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Observations on the nature of micro-cracking in brittle composites

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The degree of micro-cracking in BeO–SiC composites due to internal stresses which arise from the mismatch in the coefficients of thermal expansion was monitored by measurements of the thermal diffusivity by the laser-flash technique. The experimental results indicated that micro-cracking was most extensive at approximately 30 and 80 wt % SiC and a minimum at nearly 50 wt % SiC. A theoretical analysis indicated that the magnitude of internal stress increases linearly with SiC content, so that the above observations cannot be attributed to a low internal stress state at ~ 50 wt % SiC. Instead, this effect can be attributed to changes in the statistical variables affecting the brittle fracture as well as the degree of internal stress relaxation. Both these factors are thought to be controlled by the nature of multiaxial stress distribution. At ~ 50 wt % SiC-content, due to anticipated non-hydrostatic triaxial stress distribution, residual stress relaxation is possible in both the components of the composite. However, at low and high fractions of SiC content, such stress relaxation is less likely to occur due to the expected hydrostatic stress distribution in one of the components.

1. Introduction

The development of composites has led to materials with advantageous properties not found in single-phase materials. Mismatches in the coefficient of thermal expansion of the individual component within the composite can lead to the development of high levels of internal stresses on cooling the composite from its manufacturing temperature [1–7]. In composites in which one of the components is a polymer or a metal, relaxation of such internal stresses can occur by viscous or plastic flow even at relatively low temperatures. In brittle composites, internal stress relaxation can occur by diffusional creep, but only at high temperatures near the manufacturing temperature. Below these temperatures no stress relaxation can occur so that in brittle composites, for a given degree of mismatch of the coefficients of thermal expansion, the magnitude of the internal stresses will be particularly high. Such stresses, if sufficiently large, can lead to extensive "micro-cracking." Such micro-cracking can occur either at the boundaries between the components or within the component with the higher coefficient of thermal expansion which is generally subjected to the high value of tensile stress. It should be noted that extensive micro-cracking can also occur in single-phase non-cubic polycrystalline materials which exhibit a large degree of anistropy in thermal expansion behaviour [8, 9].

Micro-cracking in brittle composites or polycrystalline materials is not necessarily disadvantageous. In fact, it can lead to major improvements in many engineering properties. As observed experimentally, micro-cracking can lead to a significant increase in fracture toughness [10], presumably

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due to the formation of a process-zone ahead of a propagating crack. Micro-cracking can also lead to a major decrease in elastic behaviour [10–12] in addition to that in the thermal conductivity and diffusivity [13, 14]. Due to a large increase in the strain-at-fracture in combination with a stable mode of crack propagation, micro-cracked solids appear to be excellent candidate materials for use at high temperatures in applications involving severe thermal shock [15, 16]. In particular, micro-cracked materials are probably the only class of engineering materials appropriate for conditions which require high thermal shock resistance in combination with good thermal insulating ability [16]. Finally micro-cracking can substantially enhance the machinability of brittle materials to such an extent that they can be shaped by conventional metal working techniques [17].

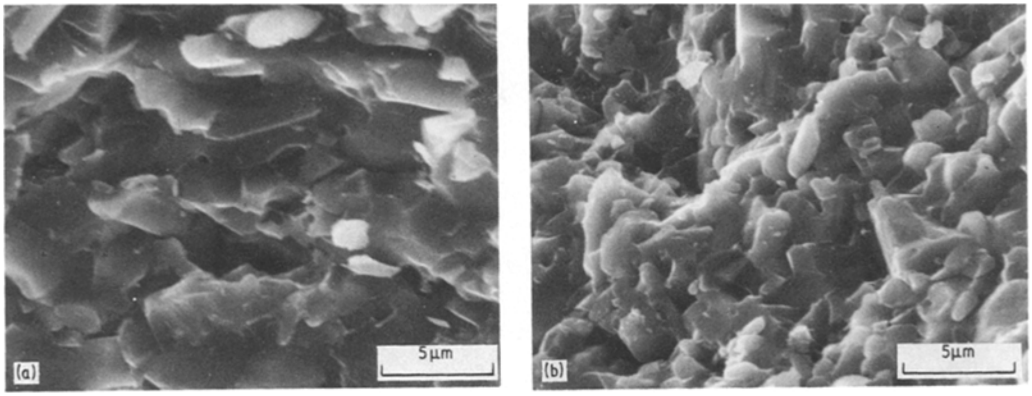
As indicated by a large number of literature studies [1–9], methods for the calculation of the magnitude and distribution of internal stresses which arise from non-uniformities of the coefficients of thermal expansion in composites or polycrystalline materials appear to be well developed. In general, these calculations show that the magnitude of these internal stresses is a function of both the mismatch or degree of anistropy of the thermal expansion coefficients and the range of temperature over which the composite or polycrystalline material is cooled without any stress relaxation, as well as being a function of the phase distribution of the individual components or the relative orientation of the individual grains within the polycrystal. Of significance to the present study is that these studies show that the magnitude of the internal stresses for a given composite or non-cubic polycrystalline material is not a function of the size of the components or the grains. For this reason, if micro-cracking were controlled solely by the magnitude of internal stress, the degree of micro-cracking in terms of the number of micro-cracks formed should be independent of the scale of the micro-structure.

