β-Sialon Ceramics with TiN Particle Inclusions

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Abstract

Fully dense composites of 0–30 wt% discrete TiN particles distributed in a β-sialon matrix of overall composition $Si_{5.5}Al_{0.5}O_{0.5}N_{7.5}$ have been prepared by hot isostatic pressing at 1650 and 1750°C. Pressureless sintering at 1775°C gave materials with an open porosity. Typical sizes of the TiN particles were 1–3 μm, and no intergranular glassy phase was observed in the prepared materials. The grain size of β-sialon was below 1 μm in the materials HIPed at 1650°C, and 1–2 μm at 1750°C. The Vickers hardness was fairly constant for the TiN–β-sialon composites with up to 15 wt% TiN added: H_V10 around 17·5 GPa for materials HIPed at 1650° and around 17 GPa at 1750°C, whereas at higher TiN contents the hardness decreased to around 16 GPa. The indentation fracture toughness of the β-sialon ceramic increased approximatively from 3 to 4 $MPam^{1/2}$ at an addition of 15 wt% TiN particulates. The fracture toughness could be further increased to 5 $MPam^{1/2}$ by addition of small amounts of Y_2O_3 and AlN to a β-sialon composite with 30 wt% TiN.

Durch heißisostatisches Pressen bei 1650 und 1750°C wurden dichte Verbundwerkstoffe bestehend aus 0–30 Masse% TiN-Teilchen verteilt in einer β-Sialon Matrix mit einer Gesamtzusammensetzung von $Si_{5.5}Al_{0.5}O_{0.5}N_{7.5}$ hergestellt. Druckloses Sintern bei 1775°C ergab Materialien mit einer offenen Porosität. Typische Größen der TiN-Teilchen lagen bei 1–3 μm. Es wurde keine intergranulare Glasphase in den hergestellten Materialien gefunden. Die Korngröße des β-Sialons war kleiner als 1 μm bei den bei 1650°C und 1–2 μm bei den bei 1750°C heißgepreßten Proben. Die Vickers-Härte war für TiN-β-Sialon-Composites mit TiN-Zugaben von bis zu 15 Masse% beinahe konstant: H_V10 lag bei 17·5 GPa für Materialien, die bei 1650°C heißgepreßt worden waren, und bei 17 GPa für die bei 1750°C heißgepreßten. Höhere TiN-Gehalte setzten die Härte auf ungefähr 16 GPa herab. Die Bruchzähigkeit der β-Sialon-Keramik, die durch Härteeindrücke gemessen wurde, stieg ungefähr von 3 auf 4 $MPam^{1/2}$ bei einer TiN-Teilchen-Zugabe von 15 Masse%. Die Bruchzähigkeit konnte weiter auf 5 $MPam^{1/2}$ erhöht werden, wenn zu β-Sialon-Composites mit 30 Masse% TiN kleine Mengen Y_2O_3 und AlN zugegeben wurden.

On a préparé par pressage isostatique à chaud à 1650 et 1750°C des matériaux composites totalement denses comprenant de 0 à 30% en masse de particules de TiN distribuées dans une matrice de β-sialon de composition totale $Si_{5.5}Al_{0.5}O_{0.5}N_{7.5}$. Un frittage à pression normale donne des matériaux présentant une porosité ouverte. Les particules de TiN avaient une taille de 1 à 3 μm et on a observé aucune phase vitreuse intergranulaire dans les échantillons. La taille des grains de sialon est inférieure à 1 μm pour les échantillons HIPés à 1650°C et de 1 à 2 μm à 1750°C. La dureté Vickers reste environ constante pour le composites TiN–β-sialons jusqu'à un ajout de 15% en masse de TiN: H_V10 vaut 17·5 GPa pour les matériaux HIPés à 1650°C et vaut 17 GPa à 1750°C, alors que pour des teneurs supérieures en TiN la dureté diminue jusqu'à 16 GPa. La résistance à la fracture du β-sialon augmente approximativement de 3 à 4 $MPam^{1/2}$ après l'ajout de 15% en masse de particules de TiN. La résistance à la fracture a pu être augmentée à 5 $MPam^{1/2}$ en ajoutant de petites quantités de Y_2O_3 et d'AlN au composite de sialon avec 30% en masse de TiN.

1 Introduction

For more than a decade, attention has been paid to the possibility of making composite materials by the addition of TiN to Si_3N_4 ceramics. By chemical vapor deposition (CVD), $TiN-Si_3N_4$ composites of several mm thickness and with up to 20 vol.% TiN were made by Hirai, and coworkers.[1-3] Very small granular TiN dispersions were found in an α-Si_3N_4 matrix prepared at 1250–1350°C, whereas CVD temperatures above 1350°C produced columnar or fibrous TiN crystallites in a β-Si_3N_4 matrix. The TiN crystals were about 1 μm long and 5 nm thick, and were homogenously dispersed. No glassy intergranular phase was present in these fully crystalline materials, and the mechanical properties were not determined. Today it is well known that reinforcement of a ceramic matrix by a second hard ceramic phase, in the form of particles, platelets or whiskers, is one way to increase the fracture toughness.[4] However, it is important that the size (and the shape) of the particle inclusions fulfill certain requirements and that the interfacial properties between the particles and the matrix are optimal. In the CVD-prepared composites already mentioned, the size of the TiN inclusions was probably much too small and the interfacial bond too strong to contribute to an increased toughness.

