

Effect of grain growth in bimodal microstructure on cutting performance of self-toughening Dy- α -SiAlON ceramic tool inserts

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Abstract

Although significant increase in mechanical properties has been achieved in α -SiAlON ceramics by introducing self-toughening bimodal microstructure, the bimodal microstructure may not always result in better cutting performance of α -SiAlON tool inserts. Herein Dy- α -SiAlON ceramics were prepared by hot-pressing at 1900 °C under 30 MPa. Diameter and aspect ratio of the large elongated α -SiAlON grains pivotal to formation of bimodal microstructures were regulated by using 0–5 wt.% Dy₂O₃ as a sintering additive. Mechanical properties and cutting performance of α -SiAlON tool inserts were evaluated. The results showed good Vickers hardness (19.0–19.7 GPa) and fracture toughness (3.61–4.18 MPa·m^{1/2}) increasing with crack propagation length. However, transverse section of the large elongated grains was revealed as critical crack to cause premature fracture of the α -SiAlON ceramics. Fracture rather than attrition wear was recognized as dominant mechanism for tool failure, indicating that for better cutting performance of the α -SiAlON tool inserts a selftoughening microstructure consisting of thinner grains with large aspect ratio is required.

Keywords: α-SiAlON, cutting tool insert, bimodal microstructure, mechanical properties, cutting performance

I. Introduction

In the early 1970s, a wide variety of solid-solutions, i.e. α - and β -SiAlON, based on α - and β -Si₃N₄ [1– 5] were discovered. During the formation of the solidsolutions, Al, O and some metal ions are incorporated into Si₃N₄ lattice in two ways: i) substitution of Al-O for Si-N, and ii) substitution of Al-N for Si-N in combination with some metal ions, such as Li, Mg, Ca, Sc and Y, occupying interstices to compensate valency inequality caused by Al³⁺ substitution for Si⁴⁺. Capacity of incorporating various species into Si₃N₄ lattice means reduction or even complete elimination of the undesired intergranular phases that are always present in Si_3N_4 ceramics [4,5]. This is because in Si_3N_4 ceramic industry practice, the unavoidable additions of oxide sintering aids, such as Al₂O₃, Y₂O₃ and MgO, facilitating densification of the strong covalent Si_3N_4 result in presence of intergranular phases, hence limit performance of Si₃N₄ ceramics, especially at hightemperatures. The SiAlON formation shows an obvious advantage over Si3N4 because of transient liquid phase sintering behaviour of both α - and β -SiAlON ceramics, i.e. the oxide sintering additives react with silica impurity present on the Si₃N₄ particles to form a eutectic liquid at low temperatures that facilitates densification, thereafter the liquid will be absorbed for formation of the SiAlON solid solution. Sharply reduced content of intergranular phases indicates better high-temperature performance of SiAlON ceramics in comparison with Si₃N₄ [1,6–8], in addition to higher Vickers hardness of α -SiAlON (19–21 GPa) relative to β -Si₃N₄ and β -SiAlON (15–16 GPa), despite inferior strength and fracture toughness of α -SiAlON relative to β -Si₃N₄ and β -SiAlON.

Knowledge about SiAlON formation, phase equilibrium, microstructures and mechanical properties of the SiAlON ceramics has been completed after 50 years of extensive researches [9–22]. Successful introduction of self-toughening bimodal microstructures to α -SiAlON ceramics has increased strength and toughness of α -SiAlON ceramics to approach those of β -Si₃N₄ and β -SiAlON, making α -SiAlON promising for applications as cutting tools, particularly suitable for roughing nickel alloys due to high reaction resistance between Si₃N₄ and nickel [5,7,23–32].

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Material	Composition	Starting composition [g]				
	Composition	α -Si ₃ N ₄	Al_2O_3	AlN	Dy_2O_3	
Dy-0	Dy1512+0 wt.% Dy ₂ O ₃	68.107	0.098	17.218	14.577	
Dy-0.5	Dy1512+0.5 wt.% Dy ₂ O ₃	68.107	0.098	17.218	15.077	
Dy-1	Dy1512+1 wt.% Dy ₂ O ₃	68.107	0.098	17.218	15.577	
Dy-2.5	$Dy1512+2.5$ wt.% Dy_2O_3	68.107	0.098	17.218	17.077	
Dy-5	$Dy1512+5$ wt.% Dy_2O_3	68.107	0.098	17.218	19.577	

Table 1. Nominal composition of the Dy-*a*-SiAlON ceramics for investigation

In the α -SiAlON ceramics, the self-toughening bimodal microstructure shows a decent flexibility in view of controlled growth of the large elongated grains in the bimodal microstructures [10,11,33,34]. Since mechanical properties of the α -SiAlON ceramics depend on the presence of these large elongated grains, the bimodal microstructure must show certain effect on cutting performance of α -SiAlON cutting tools, but it has not been well understood yet. Herein Dy³⁺ doped α -SiAlON ceramics were prepared by hot pressing. Growth of a small concentration of large elongated α -SiAlON grains was regulated by excess addition of 0–5 wt.% Dy₂O₃ to the Dy- α -SiAlON based compositions. Mechanical properties and cutting performance of Dy- α -SiAlON cutting tool inserts were investigated.

