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Mechanical Properties of $Si_3N_4 + \beta\text{-}Si_3N_4$ Whisker Reinforced Ceramics

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Abstract

The mechanical properties of $Si_3N_4 + \beta\text{-}Si_3N_4$ whisker reinforced ceramics were studied at room and elevated temperatures up to 1200°C. The four-point bend strength, Weibull modulus and fracture toughness values were analysed in connection with technological defects and fracture mechanisms. The main fracture origins were clusters of Si_3N_4 whiskers and the main toughening mechanisms were crack deflection, whisker/matrix debonding and to a lesser extent crack branching and whisker pull-out. The experimentally achieved toughening was compared with those theoretically predicted. The strength degradation was caused by subcritical crack growth at elevated temperatures.

Die mechanischen Eigenschaften eines mit $\beta\text{-}Si_3N_4$-Whiskern verstärkten Si_3N_4-Werkstoffs wurden von Raumtemperatur bis zu 1200°C untersucht. Die 4-Punkt-Biegefestigkeit, der Weibullmodul und die Bruchzähigkeit wurden bestimmt und mit Fabrikationsfehlern und den vorherrschenden Bruchmechanismen korreliert. Hauptsächlich konnten als bruchauslösende Fehler Anhäufungen von $\beta\text{-}Si_3N_4$-Whiskern beobachtet werden. Die vorherrschenden Verstärkungsmechanismen waren Rißablenkung, Risse an der Whisker/Matrix-Grenzfläche und, in geringerem Maße, Rißverzweigung und Whisker-pull-out. Die experimentell gefundene Verstärkung wurde mit theoretischen Vorhersagen verglichen. Die Festigkeitsabnahme wurde durch unterkritisches Rißwachstum bei höheren Temperaturen verursacht.

On a étudié les propriétés mécaniques de céramiques renforcées par des whiskers, $Si_3N_4 + \beta\text{-}Si_3N_4$, à température ambiante et à jusqu'à 1200°C. La résistance à la flexion 4 points, le module de Weibull et la ténacité sont analysés en relation avec les défauts technologiques et les mécanismes de fracture. Le principal facteur de fracture est constitué par les agrégats de whiskers Si_3N_4 et les principaux mécanismes de renforcement sont la déviatiation des fissures, l'interaction fibre-whisker et, dans une moindre mesure, le branchement en front de fissure et l'expulsion (pull-out) des whiskers. Le renforcement réalisé expérimentallement est comparé avec celui prédit théoriquement. La dégradation de la résistance est causée par la croissance sous-critique de fissures aux températures élevées.

1 Introduction

Recent studies of whisker-reinforced composite ceramics have shown that improvements in mechanical properties, mainly in fracture toughness and in slow crack growth resistance, can be achieved by the incorporation of strong, small-diameter whiskers into the ceramics matrix.[1–3] The toughening of ceramics by whiskers usually includes contributions from crack deflection, whisker/matrix debonding, crack bridging and whisker pull-out. The results have shown that the toughening is strongly dependent on the volume fraction, shape, size and strength/toughness of the whiskers. The contribution of whisker reinforcement can be described by analyses

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of the whisker bridging zone behind the crack tip.[4,5] Various attempts have been made at modelling the toughening effects and to compare the predicted and experimental data of composite whisker toughened ceramics with Al_2O_3, Si_3N_4 and mullite matrix and SiC whiskers. Some authors[6] are of the opinion that the toughness of the alumina based ceramics increases with the increase in whisker strength, volume fraction of the whiskers, whisker radius, the ratio of Young's modulus of the composite to that of the whisker, and the ratio of the matrix to interface fracture energy. The fracture toughness increase is described by the relation[6]

$$dK_{wt} = \sigma_w\{[V_f r/6(1-v^2)](E_c/E_w)(\gamma_m/\gamma_i)\}^{1/2}$$

where σ_w and V_f are the tensile fracture strength and volume fraction of whiskers, E_w and E_c are the Young's moduli of whisker and composite, v is the Poisson ratio, γ_m and γ_i are the fracture energies of the matrix and the interface, respectively.

According to Campbell *et al.*,[5] the steady-state toughening increase due to SiC whisker reinforcement can be interpreted as arising from four contributions, i.e. strain energy dissipation, loss of residual strain energy, debond fracture surface energy and whisker pull-out according to the relation:

$$dG/V_f d = \sigma_w^2/E - Ee_T^2 + 4(\gamma_i/r)/(1-V_f) + (\tau/d)(h_i^2/r)$$

where d is the debond length, e_T the misfit strain, h_i the pull-out length and τ the sliding resistance.

An increase in whisker/matrix debond length and pull-out were found when a decrease in the surface oxygen content and an increase in the whisker radius occurred.[6] The general opinion is that the interphase characteristics play the most important role in the debonding process and this problem should be the subject of further investigation. It also should be taken into consideration that during the development of composite whisker-reinforced ceramics, besides the toughening effects, attention should be paid to the optimization of processing parameters in order to achieve a material without defects. According to Refs 7 and 8 the processing of these systems often leads to the occurrence of several strength degrading defects with different sizes, up to 300 μm. These defects are mainly large particles, clusters of grains, whiskers, or impurities associated with the whisker production.