This latter conclusion, however, contradicts experimental observations. As observed by Davidge and Green [18], for a continuous matrix dispersed phase system, a minimum dispersion size was necessary for micro-cracking to occur. Also, in polycrystalline materials with high thermal expansion anistropy, a critical grain-size is required before micro-cracking can take place. It appears then that the presence of internal stresses is a necessary but not sufficient condition for micro-cracking and that other factors need to be considered.

In providing an explanation for their observations, Davidge and Green [18] noted that the elastic energy available for crack propagation was proportional to the third power of the inclusion size. However, the total energy required to propagate a radial crack is proportional to the crack size. For this reason, below a given inclusion-size insufficient elastic energy was available for the formation of a micro-crack of sufficient size to give sufficient stress relief. A similar explanation was offered by Kuszyk and Bradt [10] and extended by Case *et al.* [19] to explain the grain size effect on micro-cracking in polycrystalline single-phase materials. It should be noted, however, that these explanations consider only the energy conditions after micro-crack formation, but do not address the condition of the onset of formation of micro-cracks.

Lange [20] based his analysis of micro-crack formation on an energy balance approach and found that, below a given value of the product of the square of the stress and the particle radius, micro-crack formation cannot occur, regardless of the size of the pre-existing flaw from which the micro-crack originated.

More recently, Evans [9] has pointed out that micro-crack formation most likely occurs from precursor flaws in the form of pores at grain-boundaries or triple-points. The size of such pores is frequently directly related to the grain-size. For this reason, the larger the grain for a given level of internal stress, the larger will be the stress intensity factor (K_I) for the precursor flaw. Micro-cracking will occur whenever $K_\mathrm{I} \geqslant K_\mathrm{Ic}$, the critical stress intensity factor. This latter mechanism provides a ready explanation for the observed component or grain-size effect on the incidence of micro-cracking in brittle materials. It is expected that micro-cracking originating from pores at triple-points or grain boundaries would be governed by statistical variables such as the size distribution and orientation of the pores as well as the magnitude and distribution of the internal stresses. Unfortunately, in assessing these effects experimentally, a detailed statistical distribution of the orientation, geometry and size of such pores is difficult to obtain. Furthermore, solutions for internal stresses are available only for the most simple phase distributions or grain geometries.



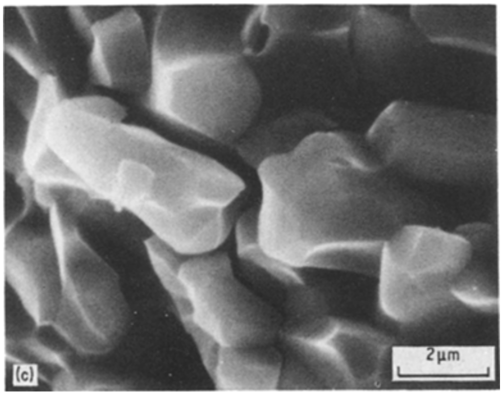


Figure 1. Scanning electron fractographs of BeO–SiC composites showing the triple-points and micro-cracks initiating from triple-point. Composition: (a) 30 wt % SiC and (b) 50 wt % SiC (c) 80 wt % SiC.

Relaxation of the internal stresses by diffusional processes should also have a pronounced effect on micro-crack formation [21]. The rate of such internal stress relaxation is an inverse function of the particle- or grain-size. For this reason, the degree of micro-cracking is expected to show a particle- or grain-size dependence in materials in which internal stress relaxation can occur.