II. Experimental details

In the general formula $Mm/vSi_{12-(m+n)}Al_{m+n}O_nN_{16-n}$ for α -SiAlON, where $M^{\nu+}$ is an ion for covalent compensation and m, n demonstrate the Al–N and Al–O substitution in the α -Si₃N₄ lattice, a nominal composition with $M^{\nu+} = Dy^{3+}$ and m = 1.5, n = 1.2 (denoted as Dy1512) was selected for investigation [10,11]. Excess amount of Dy_2O_3 in range of 0–5 wt.% was added to control growth of a few percent of large elongated grains to obtain self-toughening microstructures. The compositions under investigation are listed in Table 1. Each composition was denoted by a character Dy and a number of 0, 0.5, 0. 2.5 and 5 to refer to the content of Dy_2O_3 . For example, the notation Dy-2.5 refers to the Dy1512 SiAION ceramics with excess addition of 2.5 wt.% Dy₂O₃.

Raw materials were powders of Si₃N₄ (99.7% purity, metal base; 0.5–2 µm; Kete New Material Tech. Co. Ltd, Qinghe, China), α -Al₂O₃ (99.99% purity, 0.2 µm; Aladdin Chemical Reagent Co, Shanghai, China), AlN (TFZ-S05P grade, Toyo Aluminium K.K., Hino Works, Japan) and Dy₂O₃ (99% purity, 1 µm, Zhanhai New Material Tech. Co. Ltd, Ganzhou, China). The powder mixtures in proper proportions were homogenized in plastic bottles (500 ml capacity) for 24 h on a horizontal rolling mixer at 300 rpm, using 200 g of ZrO₂ beads and 150 ml absolute ethanol as mixing medium. The slurries were dried in a rotary evaporator (R220, Senco Tech. Co. Ltd. Shanghai, China) at 65 °C and were ready for hot pressing. Wear of the ZrO₂ beads would result in $0.0367 \,\mathrm{g}$ of ZrO₂ impurity to the powder mixture after mixing for 24 h. However, effects of the ZrO₂ impurity on microstructures and performances of the Dy- α -SiAlON ceramics were considered insignificant and thus they were not discussed in this work.

A batch of the powder mix was loaded in a graphite die with inner diameter of 30 mm for hot pressing (15t Hot Press Furnace, Materials Research Furnaces, Inc, USA). The hot-pressing process was carried out at 1900 °C under 30 MPa for 60 min in 1 atm flowing N₂ at a flux rate of 1.5 l/min. Then the temperature was decreased to 1400 °C and dwelled for 60 min to crystallize the intergranular phases of the Dy- α -SiAlON ceramics. After the 1400 °C dwell, heating power was shut down to allow cooling of the sample in the hot press.

The samples were ground, cut and polished. Bulk density was measured by the Archimedes' principle. Five samples were measured for each composition and relative density was calculated. Sample surfaces perpendicular to the hot-pressing direction were scanned by X-ray (XRD-6000, Shimadzu, Japan) for phase analysis. Before X-ray diffraction, at least 1 mm of material was by ground off from the sample surfaces to eliminate effects of possible contamination. Cu K α (wavelength λ = 0.15406 nm) radiation at 40 kV and 30 mA was used for XRD. Diffraction intensities in 2 θ range of 10–80° were collected with a scanning step 0.02° and for 0.4 s at each step.

Chemistry *m* of the α -SiAlON phase was estimated by formula $m = 3(x_a + x + c)/2$ [36,37], where x_a and x_c values were calculated by Eqs. 1 and 2, in which the lattice parameters *a* and *c* of the hexagonal α -SiAlON were determined by XRD.

$$a(nm) = 0.775 + 0.0156x_a \tag{1}$$

$$c(nm) = 0.562 + 0.0162x_c \tag{2}$$

Microstructures were observed on polished sections of the samples by a scanning electron microscope (SEM, TM4000, Hitachi High-Tech Corporation, Tokyo, Japan). Powder samples for SEM observation were prepared by pressing a small amount of the powder on conductive adhesive tape and blowing off the loose particles with compressed air. The SEM was performed at an acceleration voltage of 10 kV with working distance of ~8.5 mm.

