions of  $\text{Al}_2\text{O}_3$  and  $\text{ZrO}_2$  in glass on the strength and found that it increased in direct proportion to the increase in elastic modulus of the composite. Hasselman and Fulrath<sup>9</sup>, who investigated a range of metal and ceramic inclusions in glass, proposed that the role of the dispersions on the strength was to limit the flaw size to the interparticle spacing, thereby increasing the strength with increasing volume fraction. Miyata and Jinno<sup>10</sup> proposed an alternative explanation in that the presence of the dispersed particles initially lowered the strength through the development of a bigger flaw than present in the matrix glass. However, up to this stage none of these authors considered the thermal expansion mismatch effects.

Borom<sup>11</sup> in an excellent review of the data available at that time was able to rationalise the approaches of Frey and Mackenzie<sup>8</sup> and of Hasselman and Fulrath.<sup>9</sup> However he convincingly demonstrated that, the up to then overlooked residual stress state due to thermal expansion mismatch played a critical role. In the glass ceramic materials he investigated he found that if the thermal expansion mismatch was too great then cracking developed at the interface between the higher thermal expansion crystals and the lower thermal expansion matrix. The extent of strengthening appeared to be dependent upon the size and volume fraction of the crystalline phase, there being essentially minimal difference in elastic modulus of both phases. Borom<sup>11</sup> proved beyond doubt the importance of the residual stress by observing a monotonic decrease in strength of the glass ceramic with increasing temperature up to the softening temperature.

More recently Miyata and colleagues<sup>12,13</sup> have investigated the influence of alumina particles in two glasses with differing thermal expansion coefficients as well as a model glass-glass composite system in which the thermal expansion coefficient, diameter and volume fractions were varied. The presence of the alumina initially decreased the strength but with increasing volume fraction it increased, whereas the toughness measured using pre-cracked notched bend tests monotonically increased with volume fraction of alumina giving results identical with those of Swearingen et al.<sup>14</sup> Miyata et al<sup>12</sup> did notice that the initial decrease in strength was dependent upon the particle size whereas the toughness was nearly independent of size. The excellent model glass sphere-glass matrix composites confirmed Miyata's previous data. However in this system the glass beads used were of equal or lower thermal expansion coefficient than that of the four matrices used. In all instances the strength decreased when a thermal expansion mismatch existed whereas the toughness initially increased then decreased to a value equal to or below that of the matrix. The influence of particle size confirmed their previous data in that for an identical volume fraction the strength decreased with increasing particle size, the toughness on the other hand increased with particle size. These results were similar to the more complex alumina-zirconia composite materials developed by Claussen et al<sup>15</sup> who also explored the

influence of particle size and volume fraction of monoclinic zirconia particles. Miyata et al. followed the interpretation of Kreher and Pompe<sup>16</sup> of Claussen's data in that the increased toughness was due to the development of a microcrack process zone about the crack tip.

More recently Lutz et al.<sup>17-19</sup> have investigated another model system consisting of alumina-monoclinic zirconia spheres in various matrices. The magnitude of the compressive stress within the inclusions could be easily modified by changing the volume fraction of monoclinic zirconia. The volume fraction and size of these inclusions was also investigated. These authors were able to rationalize the strength of a very large number of these materials based upon an empirical parameter,  $K_i$ , termed the "internal stress intensity factor". This term is defined by the following expression,

$$K_i = \sigma_{\theta} \sqrt{R} \quad (1)$$

where  $\sigma_{\theta}$  is the hoop stress at the inclusion interface and  $R$  is the inclusion radius. These authors also incorporated a modification of the magnitude of the stress within an inclusion from that proposed by Selsing<sup>20</sup> for an isolated inclusion in a matrix. They used the approach developed by Lundin<sup>21</sup> who considered the residual stress state in a porcelain due to the presence of quartz grains.

The aim of this study is to extend the previous approaches mentioned above and apply the results to some recently developed ceramic-diamond composites. Initially the residual stress state in a dispersion containing material is reviewed then a simple fracture mechanics analysis for the onset of cracking will be developed in an endeavour to justify the  $K_i$  parameter mentioned above.

### Theoretical Considerations

The state of residual stress about an isolated particle in a matrix has been given by Selsing.<sup>20</sup> The magnitude of the hydrostatic residual stress within the inclusion is