The main results from our previous work concerning the $Si_3N_4 + \beta$-Si_3N_4 ceramics can be summarized as follows:[9,10] $Si_3N_4 + \beta$-Si_3N_4 whisker composite ceramics with relatively high densities can be prepared with small additions of Al_2O_3 and Y_2O_3 by hot pressing for 1 or 2 h at 1750–1850°C, under a pressure of between 27 and 32 MPa. The addition of Si_3N_4 whiskers up to 20% have no influence on the densification behaviour, but at higher whisker contents the maximum attainable density was reduced significantly. The main defects in the studied systems were caused by the presence of large Si_3N_4 whiskers in the starting mixture. These form clusters and cause inhomogeneities in the distribution of the intergranular phase. An increase in the fracture toughness is observed with increasing whisker content up to 10%, and the main toughening mechanisms are crack deflection, crack branching, whisker/matrix debonding and to a lesser extent pull-out.

Besides the analysis of the influence of technological defects on the bending strength value or the microstructural influence on the fracture toughness, a study of the high temperature behaviour of these systems is also very important.

The present study has been carried out to evaluate the room temperature strength, high temperature strength and fracture toughness of $Si_3N_4 + \beta$-Si_3N_4 ceramics with 10% and 20% of whisker additions. The failure origins and the mode of crack propagation were examined in detail and the high temperature strength degrading mechanisms were analysed. The experimentally achieved toughening was compared with those theoretically predicted.

2 Experimental

In the present paper the $Si_3N_4 + \beta$-Si_3N_4 whisker systems with 10 and 20 wt% of whiskers were studied. The processing procedures and the results from microstructural analysis of these systems were described in our previous work.[9] For β-Si_3N_4 whisker (prepared by self-propagating high temperature synthesis (SHS) in the Institute of Structural Macrokinetic ASci USSR, Chernogolovka) characterization the scanning electron microscopy (SEM), transmission electron microscopy (TEM), X-ray diffraction (XRD) and electron spectroscopy for chemical analysis (ESCA) were used.

The flexural strength was evaluated up to 1200°C in air by using a four-point bending fixture with inner/outer span 7/20 mm, respectively. The crosshead rate was 0·1 mm/min. Samples were cut from a hot pressed billet with the tensile face perpendicular to the hot pressing direction. These were ground parallel to the long axis with a resin-bonded diamond wheel to 3 × 4 × 25 mm. The tensile surface was polished to 1 μm finish. The strength data were interpreted with the help of the Weibull statistics analysis.[11] Fracture toughness was determined using the indentation strength (ISB) method. The Vickers indentation (10 kg) was used on the tensile surface of the polished four-point bend specimens.

The indentation was done carefully to introduce cracks parallel with the long axis of the specimens. The stress intensity factor was calculated according to the relation:[12]

$$K_I = 0{\cdot}59(E/H)^{1/8}(\sigma_f P^{1/3})^{3/4}$$

where E is Young's modulus, H is hardness, P_i is indentation load and σ_i is bending stress. The cross-head rate during the fracture toughness test was 0·1 mm/min.

The microstructure of these materials was studied using light microscopy, SEM and TEM. Before the SEM examination samples were chemically etched at 740°C in a mixture of K_2CO_3 and NaF, or plasma etched. TEM specimens were prepared by diamond cutting, grinding and polishing, to produce 50 μm thick discs, which were then dimpled and ion beam-thinned. The specimens were coated with a thin layer of evaporated carbon before examination in the microscope. The fracture characteristics of the materials were studied by examining fracture lines and fracture surfaces using SEM. Main attention has been paid to the study of fracture origins at room and elevated temperatures using macrofractography and to the study of the toughening mechanisms, using micro- and stereofractography (study of the related fracture micro-areas on both fracture surfaces).

3 Results

3.1 Microstructure analysis

Analysis of β-Si_3N_4 whiskers shows that they are dimensionally straight with smooth surfaces and contain oxygen, carbon and iron as the main surface impurities.[13]

Results from microstructure analysis are in agreement with our previous work,[10] Fig. 1. We found only one type of technological defect and this was clusters of β-Si_3N_4 whiskers, Fig. 2. The other defects, such as size inhomogeneities in whiskers or in the intergranular phase and microporosity are often connected with clusters. Clusters formed by whiskers are about 30–50 μm in diameter. There was a significant difference in the number of clusters found in the system with 10 and 20% of whiskers. Statistical analysis showed that the material with 20% of whiskers contained about twice as many clusters as the material containing 10% of whiskers.

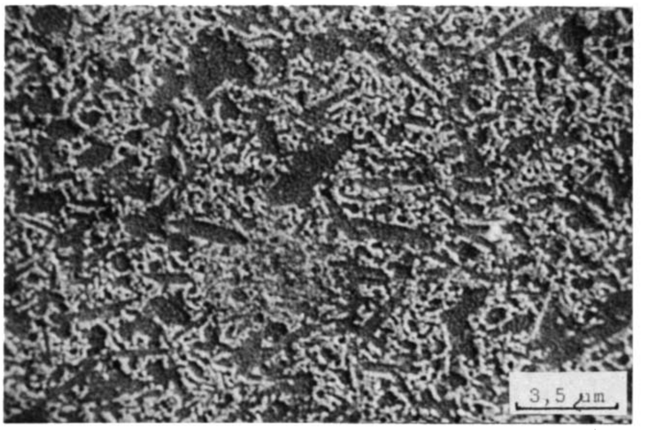


Fig. 1. Characteristic microstructure of Si_3N_4 + 20% of β-Si_3N_4 whisker ceramics.