Most commonly, $TiN-Si_3N_4$ or TiN–sialon composites prepared from powder mixtures have been described.[5-10] These composites were always sintered with 5–10 wt% Y_2O_3 and 2–10 wt% Al_2O_3 added simultaneously as sintering aids, and the densification behavior of these (Si_3N_4 + TiN) powder mixtures was very similar to that of Si_3N_4 powder with (Y_2O_3 + Al_2O_3) added prior to sintering. Thus, small amounts of added alumina (2–3 wt%) together with yttria required pressure sintering to result in full density, whereas high alumina additions (i.e. sialon ceramics) allowed pressureless sintering. No indications were found that TiN took an active part in the densification process, and solid undissolved TiN particulates remained in the sintered microstructure, which also contained residual glassy phase. Ceramic TiN–β-sialon composites have also been prepared by adding the elements Al and Ti as metal alkoxides to silicon nitride, followed by nitriding of the formed titanium oxide during sintering.[11] Other routes for so-called in-situ TiN reinforcement have also recently been presented, where added TiO_2 powder is nitrided during the sintering process.[12,13]

Typical variations in the mechanical properties at room temperature were observed when the amount of TiN inclusions in the matrix increased:

the hardness decreased and the fracture toughness increased. The toughness increased in general with increased additions up to about 30–35 vol.% of TiN, but the increase relative to unreinforced Si_3N_4 matrix material varied between different reports from about 5–15%[6,8,10,11] to about 60–70%.[7,9] The variation of the observed toughness of the composite materials might partly be due to different TiN particulate sizes or different grain boundary phases. In a recent article by Nagaoka et al.[10] it was shown that the TiN particle size distribution is important; the fracture toughness of a composite with 10 vol.% TiN inclusions showed a maximum value for a TiN particle size of about 4 μm. A number of possible toughening mechanisms caused by the presence of TiN particulates are reviewed and discussed in Ref. 6. One of the suggested mechanisms was confirmed by Nagaoka et al.[10] who observed extensive microcracking around the TiN particle inclusions which was suggested to be caused by the substantial mismatch in thermal expansion between Si_3N_4 (3×10^{-6}/°C) and TiN (9.4×10^{-6}/°C). The authors concluded from this observation that energy absorbed by the microcracking was the major mechanism for the observed improvement in toughness in the composite materials.

Studies of the oxidation resistance of glass-phase containing Si_3N_4–TiN composites have shown that these materials are inferior to Si_3N_4 ceramics.[10,14] Titanium nitride is not a very stable phase in contact with air at high temperatures, and the TiN particulates at the composite surface is thus oxidized to titanium oxide at temperatures as low as 500–600°C. When a sialon glassy phase is present the titanium oxide might further react with this phase to form low-temperature eutectic liquids, which strongly accelerates the oxidation rate.[10,14]

The composite materials already discussed are almost always prepared by additions of fairly substantial amounts of sintering aids which form significant amounts of an Y-sialon glassy phase. It is well known that the presence of such a residual glassy grain-boundary phase is detrimental for the high-temperature mechanical properties. The preparation of pure Si_3N_4 ceramics without addition of sintering aids has been demonstrated to be possible, but it requires hot isostatic pressing (HIPing) at very high temperatures, typically around 1950°C.[15,16] However, the sintering temperatures can be lowered by several hundred degrees by preparing single-phase sialon ceramics. This has been shown by the preparation of single-phase β-sialon ceramics around or even below 1700°C.[17,18] The β-sialon materials without glassy phase also have excellent oxidation resistance at

high temperatures.[19] These latter ceramic materials contain no glassy grain-boundary phase and behave like β-Si_3N_4 ceramics for low substitution degrees, i.e. they are hard and brittle with a toughness only around 3 $MPam^{1/2}$. Thus the effects of adding TiN as a reinforcement phase in the single-phase β-sialon ceramics are of great interest, as reported later.

2 Experimental Procedure

The matrix material prepared in this study corresponds to a β-sialon, $Si_{6-z}Al_zO_zN_{8-z}$ with $z = 0.5$, made from carefully weighed powder mixtures in which account of the oxygen impurities present in the Si_3N_4 and AlN starting materials has been taken. A number of composites with this sialon parent and with 5, 10, 15 or 30 wt% TiN added were prepared. The 30 wt% TiN composite material was also sintered with additions of 2–5 wt% AlN and 0.5–2 wt% Y_2O_3. The source materials were silicon nitride (UBE, grade SN10E), aluminum oxide (Alcoa, grade A16SG) aluminum nitride (H.C. Starck, Berlin, grade A), titanium nitride (H.C. Starck, Berlin) and yttrium oxide (H.C. Starck, Berlin, 99.9%). Chemical analysis of the TiN powder showed that the carbon content was below 0.01 wt% and the oxygen content 0.33 wt%. Powder compacts were either pressurelessly sintered in a nitrogen atmosphere for 2 h at 1775°C or glass-encapsulated and hot isostatically pressed for 2 h at 1650°C or 1750°C, using an argon pressure of 200 MPa. Scanning electron microscopy (SEM) was performed on carbon coated materials, using a Jeol JSM 6400 microscope equipped with Tracor System EDS and WDS analyzers. The volumes of the different phases were measured with the Tracor image-analysis program VISTA. The phase analysis was based on X-ray powder patterns recorded with Guinier–Hägg cameras. The materials were prepared for physical characterization using standard techniques. Hardness (H_V 10) and indentation fracture toughness (K_{IC}) at room temperature were obtained with a Vickers diamond indenter applying a 98 N (10 kg) load. The fracture toughness was evaluated by the formula given by Anstis et al.,[20] assuming a value of 300 GPa for the Young's modulus.