$p^T = -Ke^T$  where  $e^T$  is the volumetric strain due to thermal expansion mismatch and any volumetric phase transformation dilational strains and  $K$  is the bulk modulus. The value of  $e^T$  for such particle is given by

$$e^T = \frac{\Delta V}{V} + 3(\alpha_i - \alpha_m)\Delta T \quad (2)$$

where  $\Delta V$  is the volume dilation due to the phase transformation,  $\alpha_i$  and  $\alpha_m$  are the thermal expansions of the inclusion and matrix and  $\Delta T$  is the temperature difference between ambient and softening temperature of the matrix. According to Selsing<sup>20</sup> the hoop,  $\sigma_\theta$  and radial,  $\sigma_r$ , stresses are given by

$$\sigma_\theta = -\frac{\sigma_r}{2} = \frac{E^*e^T}{3} (R_o/r)^3 \quad (3)$$

where

$$E^* = \left[ \frac{1+\nu_m}{2E_m} + \frac{1-2\nu_i}{E_i} \right]^{-1}$$

The approach proposed by Lundin<sup>21</sup> considers the inclusion of radius  $R_B$  embedded in a homogeneous matrix sphere of radius  $R_A$ . Assuming a densely packed system and stress free voids the hydrostatic pressure within the inclusions is,

$$p^T = -a_i[1 - (R_B/R_A)^3] \quad (4)$$

where

$$\begin{aligned} a_i &= a_2/[a_3 + a_4(R_B/R_A)^3] \\ a_2 &= 2E_A E_B e^T/3 \\ a_3 &= 2E_A(1-2\nu_B) + E_B(1+\nu) \\ a_4 &= 2[E_B(1-2\nu_A) - E_A(1-2\nu_B)] \end{aligned}$$

where A and B refer to the matrix and inclusion respectively. The consequences of this model is that the hoop stress about the inclusions does not asymptotically approach zero as

$r$  becomes large as suggested by equation (3) but rather has a minimum value whereas the radial stress becomes zero at the outer shell boundary, that is

$$\begin{aligned}\sigma_r &= -a_1(R_B/r)^3 [1 - (r/R_A)^3] \\ \sigma_\theta &= \frac{a_1}{2}(R_B/r)^3 [1 + 2(r/R_A)^3]\end{aligned}\quad (5)$$

A more rigorous estimation of the stresses about an inclusion in a dispersion containing matrix has been developed by Bettles and Johnson<sup>22</sup>, and is given by

$$\sigma_\theta = 3K'_2 C_2 + 3\lambda_2 Y_2 + (2\mu_2 D_2)/V_f - 3K_2 \alpha_2 \Delta T \quad (6)$$

where  $\lambda$  = Lamé's constant,  $K'$  is the bulk modulus,  $\mu$  the shear modulus,  $V_f$  the volume fraction of inclusions and  $Y = [(1+\nu)\alpha\Delta T/3(1-\nu)]$ . The constants  $C_2$  and  $D_2$  are functions of the composite bulk modulus and thermal expansion and are solved for implicitly to determine these material quantities.

A fracture mechanics analysis of the stress intensity factor for a radial crack about an isolated inclusion has been discussed by a number of authors. Marshall and Green<sup>23</sup> have compared various proposed expressions and convincingly demonstrated that the most appropriate relationship is

$$K_R = \sigma_T (R/\pi)^{\frac{1}{2}} (R/c)^{3/2} [1 - (R/c)^2]^{\frac{1}{2}} \quad (7)$$

where  $c$  is the crack length. A maxima occurs at  $c/R = 1.29$  with  $K_R \sqrt{\pi}/\sigma_T \sqrt{R} = 0.44$  leading to a critical inclusion size  $R_c$ . Inserting  $K_R = K_{1c}$  of the matrix material leads to a value of

$$R_c = 16.9 (K_{1c}/\sigma_T)^2. \quad (8)$$

For a well developed penny shaped crack ( $c > R$ ) a simpler expression for  $K_R$  is given by

$$K_R = \sigma_T (R/\pi)^{1/2} (R/c)^{3/2} \quad (9)$$

leading to an equilibrium crack of size  $c_0$  about a super critical sized inclusion of,

$$c_0 = (\sigma_T R^2/\pi^{1/2} K_c)^{2/3} \quad (10)$$

In the presence of an applied stress  $\sigma_a$  the stress intensity factor for a crack about an inclusion is given by

$$K = K_a + K_R$$

$$K = \phi \sigma_a \sqrt{a} + \phi \sigma_T (R/\pi)^{1/2} (R/a)^{3/2} \quad (11)$$

The critical applied stress for instability of this system has been discussed by Swain.<sup>6</sup>

An alternative perspective of particulate reinforced materials has been developed by those considering the toughness of such composites. Taya et al.<sup>24</sup> have recently reviewed this topic and attempted to rationalise the toughness of a SiC – TiB<sub>2</sub> composite. These authors supported previous studies by Evans et al.<sup>25</sup>, Cutler and Virkar<sup>26</sup> in that the major toughening contribution is due to the periodic tension – compression residual stress. The toughness of such a composite is given by

$$K_{1c} = K_0 + 2\sigma_q\sqrt{2D/\pi} \quad (12)$$

where  $K_0$  is the matrix toughness,  $\sigma_q$  the (mean) residual stress and  $D$  is the average inter particle spacing. These authors used the Eshelby model to determine an averaged internal stress in the inclusions with a compensating (for volume) opposite stress in the matrix. Taya et al.<sup>24</sup> show that the mean stresses in the inclusion  $\langle\sigma_p\rangle$  and matrix  $\langle\sigma_m\rangle$  are

$$\frac{\langle\sigma_p\rangle}{E_m} = -\frac{2(1-f_p)\beta\alpha_1^*}{A}$$

and

$$\frac{\langle\sigma_m\rangle}{E_m} = \frac{2f_p\beta\alpha_1^*}{A} \quad (13)$$

where  $A = (1-f_p)(\beta+2)(1+\nu_m) + 3\beta f_p(1-\nu_m)$ ,  $\alpha_1^*$  is the mean thermal expansion mismatch and  $\beta = \left[\frac{1+\nu_m}{1-2\nu_p}\right] \left[\frac{E_p}{E_m}\right]$