Young's modulus E was measured by the pulse excitation method (IET-100, Jingyan Machinery Tech. Co. Ltd, Luoyang, China) according to national/international standards [38–40]. Vickers hardness H_{y} was measured on an indenter (HV5-50, Yanrun

Optical Machinery Tech. Co. Ltd, Shanghai, China) under different loads P of 1, 3, 5, 10 and 20 kg for 15 s. Fracture toughness K_{IC} was estimated from the propagation length of the cracks at the Vickers indent corners [41,42]. For testing *R*-curve behaviour of the Dy- α -SiAlON ceramics, crack propagation length was controlled by varying the Vickers hardness indentation load. The measured values of K_{IC} were plotted against the indentation load (i.e. the crack propagation length). Flexural strength was measured by three-point bending (UTM-4304, Sunstest Tech. Co. Ltd, Shenzhen, China) at room temperature at a loading rate of 0.5 mm/min.

Pieces of $12.7 \times 12.7 \times 5.94 \,\text{mm}$ were made from the Dy- α -SiAlON ceramics. Corners of the pieces were rounded with a radius r = 1.2 mm but edges were not chamfered. In combination with a steel piece $12.7 \times$ $12.7 \times 2 \,\mathrm{mm}$, the α -SiAlON pieces simulated a standard tool insert SNGN 120712. Turning performance of the Dy- α -SiAlON ceramics was tested on an ETC 3650h machining centre (Shenyang Machine Tool Co. Ltd, Shenyang, China) using Inconel 718 as work material. The cutting tool inserts were clamped in a tool bar (CSDNN2525M-1207, KUNP Cutting Tools Co. Lmt. Jiujiang, China) with front angle $\gamma_0 = -6^\circ$, rear angle $\alpha_0 = 6^\circ$ and main deflection angle $K_r = 45^\circ$. The cutting performance parameters included: linear speed $v_c = 300 \text{ m/min}$, cutting depth $a_p = 0.5 \text{ mm}$ and feed rate f = 0.1 mm/rev. The tool insert flanks, edges and noses were observed on a stereo microscope (OLYMPUS-SZ61, Lance Electronic Co. Ltd, Shanghai, China) at intervals of cutting time to measure wear resistance of the tools.

III. Results and discussion

3.1. Si_3N_4 starting powder

The main raw material Si_3N_4 was prepared by the self-propagating combustion nitridation route of Si powder. High temperature in combustion nitridation process facilitated anisotropic growth of some β -Si₃N₄ grains. XRD of the Si₃N₄ powder is shown in Fig. 1. The Si₃N₄ powder consisted of both α - and β -Si₃N₄. Content







Figure 2. SEM of the Si_3N_4 powder, showing needle-like β -Si3N4 grains (red arrows) and equiaxed α -Si₃N₄ of both small and large sizes (yellow arrows)

of the β phase was estimated to be about 32 wt.%.

SEM of the Si₃N₄ powder is shown in Fig. 2. Both elongated and equiaxed particles were revealed. The long needle-like particles as marked by red arrows in Fig. 2 were more likely β -Si₃N₄ grains whereas α -Si₃N₄ grains were equiaxed. Some β -Si₃N₄ grains with equiaxed morphology were also possible. The equiaxed particles in Fig. 2 consisted of both fine (average size $0.5 \pm 0.3 \,\mu$ m) and coarse (average size $1.7 \pm 0.5 \,\mu$ m) particles, forming a non-uniform distribution of the particle size.

The phase constituents and morphology of the Si₃N₄ powder shown in Fig. 2 was consistent with the Si selfpropagating combustion nitridation route for synthesis of the Si₃N₄ powder. Temperature in Si combustion nitridation process could reach 1600 °C, so that a large amount of β -Si₃N₄ grains could grow in combination with relatively low concentration of lattice defects of both α - and β -Si₃N₄ in the powder, indicating lower reaction driving force for SiAION formation in comparison with the virtual UBE-10 Si₃N₄ powder. However, a lower reaction driving force may be beneficial for the formation of self-toughening microstructures in α -SiAION ceramics [9,10].

3.2. Densification

The main raw material Si_3N_4 was prepared by the route of self-propagating combustion nitridation of Si powder. High temperature in combustion nitridation process facilitated anisotropic growth of some β -Si₃N₄ grains and the Si₃N₄ powder consisted of both α - and β -Si₃N₄ (content of the β phase was estimated to be about 32 wt.%).