The degree of internal stress relaxation, and therefore the extent of micro-cracking, is also a function of the residual stress state. As shown by Koeller and Raj [22] and Mori, Okabe and Mura [23], when the stress state in the particle or grain is hydrostatic, a state of minimum energy is achieved so that no stress relaxation will occur. In other words, internal stress relaxation requires a non-hydrostatic stress distribution.

This paper presents experimental data which are indicative of the existence of this latter effect.

2. Experimental procedure

Nearly fully-dense samples of composites of BeO and SiC made by hot-pressing were obtained from a commercial source.* Specimens of fully dense Sic made by conventional sintering were also obtained. Fig. 1 shows scanning electron micrographs of composites containing 30, 50 and 80 wt % SiC. The grain-size of BeO and SiC was below 5 μm. Due to the relatively large mismatch in their coefficients of thermal expansion, BeO–SiC composites exhibit extensive micro-cracking. As shown in Fig. 1, the isolated intergranular cracking is frequently observed to initiate from a triple-point and propagate along one or two adjacent grain faces, in agreement with Evans [9].

As shown analytically [24], micro-cracking has a pronounced effect on thermal conductivity. For this reason, the degree of micro-cracking was monitored by measurements of the thermal diffusivity by the laser-flash method [25], using equipment described elsewhere [13]. Measurements were made over a range of temperature from about 300 to 1400° C. One particular advantage of using the measurement of thermal diffusivity for monitoring the extent of micro-cracking is that the results are not affected by additional micro-crack formation due to mechanical loading, as is expected to be the case during measurements of strength and fracture toughness.

3. Experimental results

Fig. 2 shows the experimental data for the thermal diffusivity as a function of temperature for BeO, SiC and two of the BeO–SiC composites. The data shown in Fig. 2 were obtained by heating the specimens slowly from room temperature to

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*Ceradyne, Inc., Santa Ana, CA, USA; Code Ceralloy 2700 Series.

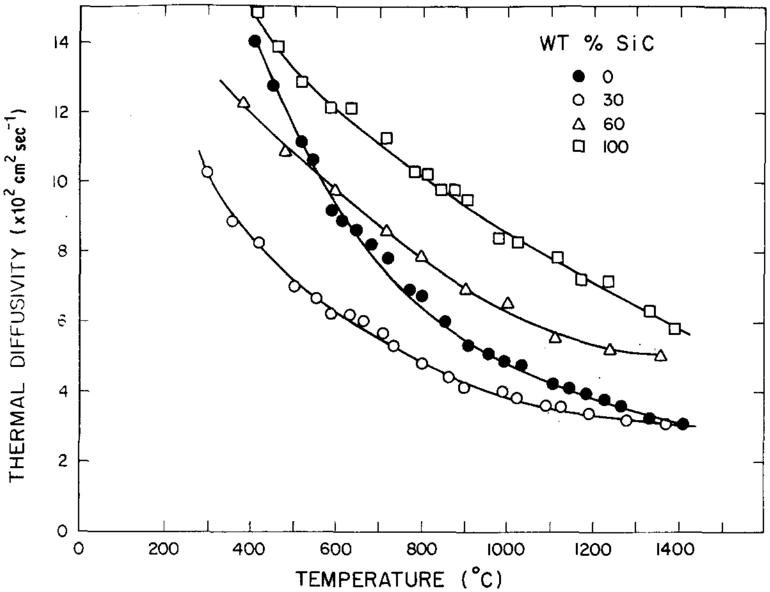

Figure 2 Thermal diffusivity of BeO–SiC composites as a function of temperature.

1400° C. On cooling, a hysteresis effect was noted. A detailed analysis of this latter observation is deferred to a later publication.

Fig. 3 shows the thermal diffusivity as a function of SiC content for a number of temperatures as obtained by smooth curves drawn through the experimental data.

Fig. 4 shows the value of the thermal conductivity (K) at 400° C calculated from the corresponding curve for the thermal diffusivity in Fig. 3 using the relation

$$K = \alpha \rho c, \tag{1}$$

where α is the thermal diffusivity, ρ is the density and c is the specific heat. The values of specific heat and density as a function of SiC content were assumed to vary linearly between the end values for SiC and BeO.