A more sophisticated model of the toughening of such a dispersion containing material has been proposed by Bennison and Lawn.<sup>27</sup> However in their approach they propose that the particles act as bridging sites and that the closure forces so developed generate R-curve behaviour over a range dictated by the critical crack opening displacement that causes bridge fracture. This approach has been applied to alumina ceramics with the bridges being a consequence of frictional grain interlocks and is able to successfully explain the grain size dependence of strength.

### Experimental Details

Emphasis will be placed on the alumina–alumina zirconia duplex ceramics developed by Lutz and Claussen<sup>17,18</sup> and the ceramic–diamond composites developed by Bettles and Swain.<sup>28</sup> The former materials were prepared by conventional sintering whereas the latter were prepared by hot pressing. Details of the processing conditions have been published elsewhere<sup>17,28</sup>.

The strengths of the former were determined in four point flexure whereas the ceramic–diamond composites were measured with the ball on ring biaxial strength method using small 12–15 mm diameter plates approximately 1 mm thick.

For one system the residual stress within the composite was determined using precision neutron diffraction methods. Observations of the lattice spacing of the diamond before and after incorporation in alumina were determined.

### Observations and Discussion

#### i) Residual stress observations

The neutron diffraction pattern of the diamond before and after fabrication of the alumina–diamond composite,<sup>29</sup> revealed a clear shift to higher angles of the major peaks of the diamond in the diamond–alumina composite. The diamond particle size was 3–5  $\mu\text{m}$  and below that necessary to initiate radial cracks about the diamonds. The shift in the peak position corresponded to a change in lattice parameter of 0.00374 nm or a volumetric strain  $\epsilon^T$  of –0.315%. This value corresponds to a hydrostatic compressive stress within the diamond of 1.49 GPa which compares with the value of  $\epsilon^T$  calculated from equation (6) using  $\alpha_{D_1} = 5.4 \cdot 10^{-6}/\text{K}$ ;  $\alpha_{Al_2O_3} = 10.1 \cdot 10^{-6}/\text{K}$ ,  $K_{D_1} = 565$  GPa and  $\Delta T = 1100$  K, giving  $p_t = 1.80$  GPa. The values calculated from the analysis of Lundin<sup>21</sup> (eqn. 4) were 1.80 GPa and according to Taya et al.<sup>24</sup> (eqn. 13) were 1.92 GPa.

The relatively good agreement between the simple expression developed by Lundin and that of equation (6) is surprising. The limited analysis presented in the thesis of Lundin makes it difficult to justify such agreement. The approximate 20% over estimation of the magnitude of the residual stress maybe reduced by a slight reduction of the  $\Delta T$  or  $\alpha$  values used in these calculations.

#### ii) Ceramic–diamond composites

Observations of the variation of strength of a range of ceramic–diamond composites is shown in Figures 1–5. Apart from the cordierite system and to a lesser extent the

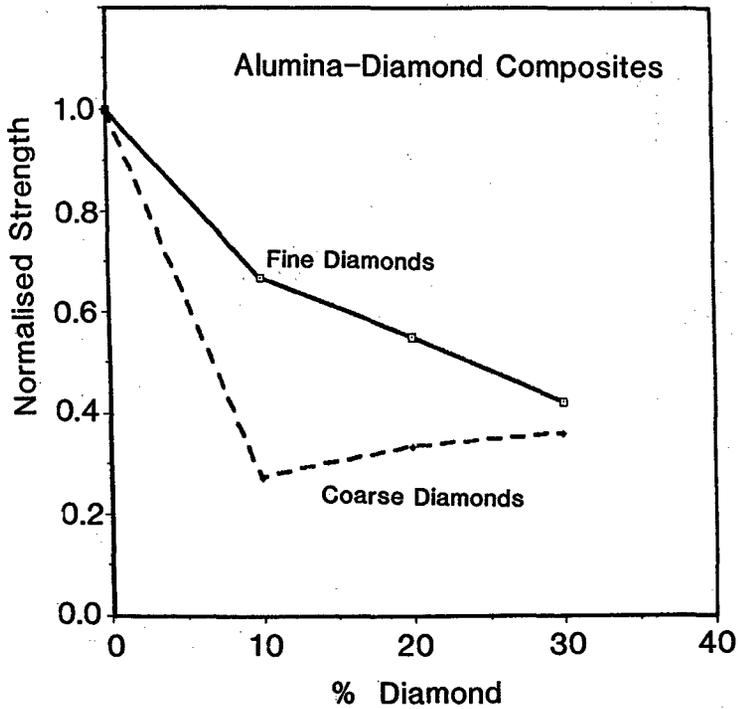


Figure 1. Normalised strength data for alumina-diamond composites as a function of volume fraction of diamonds and diamond particle size.