In hot pressing of the powder mixes, ram travel as a function of temperature was recorded and demonstrated 3 successive stages for the Dy- α -SiAlON densification, in consistency with reaction densification of α -SiAlON as detailed in the literature [43]. Densification started at about 1240 °C and full densities were already reached in process of rising the temperature to 1900 °C. Hold at

Material	Theoretical density	Bulk density	Relative	Total porosity	Open porosity
	[g/cm ³]	$[g/cm^3]$	density [%]	[%]	[%]
Dy-0	3.521	3.494 ± 0.027	99.22 ± 0.76	0.78	0
Dy-0.5	3.522	3.496 ± 0.028	99.26 ± 0.80	0.74	0
Dy-1	3.522	3.486 ± 0.026	98.97 ± 0.74	1.03	0
Dy-2.5	3.523	3.497 ± 0.019	99.27 ± 0.54	0.73	0
Dy-5	3.525	3.480 ± 0.028	98.71 ± 0.79	1.28	0

Table 2. Density, relative density and porosity of the Dy- α -SiAlON ceramics

1900 °C had no more contribution to densification of the Dy- α -SiAlON ceramics.

Density measurements results of the Dy- α -SiAlON ceramics are listed in Table 2. Relative densities >98 %TD were reached in all compositions, indicating high efficiency of hot pressing for preparation of α -SiAlON ceramics. Although a total porosity was observed in each composition, this could be a result of measurement error, because the calculated theoretical densities should deviate from the real values due to XRD accuracy limit.

3.3. Phase constituents

XRD patterns of the Dy- α -SiAlON ceramics are shown in Fig. 3. All the compositions were dominated by α -SiAlON (PDF 84-0786, Y_{0.5}Si_{9.3}Al_{2.7}O_{0.9}N_{15.1})



Figure 3. XRD of the Dy- α -SiAlON ceramics, showing dominant α -SiAlON with a very limited amount of β -SiAlON and M' phase

despite different concentrations of the excess Dy_2O_3 additions. A small amounts of β -SiAlON (PDF 76-0599, $Si_4Al_2N_2O_6$) and M' phase (PDF 50-1739, $Dy_2Si_3O_3N_4$) were also detected.

An XRD refinement result for the sample Dy-5 is shown in Fig. 4. In addition to the well indexed α -SiAlON peaks, the very weak diffraction intensities at $2\theta = 31.82^{\circ}$ and 33.26° were attributed to M' and β phases, respectively. Coexistence of α , β and M' phases was consistent with phase relation in the Si-Al-O-N systems [36,44]. However, the added hold at medium temperature of 1400 °C for 60 min during the processing of the Dy- α -SiAlON ceramics may have added some β and M' content by slight decomposition of the α phase. This additional hold at a medium temperature was rational because the α -SiAlON area in the Si-Al-O-N phase diagram shrinks as temperature decreases from high temperatures of >1800 °C to medium temperatures of 1300-1600 °C [36,44-46]. SiAlON ceramics once consisted of "pure" α -SiAlON at high temperatures would regulate its composition at medium temperature to reach a new equilibrium by forming some β and M'. The temporary hold for phase stabilization at 1400 °C was expected to help improve cutting performance of the Dy- α -SiAlON ceramics because attrition friction when cutting refractory alloys such as Inconel 718 could generate a temperature rise up to 1000-1200 °C [7,26,28] at the nose and flank regions of the cutting tools.



Figure 4. XRD refinement for Dy-5: the weak intensities at $2\theta = 31.82^{\circ}$ and 33.26° were attributed to M' and β -SiAlON phases, respectively

Contents of the constituent phases α , β and M' in each composition were calculated and the results are listed in Table 3, in combination with lattice parameters and *m* values of the α phase. Despite the different

Material	Phase constituent [vol.%]			α -SiAlON lattice parameter and			
				chemistry			
	α -SiAlON	β -SiAlON	$Dy_2Si_3O_3N_4$	a [Å]	<i>c</i> [Å]	<i>m</i> value	
Dy-0	96.68	1.73	1.58	7.8282	5.7063	1.55	
Dy-0.5	96.87	1.48	1.65	7.8215	5.7014	1.44	
Dy-1	96.93	1.49	1.58	7.8166	5.6965	1.35	
Dy-2.5	96.87	1.88	1.26	7.8290	5.7054	1.55	
Dy-5	97.46	1.48	1.06	7.8264	5.7037	1.51	

Table 3. Phase constitution of the Dy- α -SiAlON, lattice parameter and chemistry *m* of the α phase