Included in Fig. 4 is the value for the thermal conductivity as a function of SiC content for a non-micro-cracked composite calculated from the Rayleigh–Maxwell relation [26, 27]

$$K_\mathrm{c} = K_\mathrm{m} \frac{1 + 2V(1 - K_\mathrm{m}/K_\mathrm{p})/(2K_\mathrm{m}/K_\mathrm{p} + 1)}{1 - V(1 - K_\mathrm{m}/K_\mathrm{p})/(K_\mathrm{m}/K_\mathrm{p} + 1)}, \tag{2}$$

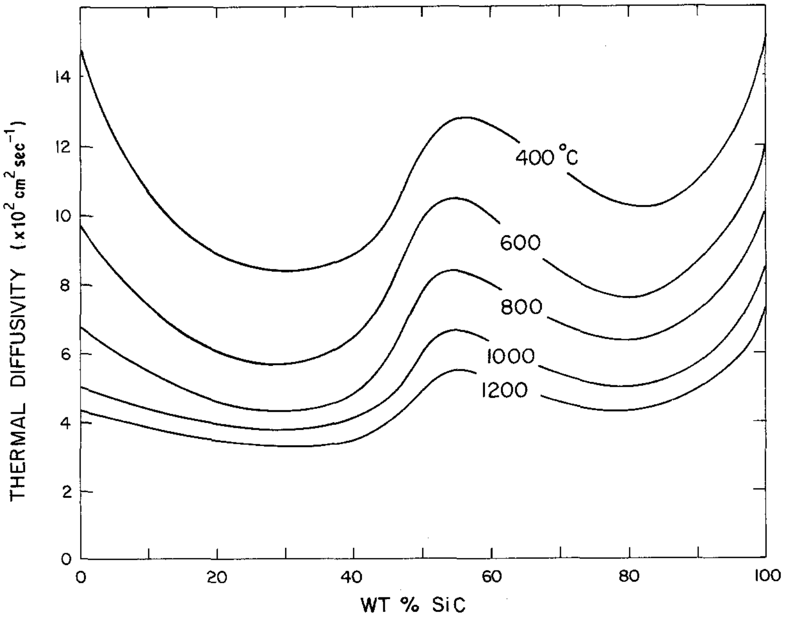

Figure 3 Effect of SiC content on thermal diffusivity of BeO–SiC composites.

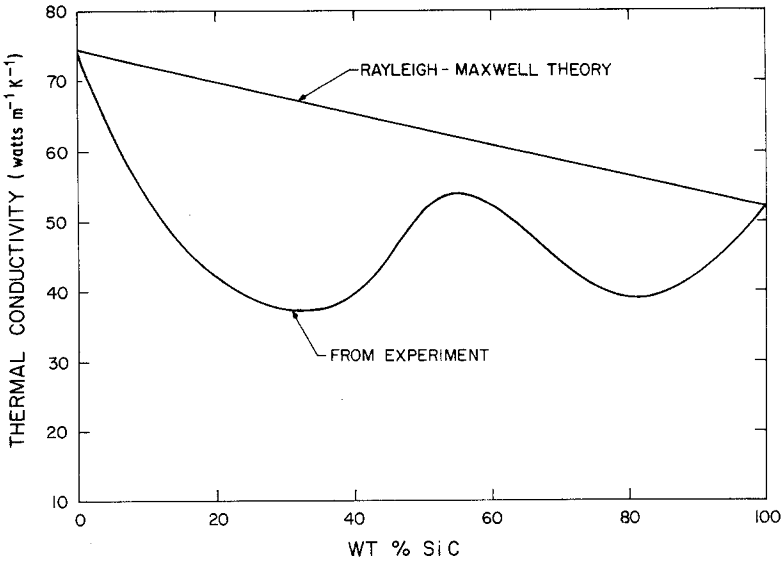

Figure 4 The values of thermal conductivity of BeO–SiC composites as a function of SiC content calculated from the diffusivity values in Fig. 3 at 400° C. Included in the figure are also the calculated values for thermal conductivity based on Rayleigh–Maxwell theory.

where K is the thermal conductivity and the subscripts c, m and p refer to the composite, matrix and dispersed phases respectively. For the numerical calculation of conductivity, given by Equation 2, the following data were used: SiC, $\rho = 3.21$ g cm^{-3}, $c = 0.255$ cal g^{-1} K^{-1} and BeO, $\rho = 3.008$ g cm^{-3}, $c = 0.408$ cal g^{-1} K^{-1}.

4. Discussion and conclusions

The discussion will concentrate primarily on the microstructural and mechanical variables which affect the formation of micro-cracks.