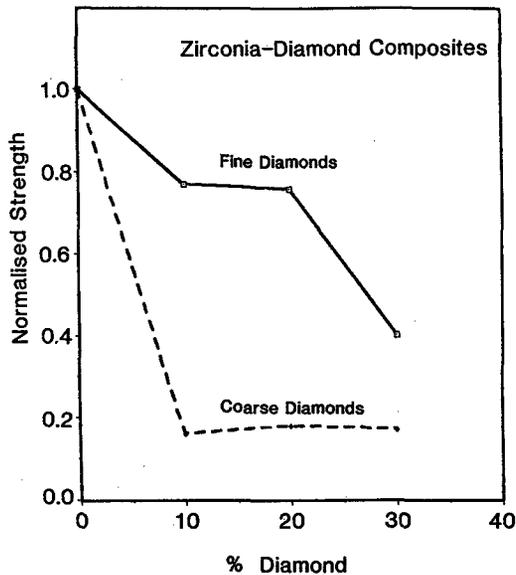


Figure 2. Normalised strength for 3Y-TZP-diamond composites as a function of volume fraction of diamonds for fine (0.5 – 3  $\mu\text{m}$ ) and coarse (20 – 35  $\mu\text{m}$ ) diamonds.

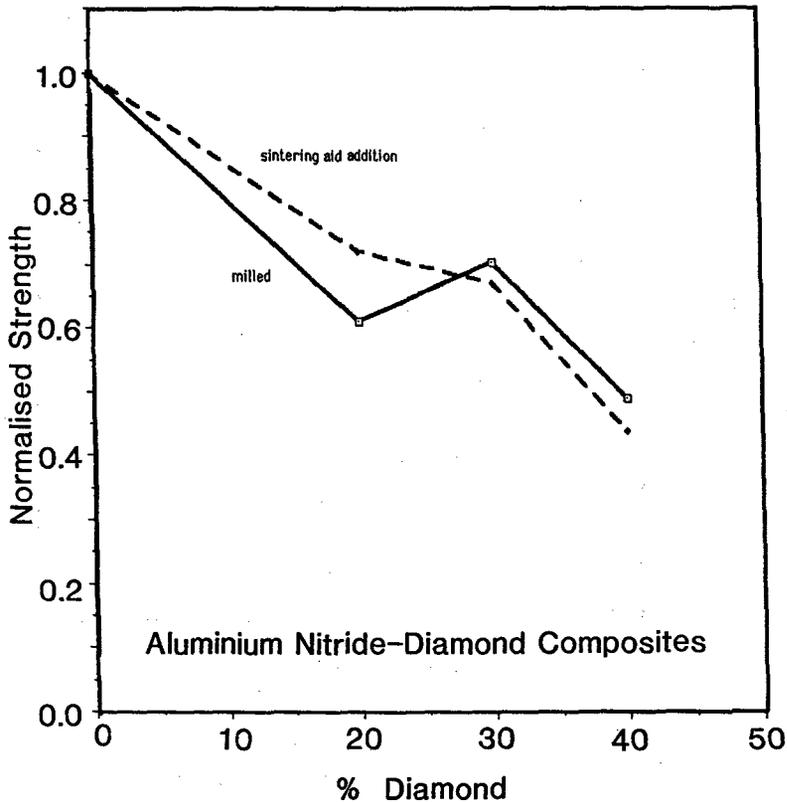


Figure 3. Strength of aluminium nitride–diamond composites for coarse grained diamonds as a function of diamond content.

aluminium nitride composite the strengths show a systematic reduction with volume fraction of diamond. The strength degradation shows a profound influence on diamond particle size. The cordierite is the only matrix that has a lower coefficient of thermal expansion (CTE) than that of the diamonds. The aluminium nitride has a CTE comparable to that of diamond whereas that of the alumina, zirconia and chromium carbide are much greater.

The plots of strength of the ceramic diamond versus the empirical relationship  $K_i$  proposed in equation (1) were completely unsatisfactory. Matrix materials with low values of fracture toughness appeared to fall on different trend lines from those with higher toughness values. Previous data generated by Lutz and Claussen<sup>18</sup> for matrices of alumina and tetragonal zirconia–alumina fell on an almost universal curve as shown in Figure 6.