3 Results and Discussion

3.1 Densification
Carefully polished cross-sections of the materials prepared by HIPing were examined for porosity

Table 1. The densities of TiN–β-silicon ($Si_{5.5}Al_{0.5}O_{0.5}N_{7.5}$) composite ceramics sintered in different ways[a]

Additions (wt%)			Density (g/cm³)		
TiN	AlN	Y_2O_3	PS1775[a]	HIP1650[a]	HIP1750[a]
0	0	0	2.56	3.133	3.140
5	0	0	2.98	3.195	3.189
10	0	0	2.88	3.256	3.278
15	0	0	3.00	3.315	3.343
30	0	0	3.12	3.525	3.502
30	2	0	3.19	3.521	3.490
30	5	0	3.16	3.541	3.508
30	5	0.5	3.18	3.578	3.524
30	5	1	3.22	3.567	3.496
30	5	2	3.22	3.561	3.506

[a] Pressureless sintering for 2 h at 1775°C (denoted PS1775) in nitrogen atmosphere yielded materials with an open porosity, whereas glass-encapsulated HIPing at 1650°C (HIP1650) and 1750°C (HIP1750) yielded almost fully dense materials; see text.

by optical or scanning electron microscopy, which showed that most of the specimens were virtually fully dense. Very little microporosity was seen and this was estimated to be below 0.5 vol.%. The exceptions among the HIPed materials were those containing extra additions of (Y_2O_3 + AlN) to the 30 wt% TiN composite and sintered at 1750°C, in which microporosity up to around 3 vol.% was seen. Normally, additions of these sintering aids to pure Si_3N_4 do not induce this behavior, and it is believed that yttria reacts to some extent with the TiN present in the composite, possibly resulting in some gas evolution.

For the pure TiN–β-sialon composites the density increased linearly with increasing TiN addition, from around 3.14 to 3.51 g/cm³, see Table 1. This is in fair agreement with a density around 3.15 g/cm³ previously reported for β-sialon[17] and with the expected densities calculated with the rule of volume mixture, using a density of 5.22 g/cm³ for pure TiN.

The materials prepared by pressureless sintering only yielded between 82–91% of the corresponding density achieved by HIPing, and all these materials had an open porosity. This was the case even for the pressurelessly sintered samples containing additions of up to 5 wt% AlN and 2 wt% Y_2O_3.

3.2 X-Ray diffraction phase analysis
The results of the phase analysis by X-ray diffraction (XRD) are summarized in Table 2. The major phase in all preparations was β-sialon, and the z-value of the β-sialon phase $Si_{6-z}Al_zO_zN_{8-z}$ was evaluated for each preparation from the observed lattice parameters, using the equations ($a = 0.7603 + 0.00297.z$ nm and $c = 0.2907 + 0.00255.z$ nm.[17,18] When only TiN was added to the β-sialon material, no significant differences (above ±0.1) were noted

Table 2. A summary of the X-ray diffraction phase analysis of the prepared β-sialon–TiN particulate composite materials[a]

Additions (wt%)			z-Value and observed phases		
TiN	AlN	Y_2O_3	PS1775[a]	HIP1650[a]	HIP1750[a]
0	0	0	0·5, —	0·5, —	0·5, —
5	0	0	0·5, —	0·4, (0')	0·5, (0')
10	0	0	0·5, —	0·5, (0')	0·5, (0')
15	0	0	0·5, —	0·5, (0')	0·5, (0')
30	0	0	0·5, —	0·5, (0')	0·4, (0')
30	2	0	0·7, —	0·5, (0')	0·7, (0')
30	5	0	0·6, —	0·5, —	0·9, (0')
30	5	0·5	0·6, α	0·8, —	0·9, —
30	5	1	0·6, α	0·7, —	0·9, (0')
30	5	2	0·7, α	0·8, —	0·9, (0')

[a] The materials were either pressurelessly sintered for 2 h in nitrogen at 1775°C (PS1775) or glass-encapsulated and HIPed at 1650°C (HIP1650) or 1750°C (HIP1750). In all materials β-sialon was the major phase, and cubic TiN was found in all composites where TiN particulates had been added. In the table the calculated z-values of the β-sialon phase $Si_{6-z}Al_zO_zN_{8-z}$ are given. Only observed phases besides β-sialon and TiN are listed, where (0') denotes minor amounts of O'-sialon (or Si_2N_2O) and α denotes α-sialon; see text.

from the expected z-value of 0·5. However, in the specimens where a small amount of AlN was added prior to sintering, a slight increase in the z-value was found. Thus it seems as if some of the aluminum from the AlN entered the β-sialon structure.