The thermal conductivity for the BeO–SiC composites calculated from the Rayleigh–Maxwell theory varies monotonically between the values of the thermal conductivity of the BeO and SiC. In fact, regardless of the phase-distribution of each component, the composite thermal conductivity should fall between the corresponding values of the two components [28]. For this reason the value for K for the SiC represents the lower bound on the thermal conductivity of the composite regardless of volume fraction. However, the variation of the thermal conductivity as a function of silicon carbide content inferred from the experimental data for the thermal diffusivity lies below the value for the SiC. Since these composites are fully-dense (or nearly so), this effect cannot be attributed to the presence of a pore phase. Instead, the decrease of the thermal conductivity must be attributed to the presence of the micro-cracks.

It is of particular interest to note that the decrease in thermal conductivity is most pronounced at approximately 30 and 80 wt% SiC. In contrast, at approximately 50 wt% SiC the decrease in thermal conductivity is rather small, if existent at all. This observation suggests that micro-cracking is most pronounced at 30 and 80 wt% content of SiC and appears to be absent at approximately 50 wt% SiC. Support for this latter conclusion is provided by experimental data [29] made available to the present authors prior to publication for the strength and fracture energy for these composites. At approximately 20 and 75 wt% SiC, these composites have higher values for tensile strength (measured in bending) and fracture energy as compared to the values for single-phase SiC and BeO. At 40 wt% SiC, however, the values of strength are comparable. In other words, at these compositions which exhibit a decrease in thermal conductivity, a corresponding increase in strength and fracture energy is found. The minima in the strength and fracture energy plots, however, occur at a slightly lower SiC content than the corresponding SiC content for the maxima in the diffusivity plot. This is believed to be due to the fact that, during the strength and fracture energy measurements, additional micro-cracks could have been formed during the loading period. These opposing effects are entirely consistent with the presence of micro-cracks which are known to enhance fracture toughness [10] and to decrease thermal conductivity [24]. Further supporting evidence for the low degree of micro-cracking can be obtained from the experimental data for the coefficient of thermal expansion of Be–SiC composites measured by Rossi [30]. At 50 vol% SiC the coefficient of thermal

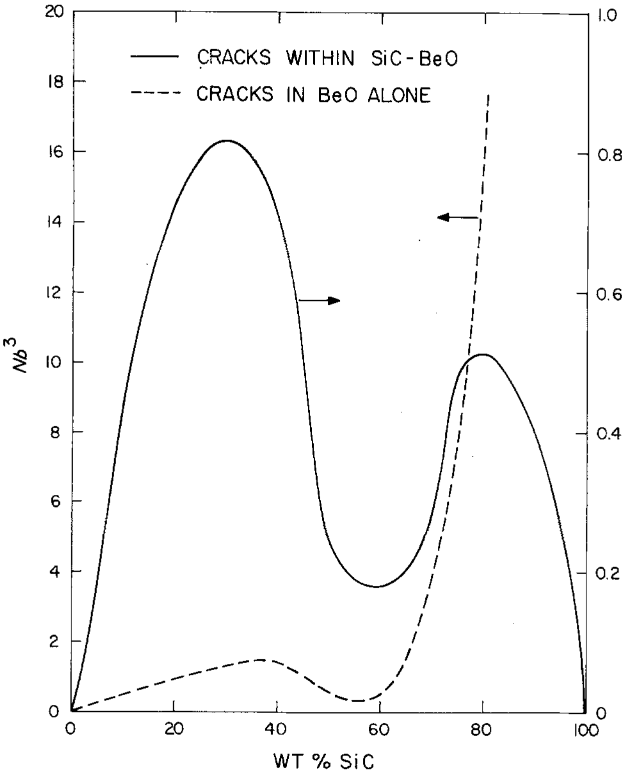

Figure 5 The effect of SiC content on the values of Nb^3 as calculated from Equations 2 and 3.

expansion lies relatively much closer to the coefficient of thermal expansion calculated from composite theory than at 25 and 75 vol % SiC.*