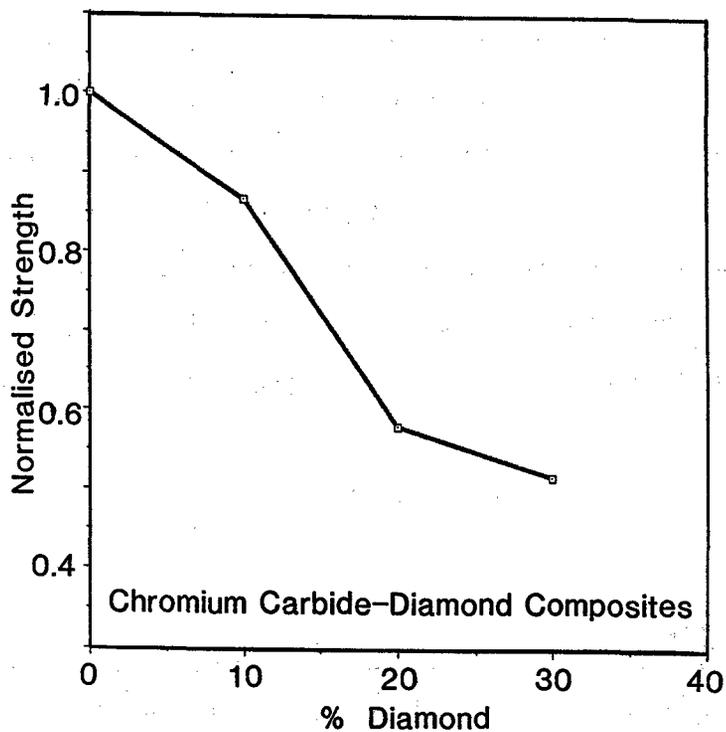


Figure 4. Normalised strength data for chromium carbide-diamond composites containing coarse diamonds as a function of the volume fraction of diamonds.

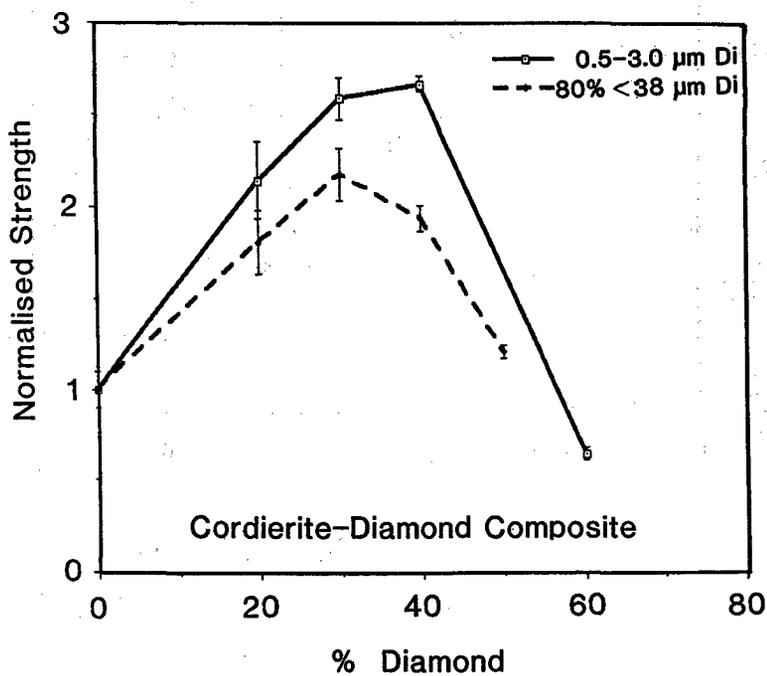


Figure 5. Normalised strength data for cordierite-diamond composites as a function of diamond content for two size fractions of diamonds.

In an attempt to rationalise the ceramic–diamond composite strength data an alternative basis for interpretation is explored using a modification of the Evans et al.<sup>25</sup> approach which was developed to rationalise toughness of dispersion containing materials. This has a distinct advantage in that there is then a more logical basis for a continuum for tensile or compressive stresses within the matrix. If we assume the strength of the composite is determined by that of the matrix alone, which is given by the sum of the compensating mean matrix stress due to the inclusions plus the applied stress, then