Minor amounts of Si_2N_2O or O'-sialon were observed in the materials prepared by HIPing. However, as only one or a few of the strongest peaks were detected, it was not possible to determine by refinement of lattice parameters if Al–O had been substituted into the oxynitride phases. In addition, materials pressurelessly sintered in nitrogen with AlN and Y_2O_3 simultaneously added contained α-sialon, which increased in amount with increasing addition of these sintering aids. Thus, the $\alpha/(\alpha + \beta)$ ratios determined from peak areas were 0·1, 0·22 and 0·34 in the materials with 0·5, 1 and 2 wt% Y_2O_3 added, respectively. The true amount of Y-containing α-phase is, however, much smaller, as the X-ray diffraction scatter of its structure is significant higher than for a pure Si–Al–O–N structure. The lattice parameters of the hexagonal α-phase were found to be $a = 0·78$ nm and $c = 0·57$ nm, proving that α-sialon rather than any residual α-Si_3N_4 was present.[21]

In all composite materials prepared by glass-encapsulated HIPing, the observed lattice parameter of the cubic TiN phase was 0·424 nm, in agreement with that expected for pure TiN. However, the cubic cell edge of the TiN phase in the pressurelessly sintered materials was longer: 0·426 nm. This may be caused by small amounts of carbon monoxide present in the nitrogen atmosphere of the graphite furnace used at high tem-

peratures, resulting in small amounts of carbon being alloyed into the TiN phase. The cubic phase Ti(C,N) is known to have a complete solid solution range to the other end member TiC, which has a lattice parameter of 0·433 nm.

3.3 Microstructure

The microstructures of the composite materials with 30 wt% TiN dispersions sintered at 1650°C and 1750°C are illustrated in Fig 1(a) and (b), respectively. Two different contrasts can be seen; the lighter areas correspond to the TiN phase and the darker contrast to the sialon phase. Image analysis showed that the addition of 30 wt% TiN resulted in an area/vol% ratio of about 15–16%, which is in agreement with TiN having almost twice the density of β-sialon. A comparison of the two microstructures shows that the diameter of the areas corresponding to TiN crystals is shorter (0·2–0·5 μm) in the material sintered at the lower temperature than at the higher temperature (0·5–
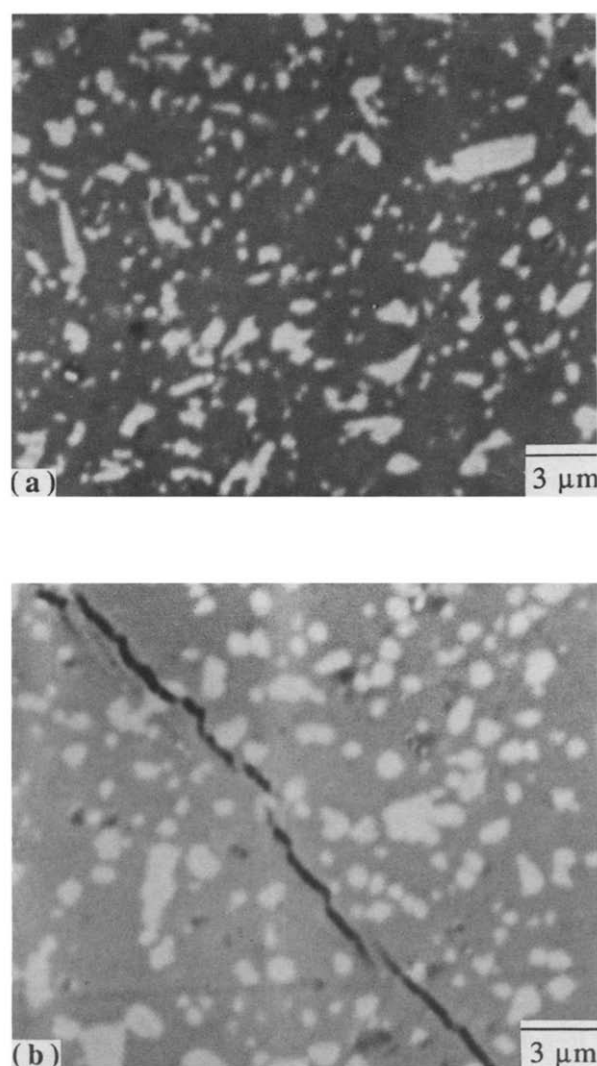

(a) 3 μm

(b) 3 μm

Fig. 1. Backscattered SEM image of the sample containing 30 wt% TiN additions sintered at (a) 1650°C and (b) 1750°C. The light areas correspond to the TiN phase. The crack path in (b) should be compared with those in Fig. 5; see text.

1 μm). Thus, the TiN particles seem to have grown larger at the higher temperature, and they also appear to have a tendency of grouping together to form agglomerates. The same trends can be seen to be even more pronounced when comparing the samples HIPed at 1650 and 1750°C which, in addition to 30 wt% TiN, also contain 5 wt% AlN and 2 wt% Y_2O_3 as sintering aids, see Fig. 2(a) and (b). One possible reason for the agglomeration of TiN is that the particles are not wetted well enough by the liquid formed during the sintering process and accordingly tend to stick together.

Despite the fact that the XRD phase analysis showed many of the HIPed samples to contain Si_2N_2O or O'-sialon in very small amounts, no contrast corresponding to these phases could be observed in the micrographs. Thus the O'-sialon crystals that form in these samples must be rather small, probably much less than a micron in size.