As shown analytically, the effect of randomly orientated circular cracks of equal size on the thermal conductivity is given by [16]

$$K = K_0(1 + 8Nb^3/9)^{-1}, \qquad (3)$$

where N is the number of cracks per unit volume, and b is the crack radius. Unfortunately, the density, size and geometry of the micro-cracks cannot be determined with any accuracy by metallographic methods. For this reason, the decrease in thermal conductivity cannot be estimated from prior information. Instead, using Equation 3, the value of Nb^3 can be assessed from the experimental data for thermal diffusivity. These results are shown in Fig. 5 for the case of uniformly distributed micro-cracks within the composite as a whole. Since the micro-cracks are more likely to be found within the BeO phase, Nb^3 was also evaluated by means of Equation 2 in which the thermal conductivity of the BeO phase was assumed to be given by Equation 3. As indicated in Fig. 5, the value of Nb^3 for micro-cracks in the BeO phase can go to high values, as expected, since at higher fractions of SiC the BeO will exhibit the highest degree of micro-cracking. A value of $Nb^3 \simeq 0.5$ for uniformly distributed micro-cracking at ~ 80 wt % SiC-content suggests that for a micro-crack radius equal to the grain-size (~ 5 μm) approximately one half of the BeO grains contain a micro-crack. It should be noted that an estimate of the thermal conductivity of a micro-cracked composite obtained by substitution of Equation 3 in Equation 2 for the conductivity of the micro-cracked phase assumes that the effect of the micro-cracking and the presence of the dispersions on the thermal conductivity, in fact, are uncoupled. This latter assumption, however, must be considered to be only an approximation since the micro-cracking in the BeO phase is expected to occur in the immediate vicinity of the SiC matrix. An analysis of the coupling effect of dispersions and micro-cracking on thermal conductivity is recommended as a fruitful area for futher theoretical and experimental work.

The question as to why, at intermediate volume fractions of the SiC phase, micro-cracking appears to be largely absent, still remains to be answered.

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*It should be noted that Rossi draws a smooth curve through his data. Close examination of the experimental data, however, suggests that a better curve would have the same general form as the curves in Fig. 3.

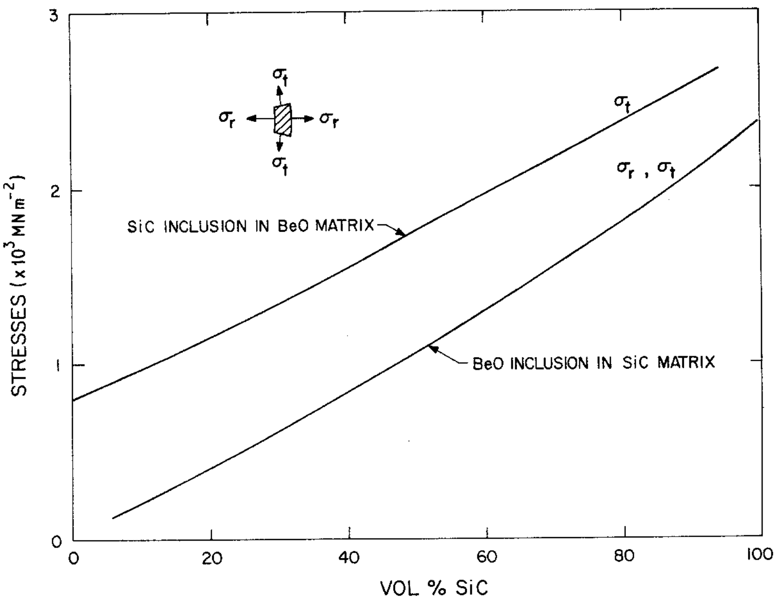

Figure 6 Calculated values of tensile stresses in the BeO phase at the BeO–SiC interface based on the composite sphere model. σ_r and σ_t represent radial and tangential stresses respectively.

One explanation which could be offered is that, near 50 wt% SiC, the number or size of the precursor micro-cracks is smaller than at the other compositions due to enhanced densification during hot-pressing. This effect, however, must be rejected as a primary explanation since the kinetics of densification due to the high flow resistance of silicon carbide are expected to decrease with increasing SiC content.

Two other hypotheses can be advanced for the apparent lack of micro-cracking at the intermediate volume contents of silicon carbide, based on changes in the manner in which the two components are distributed within the composite and on the corresponding changes in magnitude and distribution of the internal stresses. At the low and high volume contents of silicon carbide, the phase distribution is expected to consist of isolated particles of silicon carbide or beryllium oxides in continuous matrices of beryllium oxide or silicon carbide, respectively. At volume fractions of silicon carbide near fifty per cent, however, based on considerations of packing factors of well-mixed *equidimensional* powder particles, silicon carbide and beryllium oxide dispersions are expected to be in contact, such that both materials will form continuous phases. It is expected that the residual stresses for the latter phase distribution can differ significantly from those for isolated dispersions of one material contained within the continuous phase of the other.