$$\sigma_0 = \langle \sigma_m \rangle + \sigma_a \quad (14)$$

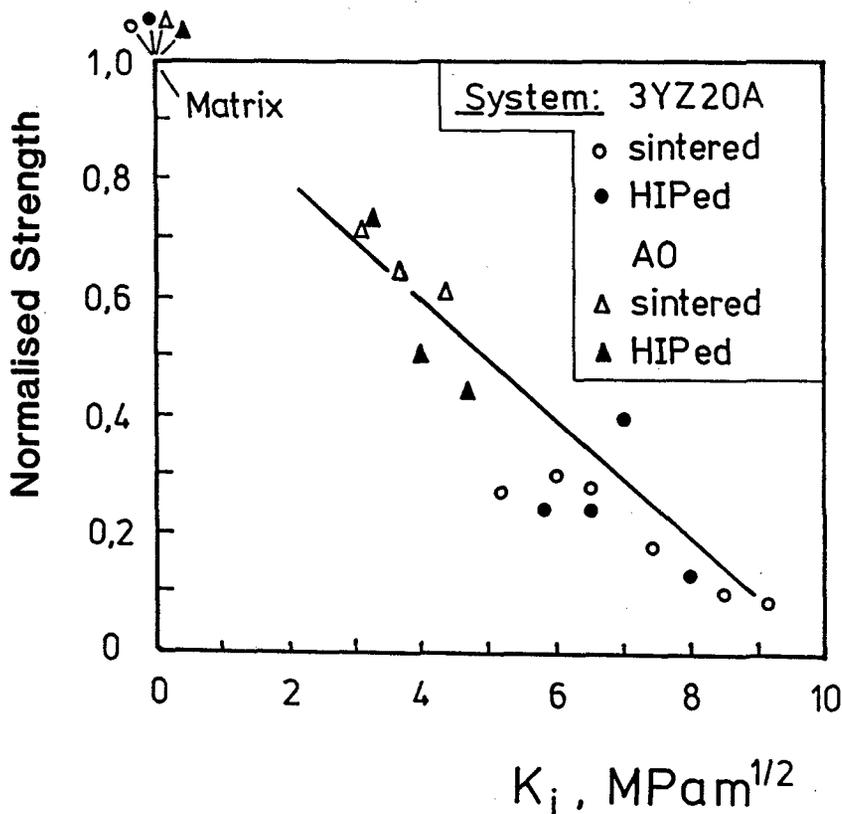


Figure 6. Strength data for a range of duplex ceramics of Lutz & Claussen normalised to the matrix strength and plotted versus  $K_i$  (equ.1).

where  $\sigma_0$  is the initial strength of the matrix and  $\langle \sigma_m \rangle$  is the mean tensile or compressive stress calculated from the expressions given by Taya et al.<sup>24</sup> Normalizing the above relation leads to

$$\sigma_c/\sigma_0 = 1 - \frac{\langle \sigma_m \rangle}{\sigma_0} \quad (15)$$

This expression suggests there is virtually no influence of inclusion particle size on the strength which does not agree with the observations in Figures 1 – 5. It would appear that only by considering a defect or flaw size related to the inclusion size, as previously suggested by Miyata et al.<sup>13</sup>, or the interparticle spacing as proposed by Hasselman and Fulrath<sup>9</sup> is it possible to improve on this approach. This is most readily seen with a fracture mechanics approach, where the crack tip stress intensity factor is given by

$$K = K_0 + K_a \quad (16)$$

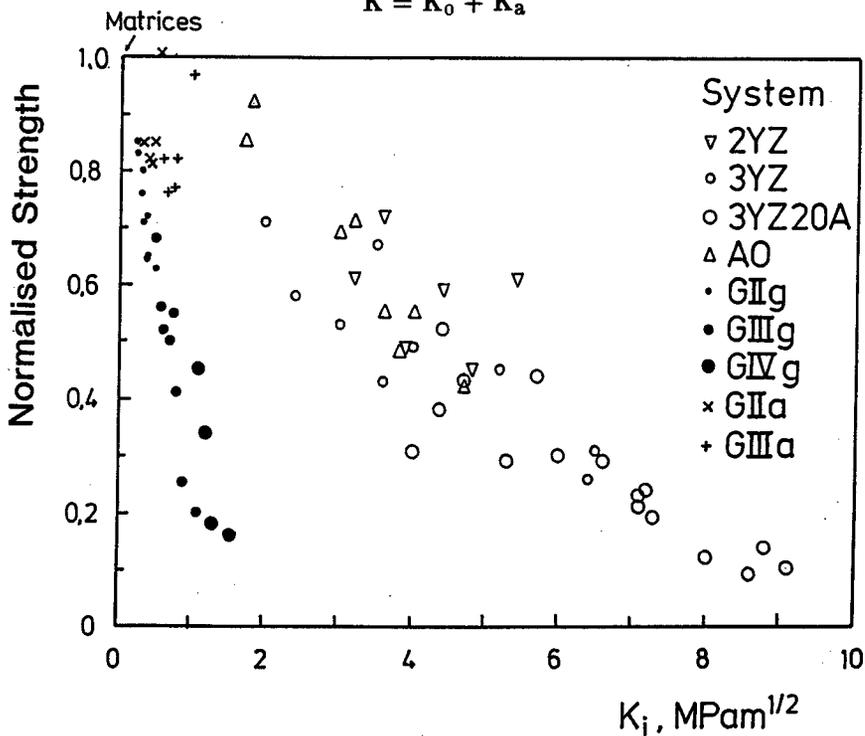


Figure 7. Normalised strength data from Figure 7 and from Miyata et al plotted against  $K_i$  and showing the unsatisfactory agreement between the various materials.