For the pressurelessly sintered materials, most of the added yttria was probably consumed by the α-sialon formation. In the HIPed materials with yttria added it may be assumed that yttrium formed an amorphous glassy phase, since no crystalline yttrium-containing phase was detected by the XRD phase analysis. However, no clear evidence of a glassy phase could be seen with the SEM technique applied. Yttrium-rich glassy pockets at the multi-grain junctions are normally easy to see, and the small amount of glassy phase present is probably present only as thin films between adjacent sialon grains. This is a well-known phenomenon for silicon nitride based ceramics,[22] and it will be studied in more detail by high-resolution TEM in connection with planned oxidation studies of these composite materials.

Since it is not possible to obtain the sizes of the β-sialon grains directly from the SEM-images, they were determined by analyzing fracture surfaces. The fracture surfaces of the pure β-sialon matrices prepared at the two HIPing temperatures are shown in Fig. 3(a) and (b). It can be seen that the β-sialon grains are larger (1–2 μm) in the samples sintered at the higher temperature than the crystals formed at the lower temperature (<1 μm). The major part of the fracture seems to have occurred by a transgranular mechanism. The grain
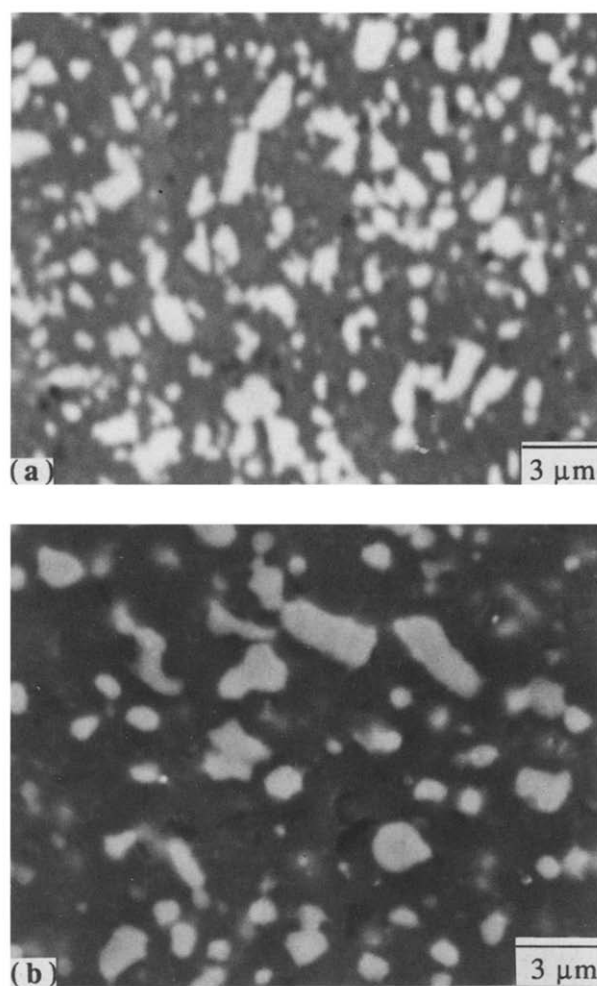

Fig. 2. Backscattered SEM image of the sample containing 30 wt% TiN, 5 wt% AlN and 2 wt% Y_2O_3 additions sintered at (a) 1650°C and (b) 1750°C. The light areas correspond to the TiN phase; note the tendency for TiN to form agglomerates at the higher temperature.
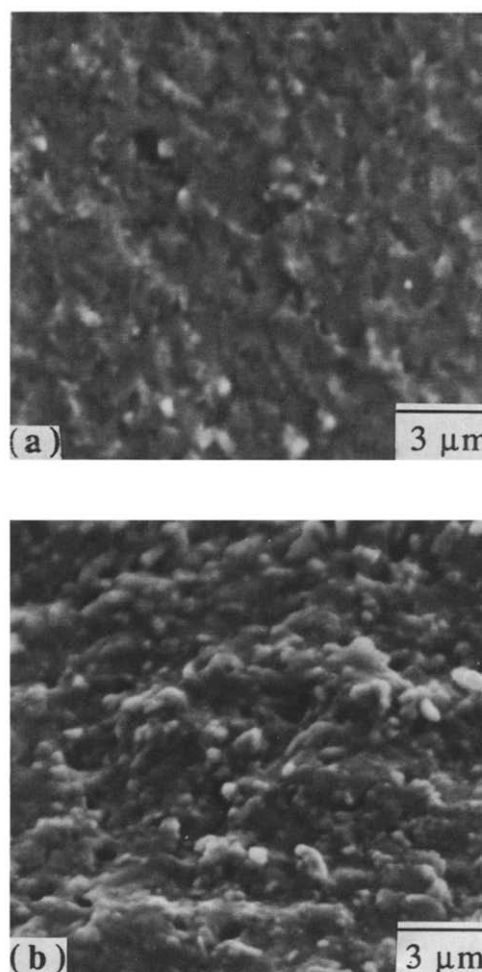

Fig. 3. Fracture surfaces of pure β-sialon, $Si_{5.5}Al_{0.5}O_{0.5}N_{7.5}$, sintered at (a) 1650°C and (b) 1750°C.

sizes found are in accordance with earlier results obtained by transmission electron microscopy on thin slices of similar materials.[17]