Figure 5. SEM back-scattered electron images of (A) Dy-0, (B) Dy-0.5, (C) Dy-1, (D) Dy-2.5and (E) Dy-5 polished sections. The Dy-0 composition without excess addition of Dy₂O₃ shows a somewhat more homogeneous grain size distribution whereas the other compositions with excessive Dy₂O₃ additions show bimodal microstructures featured by uniform dispersion of a few percent of large elongated grains on a fine-grained matrix

concentrations of Dy_2O_3 addition, contents of the dominant α phase was >96 vol.% while contents of the minor phases of β and M' did not exceed 2 vol.%. The α -SiAION lattice had *a* and *c* that well exceeded 7.81 and 5.69 Å, significantly enlarged relative to α -Si₃N₄ (PDF 76-1412, *a* = 7.791 Å, *c* = 5.649 Å), indicating formation of a solid solution. The *m* chemistries calculated by Eqs. 1 and 2 were in the range of 1.35–1.55, consistent with the *m* = 1.5 selection for investigation within reasonable measurement error.

3.4. Microstructure

SEM back-scattered electron images of polished sections of the Dy- α -SiAlON ceramics are shown in Fig. 5. The phases with bright, light grey and dark contrast were M', α and β , respectively. M' and β showed a homogeneous dispersion at multi-grain junctions.

In Figs. 5d and 5e, the Dy-2.5 and Dy-5 materials showed large elongated α -SiAlON grains having a coreshell structure. Presence of a core with dark contrast indicated epitaxial growth of α -SiAlON on heterogeneous nuclei, a phenomenon that has been well understood [47,48]. The nuclei for α -SiAlON grain growth were usually α -Si₃N₄ due to similarity in lattice symmetry and chemical composition between these two phases. In industrial practices, α -Si₃N₄ powders of high reaction activity were usually used as raw material for preparation of α -, β -SiAlON and β -Si₃N₄ ceramics [49]. In this case, sparse distribution of some heterogeneous nuclei in process of dissolution-precipitation at hightemperatures was benefitial for the growth of a few percent of large elongated grains on a fine-grained matrix. The resultant microstructures showed a bimodal distribution in grain size, which was used to improve mechanical properties of α -, β -SiAlON and β -Si₃N₄ ceramics.

In Fig. 5, the Dy-0.5, 1, 2.5 and 5 compositions with excess additions of Dy_2O_3 indeed formed bimodal microstructures, whereas bimodal microstructure (i.e. enlarged growth of α -SiAlON grains) in the Dy-0 composition (without excess addition of Dy_2O_3) was insignificant. More precisely, the large grains in the Dy-0 composition measured an average diameter of $2.4 \pm 0.8 \,\mu\text{m}$ with an aspect ratio 4.1 ± 1.7 . With addition of excess Dy_2O_3 , the average diameter ($4.7 \pm 2.6 \,\mu\text{m}$) and aspect ratio (8.6 ± 2.2) of the large grains were doubled in the sample Dy-0.5 relative to Dy-0. Further increase of the Dy_2O_3 concentration induced a slight increase in the av-

Material	E [GPa]	σ [MPa]	$^{*}H_{\nu}$ [GPa]	* K_{IC} [MPa·m ^{1/2}]	Critical crack length calculation by Griffith's theory [µm]
Dy-0	337 ± 15	518 ± 36	19.7 ± 0.2	3.94 ± 0.18	18.4
Dy-0.5	341 ± 18	622 ± 41	19.3 ± 0.6	4.18 ± 0.19	14.3
Dy-1	336 ± 13	616 ± 39	19.1 ± 0.4	3.50 ± 0.32	10.2
Dy-2.5	329 ± 10	609 ± 52	19.0 ± 0.3	3.61 ± 0.07	11.1
Dy-5	327 ± 15	594 ± 47	18.7 ± 0.1	3.69 ± 0.11	12.3

Table 4. Young's modulus, bending strength, Vickers hardness and fracture toughness of the Dy- α -SiAlON ceramics

*Tested at an indentation load of 196 N

erage diameter of these large grains, but the aspect ratio decreased significantly. The average diameters of the large grains in the samples Dy-1, Dy-2.5 and Dy-5 were 5.4 ± 2.8 , 6.3 ± 3.1 and $7.6 \pm 3.3 \mu$ m, respectively, and the aspect ratios were 4.9 ± 3.7 , 3.9 ± 1.7 and 3.5 ± 1.2 .

The bimodal microstructures in Fig. 5 illustrated a complex dependence of the microstructure on the nucleation and growth of the specific large elongated α -SiAlON grains. Size of the individual grains resulted from competitive growth of the grains. Heterogeneous nucleation in combination with addition of excess Dy₂O₃ seems as a feasible mean to control the grain growth rate.