Assuming that the flaw size of the composite is  $c$ , which may be equal to the inclusion radius plus the pre-existing flaw size or the interparticle spacing, that is

$$c = R + a \quad (17a)$$

$$\text{or} \quad c = l = 4R(1 - V_f)/3V_f \quad (17b)$$

then

$$K = \langle \sigma_m \rangle \sqrt{\pi c} + \sigma_a \sqrt{\pi c} \quad (18)$$

If we let  $K = K_{1c}$  of the matrix then  $K_{1c} = \sigma_o \sqrt{\pi a}$  where  $\sigma_o$  is the matrix strength and  $a$  is the flaw size. Rearranging, the above expression maybe written as

$$\sigma_a / \sigma_o = \sqrt{\frac{a}{c}} \left[ 1 - \frac{\langle \sigma_m \rangle}{\sigma_o} \sqrt{\frac{c}{a}} \right] \quad (19)$$

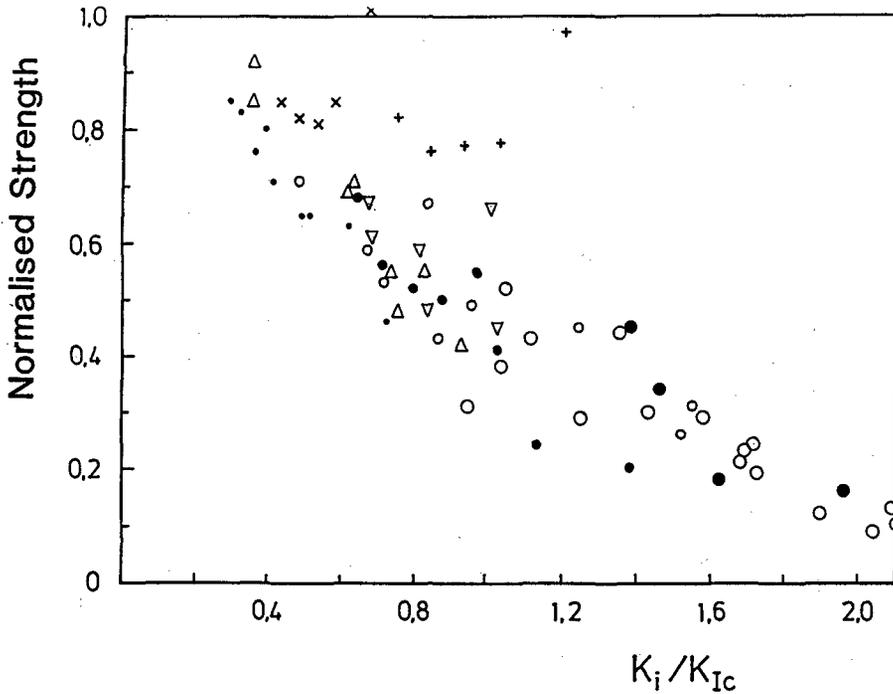


Figure 8. Normalised strength data from Figure 7 plot versus  $K_i$  normalised by the matrix  $K_{1c}$  as suggested by equations (22) and (23).

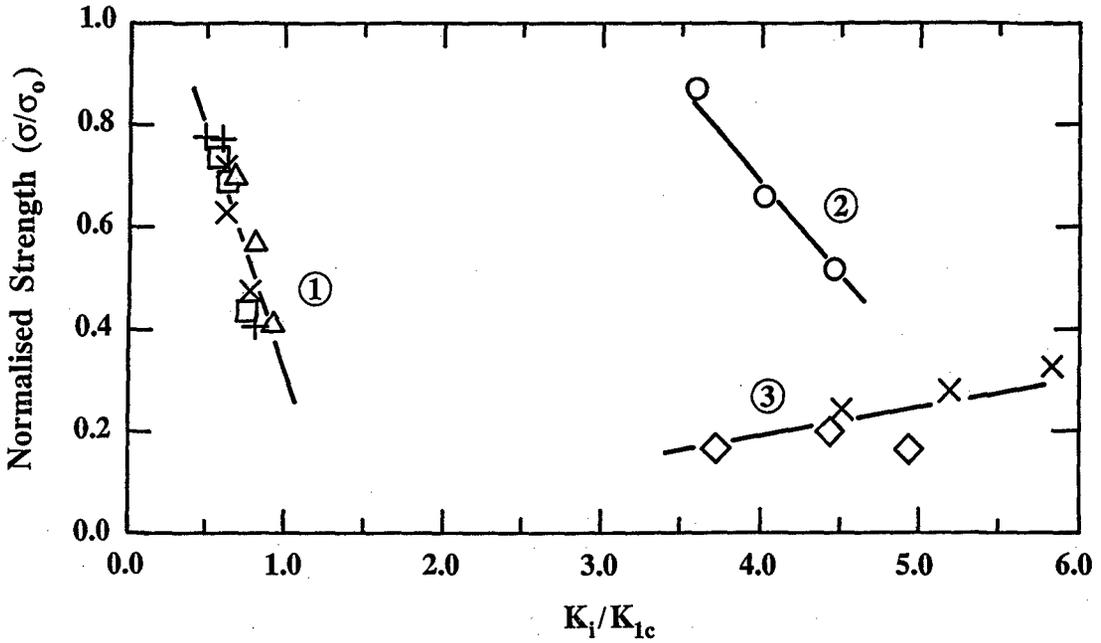


Figure 9. Normalised strength data for the ceramic-diamond composites (Figures 1 - 5) plotted versus normalised  $K_i$ .