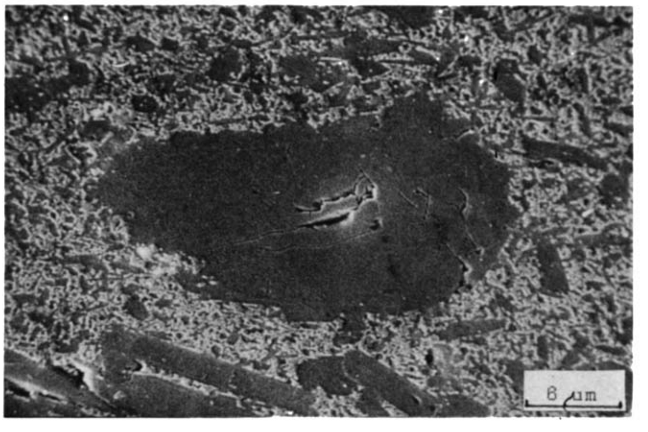


Fig. 2. Whisker agglomerate in Si_3N_4 + 20% of β-Si_3N_4 whisker ceramics.

Using a TEM, precipitates were usually found inside the large Si_3N_4 grains and only occasionally in the intergranular phase. The precipitates inside the Si_3N_4 grains contained Al and precipitates in the intergranular phase contained Fe, Cr and Ti.[10] A non-crystalline phase is generally dispersed throughout the microstructure at triple grain junctions and in most Si_3N_4/Si_3N_4 grain and Si_3N_4/Si_3N_4 whisker boundaries. The understanding of the nature of the whisker/matrix interface needs more detailed analysis by high-resolution electron microscopy.

3.2 Room temperature bending strength and fracture toughness

In order to determine the fracture strength at room temperature for materials with 10 and 20% of whiskers, 10 and 25 specimens were tested in four-point bending mode, respectively. Statistical variation of the fracture strength for the system with 20% of whiskers is shown in Fig. 3. The strength varied from a minimum of 421 MPa to a maximum of 584 MPa with an average value of 512 MPa. The

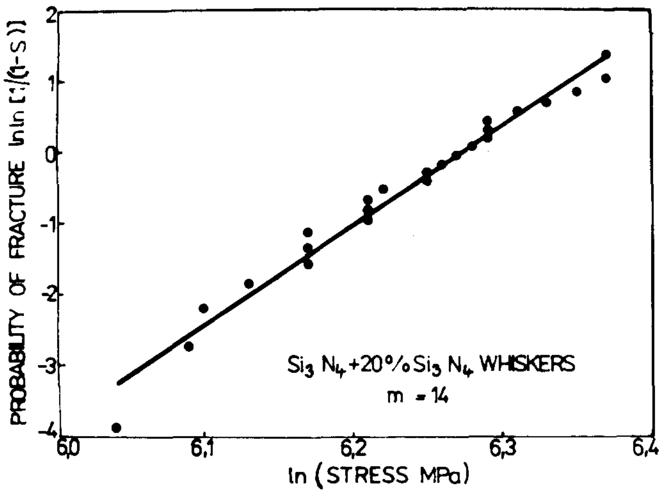


Fig. 3. Statistical variation in fracture strength for Si_3N_4 + 20% of β-Si_3N_4 whisker ceramics.

Weibull modulus for this material had a value of 14. Earlier strength evaluation of the yttria-doped hot pressed silicon nitrides (without whiskers) reported an average strength of 851 MPa with a Weibull slope of 12.[14] The lower average strength and higher Weibull slope in our case are caused by the presence of clusters. Examination of the fracture surface of this material reveals failure initiation sites as surface and subsurface defects in the form of above-mentioned clusters, Fig. 4. The size of these defects on the fracture surfaces is in agreement with the average dimension of those in the bulk material measured on the polished cross-sections. In most cases these clusters have an almost regular half-circle or circle shape, Fig. 5(a,b). As the microstructure analysis reveals and the fractographic analysis confirmed voids or cracks were often found between the whiskers inside the clusters. These are very dangerous from the point of view of crack initiation; especially in instances where the clusters are surface or near surface defects. In these cases the maximal tensile stresses together with the sharp microcracks on the tensile surface of the specimens caused a