Figure 5 illustrated some large α -SiAlON grains having a heterogeneous nucleus inside. These nuclei were most likely α -Si₃N₄. In Fig. 2, there were large equiaxed particles marked by yellow arrows in the Si₃N₄ powder. At high temperatures fine particles dissolved rapidly to saturate the transient liquid whereas the large ones survived the dissolving and served as nuclei for elongated growth of the large α -SiAlON grains [47,48,50]. β -Si₃N₄ crystals could also nucleate α -SiAlON grain growth but only occasionally due to the large dissimilarity between β -Si₃N₄ and α -SiAlON.

Figure 5 also illustrated some small needle-like grains having a dark image contrast and uniform distribution in the microstructures. These small grains could be either β -SiAlON or β -Si₃N₄, the only difference between them was Al–O content in their compositions [46,51]. The high content of β -Si₃N₄ particles (about 32 wt.%) in the Si₃N₄ starting powder caused presence of needle-like particles, Fig. 2. Inertness of β -Si₃N₄ relative to α -Si₃N₄ may help them survive dissolution-precipitation at high-temperatures. As a weak support, α -SiAION ceramics made from fine H. C. Starck or UBE SN-E10 silicon nitride precursor powders (~5 wt.% β -Si₃N₄) [10,11,15,20,21] seldom showed presence of β -SiAION or β -Si₃N₄ in α -SiAION ceramics particularly for SiAION compositions at centre of the α -SiAION area in the Si-AI-O-N phase diagram, such as the m = 1.5, n = 1.2 composition [36,44,45,46]. On the other hand, if the nominal composition was located in the $\alpha + \beta$ dual-phase region, the hold at 1400 °C for 60 min would introduce some β -SiAION at this lower temperature.

3.5. Mechanical properties

Young's modulus

Young's modulus of the Dy- α -SiAlON ceramics is listed in Table 4. The Young's modulus was in the range of 327–341 GPa, consistent with the values reported for α -SiAlON ceramics. In Table 4, the Young's modulus of the Dy-0 composition was 337 ± 15 GPa. When excess Dy₂O₃ was added, the Young's modulus of Dy-0.5 was increased slightly to 341 ± 18 GPa. As the Dy₂O₃ addition was increased to 1, 2.5 and 5 wt.%, however, the Young's modulus of the respective materials showed an insignificant decrease. More particulary, the Young's moduli of the Dy-1, 2.5 and 5 ceramics were 336 ± 13, 329 ± 10 and 327 ± 15 GPa, respectively.

Elastic modulus of a material is a function of the bond strength and is insensitive to microstructure. The slight variation in the Young's modulus of the Dy- α -SiAlON ceramics could be due to the content of the intergranular phases.



Figure 6. Vickers hardness (A) and fracture toughness (B) of the Dy-α-SiAION ceramics as a function of indentation load. The hardness value shows an increase dependence on indentation load while the fracture toughness shows a rising *R*-curve behaviour

Vickers hardness

Vickers hardness of the Dy- α -SiAlON ceramics measured at different indentation loads (1, 3, 5, 10 and 20 kg) is shown in Fig. 6a. As the indentation load was increased from 1 kg to 10 kg, the Vickers hardness value for a specific composition showed a decrease trend in certain range. When the indentation load was further increased to 20 kg, however, the hardness value did not decrease any more in comparison with 10 kg indentation. This hardness value increase as indentation load decreases is more common in nano hardness test. Vickers hardness of the Dy-0 ceramics was 19.7 ± 0.2 at 20 kg indentation load and 19.6 ± 0.4 GPa at 10 kg load (Fig. 6). It was increased to 20.2 ± 0.5 , 20.4 ± 0.4 and 21.2 ± 0.7 GPa at 5, 3 and 1 kg load. The hardness at 1 kg load was about 7.6% higher than that at 20 kg. The other samples Dy-0.5, Dy-1, Dy-2.5 and Dy-5 showed the same dependence of Vickers hardness on the indentation load.

In Fig. 6a, Vickers hardness of the Dy- α -SiAlON ceramics shows a slight decrease as the addition of excess Dy₂O₃ increased. The Vickers hardness values of each sample at 20 kg indentation load are listed in Table 4. The Dy-0 composition showed the highest Vickers hardness of 19.6 ± 0.2 GPa. The hardness values of the Dy-0.5, Dy-1, Dy-2.5 and Dy-5 ceramics were 19.3 ± 0.6, 19.1 ± 0.4, 19.0 ± 0.3 and 18.7 ± 0.1 GPa, respectively. The hardness changes may be related to the content of intergranular phase. The grain coarsening, as shown in Fig. 5, will cause a slight decrease in Vickers hardness.

Fracture toughness

When brittle materials such as ceramics and glass are subjected to Vickers hardness measurement, cracks initiate and propagate at corners of the indents. As shown in Fig. 7, crack propagation illustrated interfacial debonding, crack deflection, bridging and friction pullout of the large elongated α -SiAlON grains as toughening mechanisms [33–35]. However, large α -SiAlON grains would fracture if incident angle of the crack tip to the grain axis is larger than a critical value determined by interfacial bonding strength.