If  $c$  is given by equ(17a), then equ(19) reduces to

$$\sigma_a/\sigma_o = \sqrt{\frac{a}{R+a}} \left[ 1 - \frac{\langle \sigma_m \rangle}{\sigma_o} \sqrt{\frac{R+a}{a}} \right] \quad (20)$$

In the limit  $R \ll a$  this is identical to equ(15), whereas if  $R \gg a$  it reduces to

$$\sigma_a/\sigma_o = \sqrt{\frac{a}{R}} \left[ 1 - \frac{\langle \sigma_m \rangle}{\sigma_o} \sqrt{\frac{R}{a}} \right] \quad (21)$$

But  $\langle \sigma_m \rangle = -\sigma_i V_f / (1 - V_f)$  where  $\sigma_i$  is the inclusion internal stress. The term  $\langle \sigma_m \rangle \sqrt{R}$  is then

$$\langle \sigma_m \rangle \sqrt{R} = -\sigma_i \sqrt{R} \left[ \frac{V_f}{1 - V_f} \right] = -\phi K_i$$

where  $K_i$  is the empirical relation given by equation (1). The normalised strength is then given by

$$\sigma_a/\sigma_o = \sqrt{\frac{a}{R}} [1 - \phi' K_i/K_{1c}] \quad (22)$$

where  $\phi' = \phi / 2\sqrt{\pi}$ .

The alternative expression for  $c$  given by equation (17b) leads to a very similar expression, namely

$$\sigma_a/\sigma_o = \sqrt{\frac{a}{\omega R}} [1 - \Omega K_i/K_{1c}] \quad (23)$$

where  $\omega = 2\sqrt{(1-V_f)}/3V_f$  and  $\Omega = \sqrt{V_f}/3\pi(1-V_f)$ .

This expression is nearly identical with the empirical  $K_i$  relationship and its further extension by Lutz.<sup>30</sup>

For many of the materials previously investigated by Lutz and Claussen<sup>18</sup> the  $K_{1c}$  value of the matrix materials was nearly identical and the range of the inclusion sizes studied was limited. Their data plotted in Figure 6 confirms the basis of the above expression. A much broader range of materials including data from Miyata et al<sup>13,14</sup> is compared in Figure 7 and illustrates the unsatisfactory relationship with  $K_i$ . However when this data is plotted normalised to the matrix  $K_{1c}$  as proposed in equations (22) and (23), a much better universal plot of the strength of a large number of dispersion containing composites is obtained, Figure 8.

The ceramic-diamond data is plotted in Figure 9 and again confirms the trend in Figure 8. This figure does not include the cordierite data because this material would have a negative  $K_i$ . The data falls into three regions, those with low  $K_i$  values which show a monotonic decrease with increasing  $K_i$ ; the alumina and zirconia composites containing large diamonds, with very high  $K_i$  values which were micro-cracked after specimen fabrication. The chromium carbide-diamond composites also with very high  $K_i$  values would have been expected to exhibit similar behaviour however microscopic

observations revealed considerable reaction between the diamond and the chromium carbide during fabrication. This undoubtedly reduced the size of the diamonds and changed the stoichiometry of the chromium carbide about the diamond and thereby the thermal expansion coefficient, both effects would reduce the calculated  $K_I$  value for this material.

The expressions given in equations (22 & 23) enables a basis for the development of very strong composites. This is most apparent for the cordierite–diamond composite where the lower CTE of the cordierite leads to tensile stresses within the diamonds (150–250 MPa) and compensating compressive stresses in the matrix. The maxima in strength of this system at 30 volume % diamonds corresponds with the maximum achieved density of these composites. At higher volume fractions of diamonds the porosity of the composites increases substantially which leads to a reduction in the residual stress component and an increase in the flaw size. As discussed by Lutz and Swain<sup>31</sup> another attractive feature of these materials is that those materials that have a high  $K_I$  display excellent thermal shock resistance and contact damage tolerance.

## Conclusions

This study has provided a relatively simple basis for the prediction of the strength of dispersion containing brittle composites. A simple expression is derived that appears to reasonably well rationalise a large variety of strength data for duplex ceramics developed by Lutz & Claussen as well as a large variety of ceramic–diamond composites. The analysis provides a good estimate of the strength of the dispersion containing composite provided the matrix is not microcracked due to too severe residual stresses developed after fabrication. Also it does not take into account any reaction or reduction of inclusion size as observed with the chromium carbide diamond composites. Further extensions of the simple analysis developed here to include the observed R–curve behaviour are required before a completely satisfactory rationalisation of the strength of these dispersion containing ceramics is established.

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