To exemplify the effect of sintering aids, two further examples of fracture surfaces are shown in Fig. 4(a) and (b). Besides the fact that the sample at higher temperature contains larger grains, note that both these materials have much rougher fracture surfaces than the undoped material shown in Fig 3, which indicates a higher extent of inter-granular crack path in these cases. This conclusion is confirmed by studying how indentation cracks propagate through the microstructure of a pure β-sialon and a β-sialon with extra sintering aids and TiN particulates added, see Fig. 5(a) and (b). A much straighter transgranular crack path is seen in microstructure of the pure β-sialon material. This can also be compared with the cracks in the TiN–β-sialon composite ceramic shown in Fig. 1(b), where the crack also showed deflection and a tendency of crack branching close to TiN inclusions. These effects of the TiN inclusions on a propagating crack as well as the corresponding increase in fracture energy when the crack follows
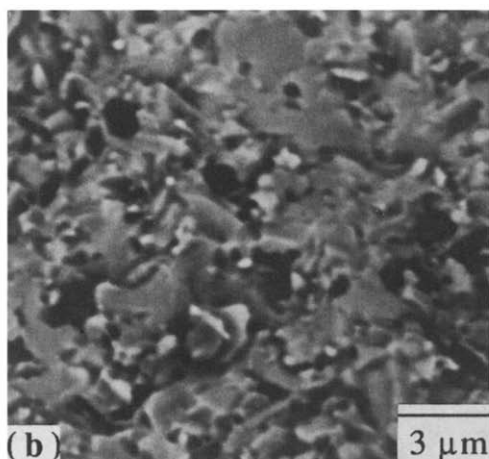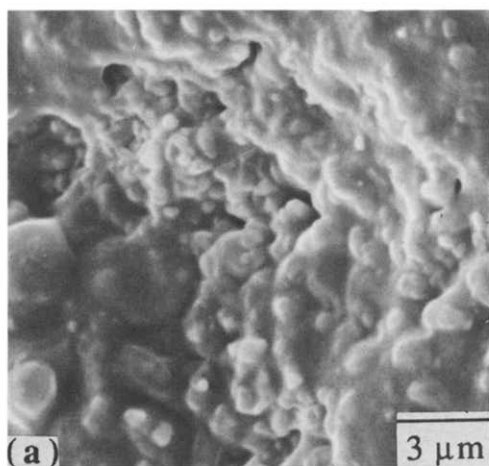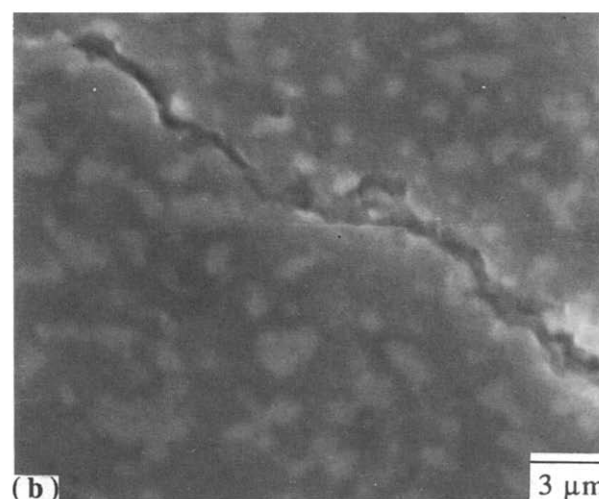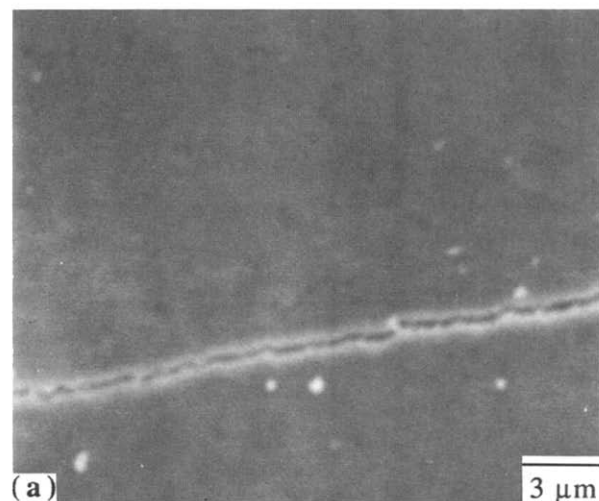

Fig. 5. The indentation crack path (a) in a pure β-sialon matrix material and (b) in the β-sialon material containing 30 wt% TiN, 5 wt% AlN and 2 wt% Y_2O_3 sintered at 1750°C.

a glassy grain-boundary phase (when present) will give rise to an increase in the observed fracture toughness of the sialon materials, see later.

3.4 Mechanical properties

The Vickers hardness, H_V10, and indentation fracture toughness of the HIPed fully dense TiN–β-sialon composites are summarized in Figs 6(a) and (b), respectively. It can be seen that the materials HIPed at the lower temperature are generally somewhat harder. This is probably an effect of the smaller β-sialon grain size noted in the low-temperature materials, cf. the previous section. The hardness at both 1650 and 1750°C is fairly constant up to a content of 15 wt% TiN particles in the β-sialon matrix. However, an addition of 30 wt% TiN material causes a clear decrease in hardness (about 8%). The observed indentation fracture toughness increases with the TiN addition from around 3 to 4 $MPam^{1/2}$. The increase in toughness is similar to that from 3·3 to 3·7 $MPaM^{1/2}$ obtained for a TiN–β-sialon composite without sintering aids, reported earlier by Kishi & Umbayashi.[11]