Fracture toughness of the Dy- α -SiAlON ceramics was calculated from crack length, Vickers hardness and Young's modulus, Eq. 3 [41,42]. The results are presented in Fig. 6b, illustrating a significant fracture toughness increase as the indentation load increased from 1 to 10 kg (i.e. larger crack propagation length). Further increase of the indentation load to 20 kg, however, showed saturation of the fracture toughness. This *R*-curve behaviour was common in toughened ceramics, due to rendering of the toughening mechanisms with crack propagation length [11,35].

$$K_{IC} = 0.016 \cdot \left(\frac{E}{H}\right)^{0.5} \cdot \frac{P}{c^{1.5}}$$
(3)

The Dy-0 sample demonstrated crack propagation with an average length of $64.7 \pm 2.7 \,\mu\text{m}$ at indentation load of 1 kg, and the fracture toughness was calculated to be $3.41 \pm 0.10 \,\text{MPa} \cdot \text{m}^{1/2}$. When the load is increased to 3, 5 and $10 \,\text{kg}$, the crack propagation length increased to 132.5 ± 5.7 , 181.3 ± 17.6 and $277.3 \pm 16.8 \,\mu\text{m}$, respectively, to estimate a fracture toughness value of 3.56 ± 0.11 , 3.73 ± 0.17 and $4.00 \pm 0.15 \,\text{MPa} \cdot \text{m}^{1/2}$. As indentation load was further increased to $20 \,\text{kg}$, the cracks propagated to an average length $444.7 \pm 28.8 \,\mu\text{m}$, to calculate a fracture toughness value of $3.94 \pm 0.18 \,\text{MPa} \cdot \text{m}^{1/2}$, indicating saturation of the toughening mechanisms at $20 \,\text{kg}$ indentation load.

In Fig. 6b, fracture toughness values of the Dy- α -SiAlON ceramics were compared in terms of different



Figure 7. SEM secondary electron images of crack propagation at Vickers hardness indent corners in (A,B) Dy-0, (B,C) Dy-2.5 and (E,F) Dy-5 (fracture toughness could be calculated by crack propagation length - interfacial debonding, crack deflection, bridging and friction pull-out as toughening mechanisms are demonstrated)

 Dy_2O_3 content. The compositions of Dy-0.5, Dy-1, Dy-2.5 and Dy-5 showed the same *R*-curve behaviour as Dy-0 which was detailed in the previous section. The fracture toughness values calculated at 20 kg indentation load are listed in Table 4 for comparison. In contrary to the generally accepted idea that development of a bimodal microstructure could induce *R*-curve fracture to render better bending strength and fracture toughness [34,53,54], the Dy-0 composition had a microstructure with uniform distribution of fine grains, which showed higher fracture toughness relative to that of Dy-0.5, 1, 2.5, 5 (Fig. 6) where clear bimodal microstructures were formed. The better fracture toughness of the Dy-0.5 ceramics at 20 kg in Fig. 6 may be due to the larger aspect ratio of the large elongated grains.

Bending strength

Bending strengths of the Dy- α -SiAlON ceramics are listed in Table 4. The Dy-0 composition without excess Dy₂O₃ addition showed a strength of 518 ± 36 MPa. With excess Dy₂O₃ addition of 0.5 wt.%, bending strength of Dy-0.5 was increased to 622 ± 41 MPa. Other compositions with 1–5 wt.% excess Dy₂O₃ additions, though slightly lower than that of Dy-0.5, all had bending strength around 600 MPa. Thus, bending strengths of the Dy-1, Dy-2.5 and Dy-5 materials were $616 \pm 39, 609 \pm 52$ and 594 ± 47 MPa, respectively.

The slight decrease in bending strength as the Dy_2O_3 excess additions were increased from 0.5 to 5 wt.%, may be due to over growth of the large elongated grains in the bimodal microstructures, Fig. 5. Critical crack size initiating premature fracture of the materials was estimated by the Griffith's fracture theory, Eq. 4 and the results are listed in Table 4. The critical crack was estimated to be ~18.4 µm in the Dy-0 sample. This value was much larger than the size of the grains (width ~2.4 µm and length ~10 µm, Fig. 5a) indicating other defects (usually processing flaws) rather than grain growth initiate fracture in the Dy-0 ceramics. The grain size in Dy-0 could estimate a bending strength as high as 703 MPa by Eq. 4.