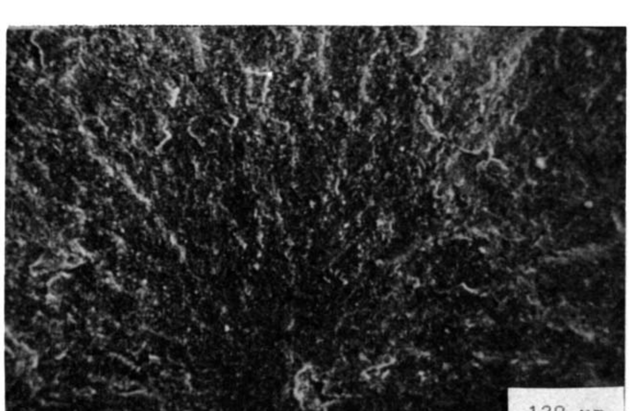
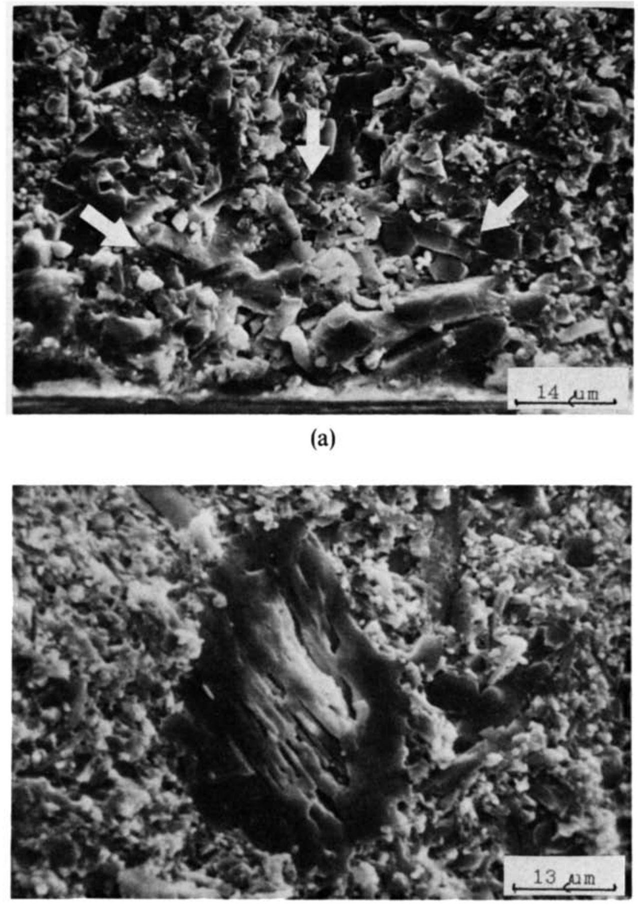


Fig. 4. Cluster of whiskers as the origin of fracture.



(b)

Fig. 5. Detail from the fracture origin: (a) subsurface defect; (b) volume defect.

significant strength degradation. On the other hand, the nature of these clusters, their similarity in shape, size and position, causes the relative low scatter of strength values and a relatively high Weibull modulus. The fractographic analysis of every sample from this series shows that in about 60% of cases the fracture was initiated by clusters of whiskers situated near the tensile surface. These were similarly sized and shaped. In residual cases the fracture origins were not remarkable.

The situation is a little different in the case of a system with 10% of Si_3N_4 whiskers. The statistical variation of fracture strength for this system, together with that for a system with 20% of whiskers is shown in Fig. 6. The strength values for this system varied from a minimum of 519 MPa to a maximum of 858 MPa, with a mean value of 676 MPa. The Weibull modulus for this system is 7·1. For this system, in 25% of the cases the fracture origins were very similar to those found in the system with 20% of whiskers, i.e. clusters of whiskers. For the remaining samples the fracture origins were small surface cracks, corners or undetermined items. These facts explain the higher strength and lower

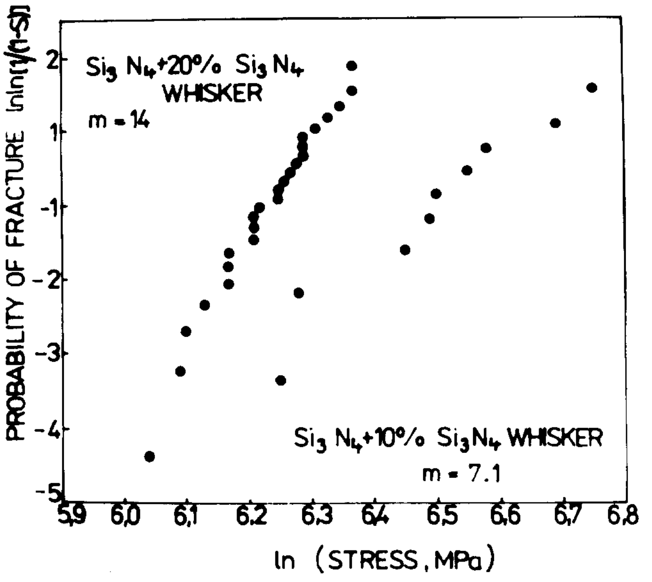


Fig. 6. Statistical variation in fracture strength for Si_3N_4 + 20% of β-Si_3N_4 whisker ceramics and for Si_3N_4 + 10% of β-Si_3N_4 whisker ceramics.