In order to check the validity of the latter statement, the internal stresses in the BeO–SiC system were evaluated on the basis of two simple mechanical models. The mechanical model considered most appropriate for the low and high volume fractions of silicon carbide, consisted of a composite sphere, with the silicon carbide as the inner and outer sections of the sphere, respectively. At the intermediate volume fractions of silicon carbide, the internal stresses were evaluated using a composite concentric cylindrical model. Admittedly, this latter model represents an oversimplification of the far more complex phase geometry within the composite. Nevertheless, it reflects the continuous nature of the distribution of both the silicon carbide and beryllium oxide. Furthermore, the evaluation of internal stresses for this model is easily obtained by analytical methods, as presented by Gatewood [31]. The following property data were used for the calculations of the internal stresses: $E_{\text{SiC}} = 4.83 \times 10^5$ MN m^{-2}; $E_{\text{BeO}} = 3.79 \times 10^5$ MN m^{-2}; $\nu_{\text{SiC}} = 0.19$; $\nu_{\text{BeO}} = 0.34$; $\alpha_{\text{SiC}} = 5.4 \times 10^{-6}$ °C^{-1} and $\alpha_{\text{BeO}} = 10^{-5}$ °C^{-1}. A temperature difference of 1000° C was assumed as a conservative estimate of the range of temperature in cooling over which purely elastic behaviour is assumed for the composites.

Figs 6 and 7 show the calculated values of the maximum tensile stresses in the beryllium oxide phase as a function of silicon carbide content for the composite sphere and cylinder models, respectively. Examination of these data reveals that for both models, the value of internal stress in the BeO increases with increasing SiC content. For this reason, the low degree of micro-cracking near

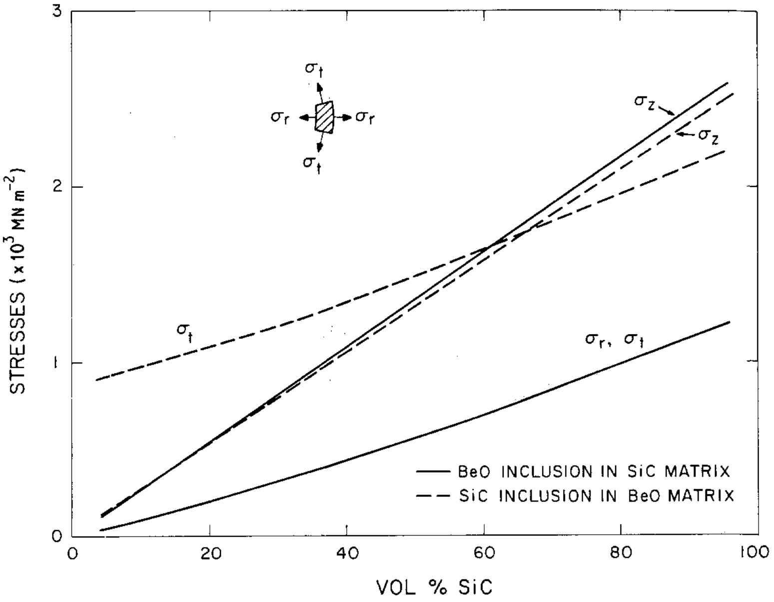

Figure 7 Calculated values of tensile stresses in the BeO phase at the BeO–SiC interface based on the composite cylinder model. σ_r, σ_t and σ_z represent radial, tangential and axial stresses respectively.

50 wt % SiC cannot be attributed to a decrease in magnitude of internal stresses if these could be described by any one of the two models over the total range of SiC content. It should be recognized, as discussed earlier, that the composite cylinder model is expected to describe the internal stress state near 50 wt % SiC to a better degree than the composite sphere model, which is more appropriate at low and high contents of silicon carbide. On this basis, comparison of the data for the internal stresses for both models shows that near 50 vol % SiC, both the tensile internal stresses in the BeO surrounding a SiC inclusion in the composite cylinder are approximately 20% lower than the corresponding stresses in the composite sphere model. This observation by itself could be sufficient to explain the experimental observations for those phase distributions best described by a SiC inclusion in a BeO matrix. However, at least two additional effects related to the nature of the internal stress distributions are expected to be operating as well.