Fig. 4. Fracture surfaces of the samples containing 30 wt% TiN, 5 wt% AlN and 2 wt% Y_2O_3 sintered at (a) 1650°C and (b) 1750°C.
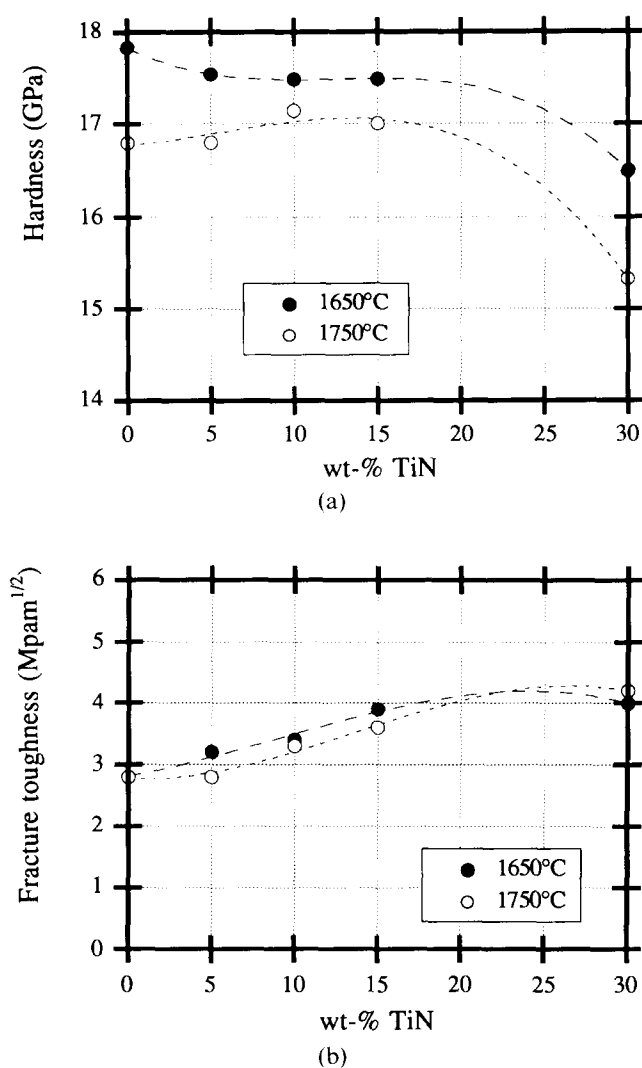
Fig. 6. (a) The Vickers hardness at a 98 N load ($H_V 10$) and (b) the indentation fracture toughness measured at room temperature of the β-sialon matrix with overall composition $Si_{5.5}Al_{0.5}O_{0.5}N_{7.5}$ and with 0–30 wt% TiN particles added.

Fig. 7. The variation in (a) hardness and (b) indentation fracture toughness with AlN additions. The samples contain 30 wt% TiN.

The addition of 2 or 5 wt% AlN alone to 30 wt% TiN composites does not significantly change either the hardness or the fracture toughness, see Fig. 7(a) and (b), respectively. However, a simultaneous addition of 0·5–2 wt% Y_2O_3 and AlN did significantly influence the mechanical properties, as demonstrated in Fig. 8(a) and (b). The fracture toughness reached a value above 5 MPam$^{1/2}$ for the HIPed composites containing 2 wt% Y_2O_3. The same trend for the fracture toughness was observed for the samples sintered at both temperatures, but there is a significant difference in hardness between the two series. The hardness of the series sintered at the higher temperature decreased from 16 to 14 GPa compared to a decrease from 16 to 15 GPa at the lower temperature. The larger decrease for the former materials might be a combined effect of coarser grains, films of glassy phase and possibly the tendency for TiN agglomeration in the high-temperature materials.
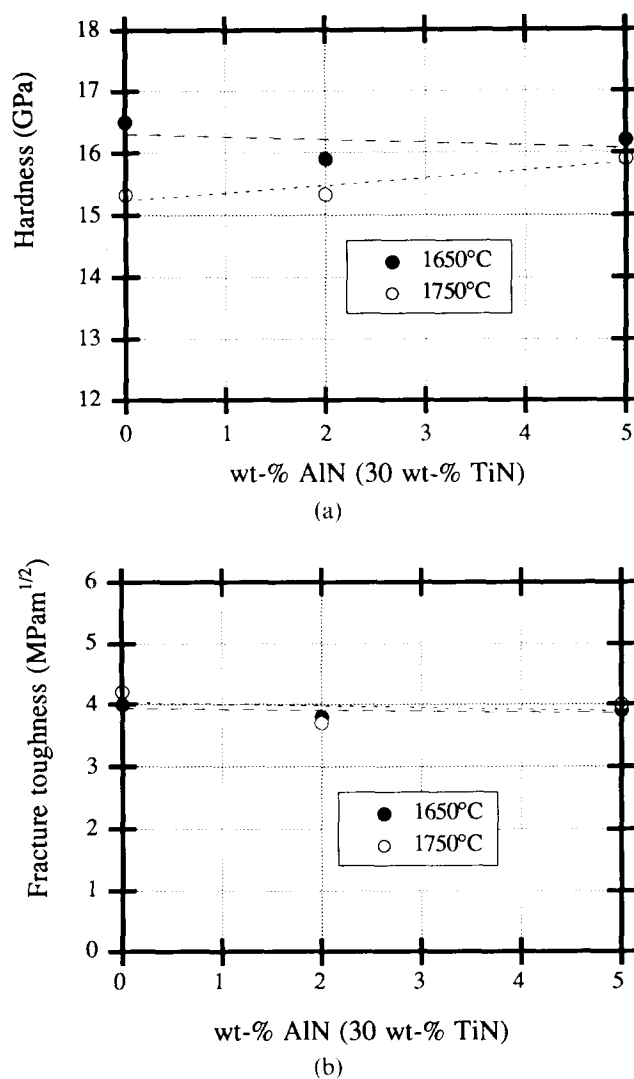
It seems that a sintering temperature of 1750°C