Weibull modulus for this system as compared with previous ones. Our recent experiments show that by elimination of the whisker agglomerates, a mean value of the four-point bend strength of 930 MPa was achieved.[15]

The room temperature fracture toughness for the systems with 10 and 20% of whiskers measured by the ISB method are $6{\cdot}4 \pm 0{\cdot}3\ \mathrm{MPa/m^{1/2}}$ and $6{\cdot}3 \pm 0{\cdot}2\ \mathrm{MPa/m^{1/2}}$, respectively. These values are a little lower than values achieved in the same systems (but not the same batches) by the indentation fracture method.[9,10] The differences can be caused by different testing methods. For the system without the addition of whiskers, processed exactly the same way as the whisker-containing systems, a fracture toughness value of $6{\cdot}1 \pm 0{\cdot}2\ \mathrm{MPa/m^{1/2}}$ was found. There is an agreement between the fracture toughness values achieved with indentation fracture and the ISB method as regarding their dependence on the whisker content. In both cases the system with 10% of whiskers has a higher fracture toughness than the system with 20% of whiskers. This is caused by the non-uniform distribution of whiskers. A homogeneous whisker distribution will probably lead to a further improvement in fracture toughness. After comparing the calculated and measured flaw sizes for the systems studied it is evident that the calculated ones are higher in all cases. This fact indicates the possibility of subcritical crack growth during the bending strength test and/or seriousness of the problem of application of the geometric constant Y for defects with different sizes and/or irregular shapes.[16]

3.3 Elevated temperature strength and fracture toughness

The high temperature flexural strength and fracture toughness were evaluated at temperatures of 800, 1000 and 1200°C. The variation in strength and fracture toughness values as a function of temperature is shown in Figs 7 and 8. Only a small decrease in the strength values in the system with 10% of whiskers was noted at 800°C in comparison to the room temperature strength. On the other hand, a sharp drop was noticed in the system with 20% of whiskers. At temperatures of 1000 and 1200°C the strength of the system with 10% of whiskers decreases to 450–500 MPa, while the strength of the system with 20% of whiskers decreases only slightly, when compared with the strength values at 800°C. The fracture initiation in the system with 10% of whiskers tested at 800°C was very similar to fracture initiation at room temperature. The fracture origins were clusters of whiskers and no significant subcritical crack growth region was found around the fracture origins. A different outlook on the fracture

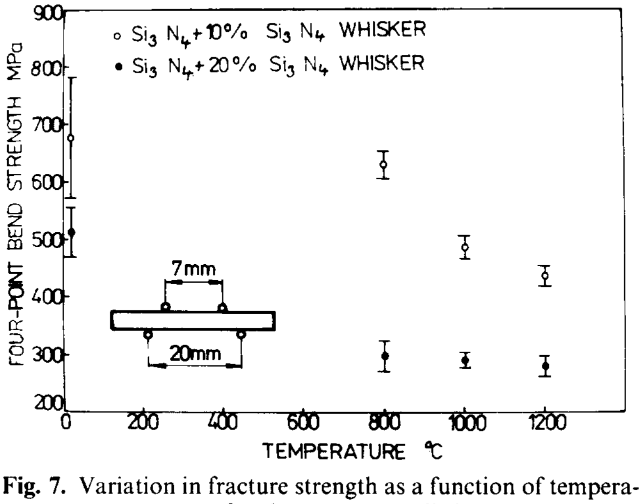


Fig. 7. Variation in fracture strength as a function of temperature for the systems studied.

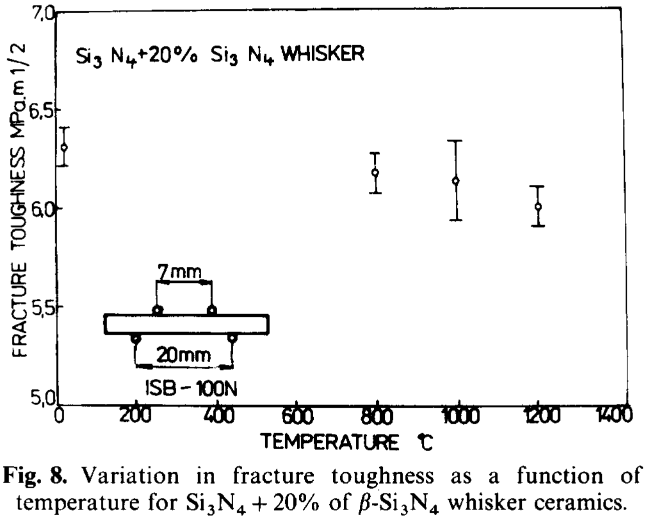


Fig. 8. Variation in fracture toughness as a function of temperature for Si_3N_4 + 20% of β-Si_3N_4 whisker ceramics.

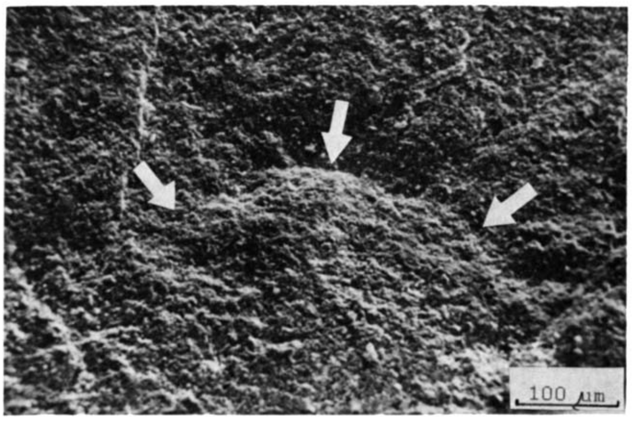


Fig. 9. SEM fractograph of the slow crack region.