For the first effect, it should be noted that for the composite sphere, the tensile stresses in the BeO are uniform biaxial for the SiC inclusion and uniform triaxial for the BeO inclusion. In contrast, for the composite cylinder model, uniform biaxiality of the internal stresses is encountered near 70 vol % SiC, for the SiC inclusion within the BeO matrix only. As indicated by an extensive literature [32–38], the effect of stress state can have a profound effect on the stress level at which tensile fracture occurs. The tensile failure stress for a uniform biaxial stress state can be considerably less than the failure stress under uniaxial conditions. Presumably for a triaxial tensile stress state even lower fracture stresses can be observed. These effects are related to the probability of finding a crack orientated nearly perpendicular to the direction of the applied stress. For a uniform triaxial stress state, propagation of a flaw can be induced regardless of its orientation. The same is true for a uniform biaxial stress state for any flaw orientated perpendicularly to the plane in which the stress acts. For non-uniform multiaxial stress states, the stress ratio* and the orientation of the flaws with respect to the direction of the maximum stress is important for fracture initiation. Applying these general observations to the present study, it is expected that micro-crack formation in brittle composites is more likely to occur in uniform multiaxial than in non-uniform multiaxial internal stress fields for the same value of maximum stress. For this reason, the number of micro-cracks formed near 50 wt % SiC-content in the cylindrical model with a stress ratio ~ 0.8 is expected to be less than in the spherical model with a stress ratio of unity, based on the difference in the value of the peak stress alone. This effect is also expected to occur for the BeO inclusion in the SiC matrix. A comparison of Figs 6 and 7 indicates that the axial stress in the composite

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*Stress ratio indicates the ratio of the stresses in different directions with repect to the smallest stress.

cylinder is higher than the value of stress in the composite sphere. For this reason, based on the magnitude of stress alone, the composite cylinder should exhibit more extensive micro-cracking than the composite sphere. However, for the composite sphere, the stress state in the BeO is uniform triaxial, whereas in the composite cylinder the stress is non-uniform triaxial with stress ratios of 2:1:1. The relative contributions of the effect of magnitude of stress or flaw orientation on micro-crack formation will be a function of the total number of precursors available for micro-crack formation. In brittle composites, this number is expected to be only one or at most a few per inclusion. For this reason, at stress conditions which are more uniaxial, such as for the BeO inclusion in the composite cylinder, the fraction of inclusions with a properly orientated micro-crack precursor is expected to be much less than that in the uniform triaxially stressed condition for the composite sphere. If so, the effect of orientation of micro-crack precursor is expected to play a major role in determining micro-crack formation. Clearly a fully quantitative description of these relative effects will require a complete statistical assessment of all micro-crack precursors within a given composite, including their size, geometry and orientation. In addition, a complete description of the residual stress state for each precursor as well as the corresponding stress intensity factors will be required.

The second effect of the stress state on micro-crack formation which must be considered is the possibility of the relaxation of the internal stresses during cool-down from the manufacturing temperature. Such stress relaxation could occur by diffusive or other processes at levels of temperature of the composite which are still high enough for such processes to occur. As discussed earlier, whether or not such relaxation can occur depends on the state of residual stress. Under conditions of uniform triaxial (hydrostatic) state of stress, the inclusion is in a state of minimum energy and no opportunity exists for stress relaxation within the inclusion. This condition applies to the composite sphere model which, in the present case, is appropriate at the low and high volume contents of silicon carbide. The stress state in the composite cylinder model, appropriate for the intermediate volume fractions of silicon carbide, is not hydrostatic such that internal stress relaxation is feasible in both components. For this reason, depending on the rate of cooling, the stress values for the composite cylinder shown in Fig. 7 are overestimates of the stress levels found in the actual composite. In particular, considerable relaxation of the axial stress in the BeO inclusion could occur. If so, the internal stresses in both the BeO and SiC in the cylinder composite, would fall below the corresponding values in the composite sphere. A quantitative assessment of this effect would require detailed information of the diffusion characteristics of each component within the composite as well as exact details on temperature history followed during processing [39].

In making an admittedly subjective comparison of the relative importance of the various mechanisms, the writers feel that the internal stress relaxation is the most probable mechanism to explain the experimental observations.

In general, then, the present results and discussion suggest that theories for the formation of micro-cracks should include the statistical aspects of brittle fracture such as the size distribution of the precursor flaws, their orientation and the state of residual stress fields as well as the degree of stress relaxation. In addition, the stress relaxation and the residual stress field being time dependent, the degree of micro-cracking and the associated properties of the micro-cracked materials are expected to be a function of the thermal history during the fabrication and cooling processes.

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