is too high for a good combination of high hardness and fracture toughness. Furthermore, the samples containing Y_2O_3 and sintered at 1750°C contained agglomerates of TiN, which might be detrimental for the strength, as these large agglomerates might act as defect regions when stress is applied.

The observed decrease in hardness and the increase in toughness with increased TiN additions compares fairly well with previous reports of the effects reported when 2–3 wt% yttria plus 5–8 wt% alumina were added to other TiN–Si$_3$N$_4$ ceramics.[6,9] However, in an earlier report in which very high additions of sintering aids (10 + 10 wt%) were used, the indentation fracture toughness increased only marginally up to about 20 wt% TiN and dropped again at higher TiN additions, whereas the hardness decreased continuously from 14·5 to 13 GPa at 40 wt% TiN.[8]

One way to understand the observed difference in fracture toughness between different samples is to compare the crack propagation in the micro-
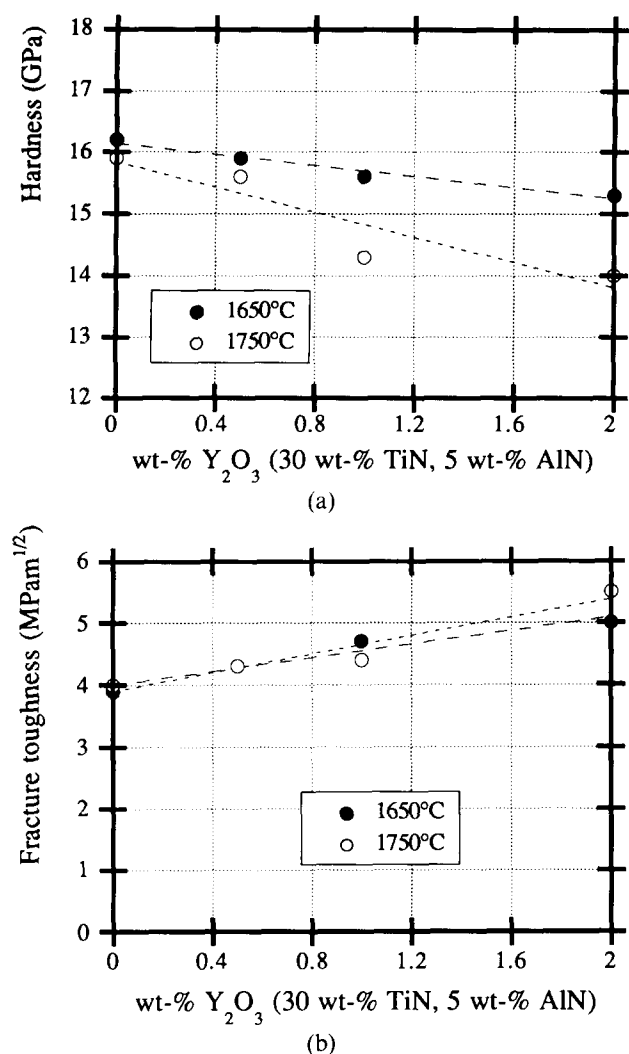
Fig. 8. The variation in (a) hardness and (b) indentation fracture toughness with Y_2O_3 additions. The samples contain 30 wt% TiN and 5 wt% AlN.

structures. It was shown above that addition of TiN particles causes the crack to deflect or branch at some of the particles. When TiN particles are dispersed in the β-sialon matrix, tensile or shear stresses will occur at the grain boundaries after sintering, because the thermal expansion of TiN is very different from that of β-sialon. These stresses will make the mentioned toughening mechanisms favoured, since microcracking at the TiN–matrix grain boundaries might easily occur as a result of additional shear or tensile stress loads from the propagating crack front.

4 Conclusions

It can be concluded that it is possible to prepare fully dense β-sialon–TiN composites at a temperature of 1650°C, which is several hundred degrees below the preparation temperatures reported for pure β-Si$_3$N$_4$ ceramics.

The TiN particulate inclusions had a positive

effect on the fracture toughness measured at room temperature. The indentation fracture toughness of the β-sialon ceramic increased from around 3 to 4 MPam$^{1/2}$ with an addition of 15 wt% TiN particulates. The fracture toughness could be further increased to 5 MPam$^{1/2}$ by addition of small amounts of Y_2O_3 and AlN to a β-sialon composite with 30 wt% TiN.

The Vickers hardness, H_V10, was fairly constant around 17·5 and 17 GPa for the TiN–β-sialon composites up to 15 wt% TiN added and HIPed at 1650 and 1750°C, respectively, whereas at higher TiN contents the hardness decreased to around 16 GPa.

In single-phase β-sialon ceramics the cracks propagate transgranularly in the material, but in the TiN–β-sialon composite the crack propagates by deflection and branching.

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