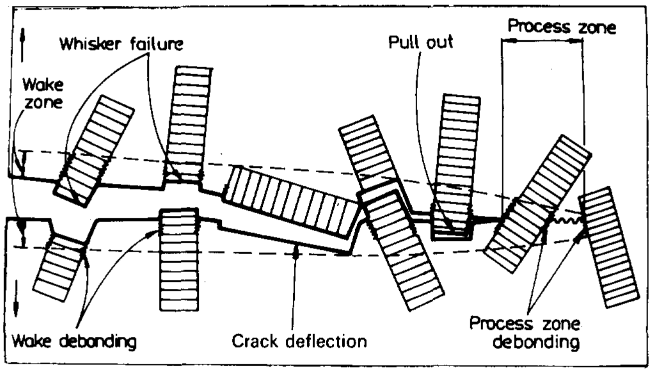
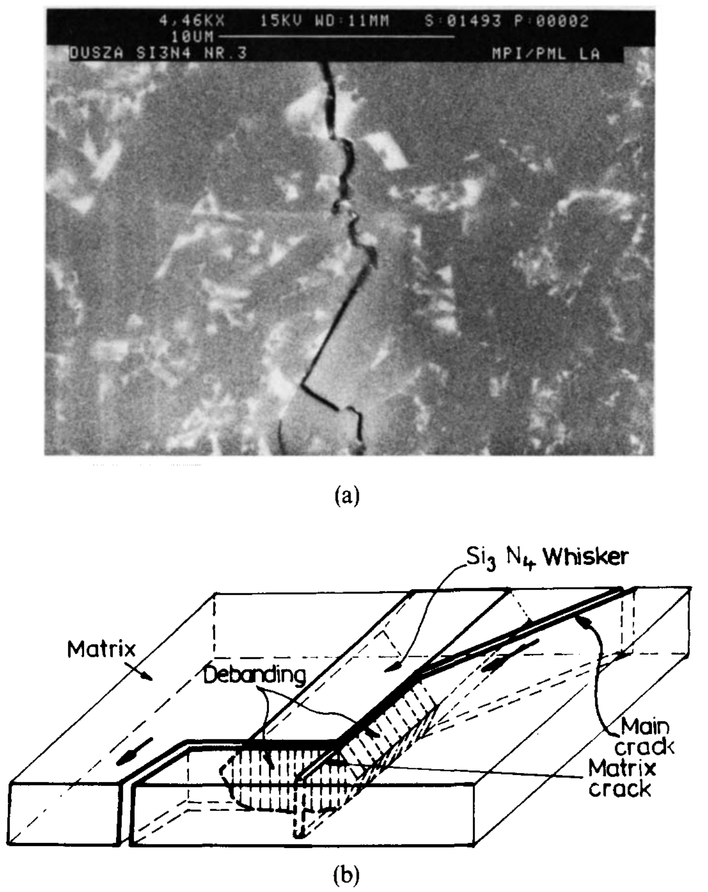


Fig. 10. Schematic illustration of crack propagation in whisker reinforced ceramics.

surface was found in the case of samples tested at 1000 and 1200°C. The fracture origins were surface defects which arose during the test through subcritical crack growth, Fig. 9. Similar fracture surfaces were found for the system with 20% of whiskers in the majority of samples tested at elevated temperatures. In these samples the fracture origins were more evident when compared to the fracture origins in the system with 10% of whiskers. On the basis of mechanical testing results and fractographic analysis it seems that for the high temperature strength degradation in studied systems subcritical crack growth is responsible. Previous results[14,17] achieved on Si_3N_4 ceramics show that the subcritical crack growth began to occur at temperatures between 800 and 1000°C depending on the type of processing and additives. Results also show that the subcritical crack growth occurs at the different stress levels ($K_I/K_{IC} = 0{\cdot}5$–$0{\cdot}75$) and its rate is different (10^{-11}–10^{-6} m/s) for different stress levels, temperatures and additives.[18] In our case the differences in cluster size and their percentage portion causes strength degradation by subcritical crack propagation for the system with 20% of whiskers at about 800°C and for the system with 10% of whiskers at about 1000°C. The subcritical crack propagation in samples tested at the same temperatures is probably occurring in the system with 20% of whiskers at a lower K_I/K_{IC} ratio and the propagation rate is probably higher for this system when compared with the system with 10% of whiskers.

The high temperature fracture toughness dependence on the temperature for the system with 20% of whiskers is shown in Fig. 8. According to our results there is no significant difference between the room and high temperature fracture toughness. The scatter in the measured values are probably connected with the artificial crack geometry induced with a hardness indentor. Because the fracture toughness values were calculated on the basis of initial crack conditions, the real fracture toughness values are probably higher when subcritical crack propagation is occurring. On the other hand the softening of the intercrystalline phase and changes in the toughening mechanics at the elevated temperatures also have an influence on the fracture toughness values. The study of the high temperature creep and subcritical crack growth characteristics are the object of our present investigation.



(a)

(b)

Fig. 11. Crack deflection in whisker-reinforced ceramics: (a) example of the crack deflection in $Si_3N_4 + 10\%$ of β-Si_3N_4 whisker ceramics; (b) schematic illustration of the deflection process.

3.4 Toughening mechanisms

The following toughening mechanisms were observed on the fracture lines and fracture surfaces: crack deflection, debonding and bridging and to a lesser extent crack branching and whisker pull-out, Fig. 10. The mechanism occurring most often is crack deflection, Fig. 11, usually arising when the angle between the propagating crack direction and the whisker axis is about 20–50°. The length of the deflected portion of the main crack strongly depends on the radius and length of the whiskers. The deflected portion is very small in the case of whiskers with a small radius and small aspect ratio. Our observations show that crack deflection is often accompanied by a matrix crack. In most cases it arises at the long whiskers, Fig. 11. When the whisker at the front of the propagated crack is long enough or when the distance between the crack front and the whisker end is too large whisker/matrix debonding takes place on both sides of the whisker in the bridging and/or wake zone, Fig. 12. Similar mechanisms were found in the wake zone only in the case of long and favourably inclined whiskers. In agreement with other authors[5] it was found that the process/bridging zone is usually very small because non-aligned inclined whiskers fail behind the crack front by bending, in spite of the fact that, before this, whisker/matrix debonding occurs, Fig. 13. Unfortunately, this debond length is very short in our case: the average value is about 0·5*r*, where *r* is the whisker radius. This debonding arose before whisker failure and in the wake zone no debonding was observed in such cases. From this it is evident that effective pull-out cannot occur in similar systems. Our observations give a similar result, we found only short pull-out, in the case when the end of the whisker perpendicularly inclined to the main crack was very close to the crack front. Long pull-out was observed only occasionally, Fig. 14.

Fractographic analysis of fracture surfaces of specimens ruptured at high temperatures showed that the mode of fracture during subcritical crack growth was primarily intergranular and outside the subcritical crack growth region it was a mixture of transgranular and intergranular.

At elevated temperatures, above 1000°C, we found small differences in observed toughening mechanics as compared with room temperature mechanics. It seems that the deflected portion of the crack is higher and the pull-out seems to be a little more evident. In Fig. 15 we compare our measured values (increase in fracture toughness with whisker content, measured by indentation method[9,10]) and calculated values according to the relations given by Campbell and Becher.[5,6] To compute the steady state toughening we used 5 and 8 GPa for the whisker tensile strength value and $\gamma_i = 8\ J/m^2$ for interface energy, and we supposed that the Young modulus of Si_3N_4 whisker is similar to the Young modulus of the matrix. In Campbell's formula we neglected the contribution from residual strain energy and from pull-out. Comparing our results

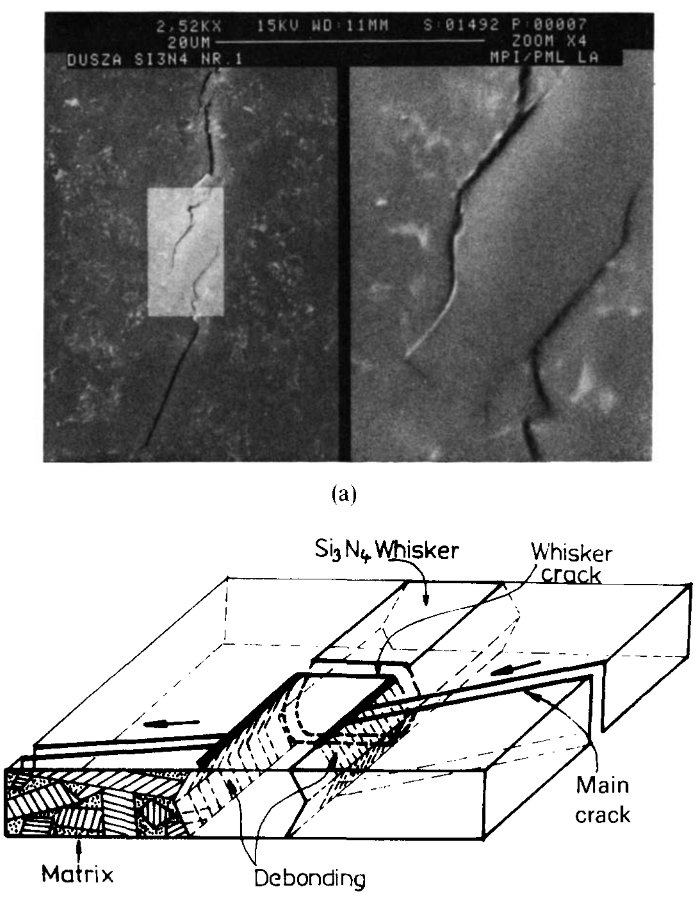


Fig. 12. Whisker/matrix debonding and bridging processes: (a) example in the system $Si_3N_4 + 20\%$ of β-Si_3N_4 whisker ceramics; (b) schematic illustration of debonding and bridging processes followed by whisker failure.

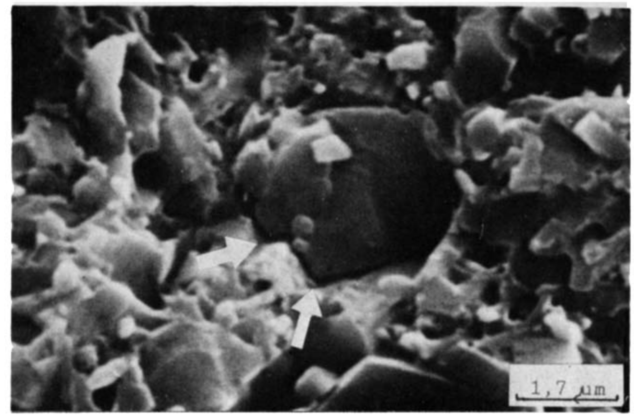
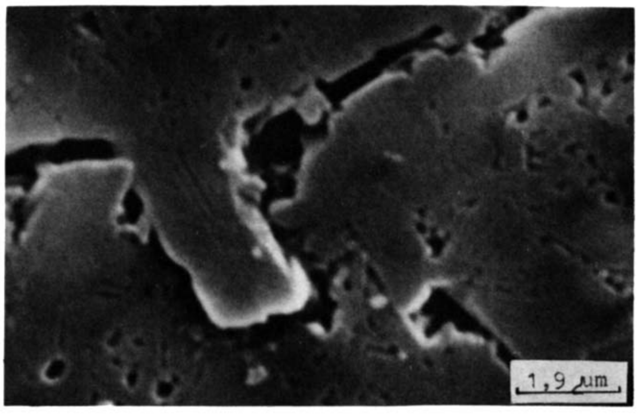
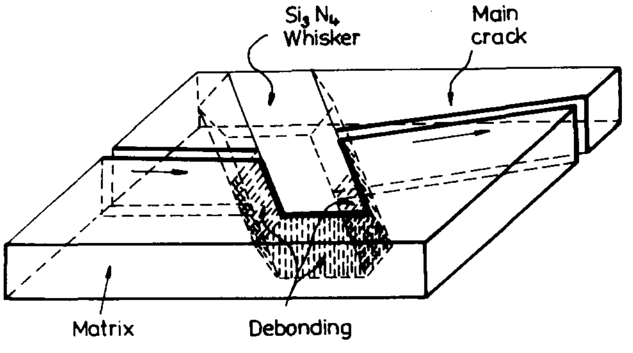


Fig. 13. Whisker/matrix debonding around the whisker perpendicular to the main crack plane.



(a)



(b)

Fig. 14. An occasionally found whisker pull-out: (a) example in the system $Si_3N_4 + 20\%$ of β-Si_3N_4 whisker ceramics; (b) schematic illustration of the pull-out.

with calculated values it can be seen that systems with 5 and 10% of whiskers are in agreement with theoretically predicted ones. Campbell's relation gives similar results at a whisker tensile strength of 5 GPa, while Becher's relation gives similar results at the whisker strength of 8 GPa. The experimental values for the system with 20% of whiskers is low compared with the predicted curves. This can be explained by clustering of whiskers, which in the form of clusters are ineffective for the toughening process. According to our experiments, beside the clustering, the toughening effect is limited mainly by the aspect ratio of the whiskers and by the whisker/matrix infterface bonding in these materials.

4 Conclusions

—Mechanical properties: four-point bend strength, Weibull modulus and fracture toughness of β-Si_3N_4 whisker-reinforced ceramics are very sensitively influenced by their microstructural characteristics.

—The room and high temperature strength and room temperature Weibull moduli are determined by the β-Si_3N_4 agglomerates, mainly in the case of the system with 20% of whiskers. Moreover, these agglomerates have a negative influence on the fracture toughness.

—The fracture toughness increase due to β-Si_3N_4 whisker addition is lower than would be expected according to the theoretically based predictions. This fact can be explained by the low aspect ratio of whiskers and by a strong whisker/matrix bonding and also by whisker clustering. The whisker pull-out is strongly limited by the failure of whiskers in bending behind the crack tip and by the Si_3N_4 matrix grains which grow into the whiskers during the hot pressing and prevent their pull-out.

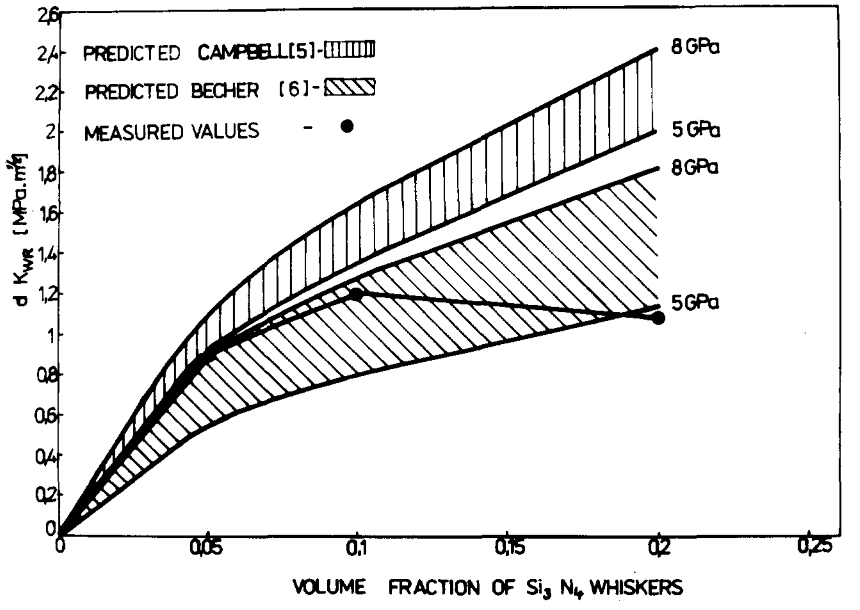


Fig. 15. Comparison of the experimentally achieved and theoretically predicted steady state toughening for the systems studied.

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