In the compositions with excess Dy_2O_3 additions, the calculated critical crack sizes were in the range 10.1–14.3 µm. This value range was consistent with diameters of the large elongated grains in the bimodal microstructures. This means that structural defects causing premature fracture of the Dy-0 ceramics at a lower strength, could be eliminated by excess addition of 0.5–5 wt.% Dy_2O_3 . However, over-grown SiAlON grains should be avoided in view of increasing bending strength [34,53,54]. This indicates that a better self-toughening bimodal microstructure should consists of thin grains with large aspect ratio rather than over-grown large grains.

SEM of fracture surfaces of the Dy- α -SiAlON ceramics is shown in Fig. 8. The rough surface morphology was a result of interfacial debonding, crack deflection, bridging and pull-out of elongated α -SiAlON grains during fracture, which must have acted as tough-



Figure 8. SEM secondary electron image of fracture surface of (A) Dy-0, (B) Dy-2.5 and (C) Dy-5: the large elongated grains in the bimodal microstructures were trans-sectioned (critical crack size calculated by the Griffith's theory was in agreement with diameters of the large grains)

ening mechanisms in consistency with the increasing *R*-curve behaviour of fracture toughness with the crack propagation length, Fig. 6b.

On the fractographs shown in Fig. 8, transgranular fracture of large grains is demonstrated. The sizes of the transgranularly fractured large grains were consistent with the calculated critical crack sizes (10.1– 14.3 μ m). Therefore, it was assumed that when the bimodal microstructures were subjected to stress, load was mainly born by the large elongated grains while the fine-grained matrix transferred the load. However, once the large grains break, local stress concentrations around the large grains cause immediate catastrophic



Figure 9. (A) Flank wear, (B) nose wear and (C) notch wear of the Dy-α-SiAlON cutting inserts as a function of cutting length against Inconel 718, reflecting cutting performance of the tool inserts

fracture of the material. Therefore, again, an ideal selftoughening bimodal microstructure should consists of thin grains with large aspect ratios to avoid such premature fracture.

Cutting performance

Flank, nose and notch wear of the Dy- α -SiAlON cutting tool inserts tested by turning Inconel 718 nickel alloy are shown in Fig. 9. In this test, depth was measured from the top surface of the tool inserts to the tip of the wear marks.

A complete plot for cutting performance of a tool inserts is characterized by the S-like curve that would consist of three stages, i.e. stable wear, rapid wear and ultimate failure of the tool inserts. The transition point from the first stage (stable wear) to the second state (rapid wear) was of indicative significance for a rapid performance test of a cutting insert. So during the cutting performance test for each composition, the experiments were stopped after appearance of the transition. In Fig. 9a, the flank wear showed a steady increase for the Dy-2.5 composition as the cutting length increased to 852 m. The Dy-1 and Dy-0 ceramics showed stage I to stage II transition at a cutting lengths of 284 and 568 m, respectively. Figure 9b showed sudden worsening of nose wear at a cutting length of 284 m for the Dy-0.5 composition, while the other compositions could safely complete 568 m of cutting length. Notch wear of the tool inserts is shown in Fig. 9c. The notch depth illustrated a steady wear of 0.3–0.5 mm at 284 m cutting length. The decrease in curve slopes at higher cutting length indicated saturation in term of notch wear.

Figures 10 and 11 are stereomicroscope images of the wear marks on the Dy- α -SiAlON cutting inserts at the flank, nose and back flank positions. Severe friction during ultra-high-speed cutting could have caused a high temperature locally at the nose, which may excess 1000-1200 °C as evidenced in the literature [23,26,31,32,55]. Slight oxidation, diffusion and interaction between the nickel alloy work and the tool material lead to discoloration of the tool nose. However, there was no sign of significant deformation of the edge, neither chemical reaction attack nor built-up edge (BUE) was observed. These observations revealed high reaction resistance of α -SiAlON ceramics against nickel work during the cutting operation test, a good reason for application of α -SiAlON cutting tools for roughing nickel alloys.

In Figs. 10 and 11, attrition wear was minimal whereas premature fracture and notch wear were demonstrated as the main mechanisms for fool failure. For detail, Figs. 10a,b,c showed fracture at the tool tip in scale of 0.5–1 mm at the tool nose location and a type of notch wear at the position where the outer edge of the chip crossed the tool edge. Brittle fractures of a smaller scale were also present inside the notches. Therefore, it was assumed that stresses (usually of a tensile nature) at the delicate nose/edge of the tool inserts induced cracking and ultimately failed the tool inserts. The premature fracture pointed to the large elongated grains with a size that exceeds critical flaw sizes in the bimodal microstructures to initiate premature cracking, consistent with bending strength analysis in the previous sections.

Based on the failure analysis of the cutting tools, it seems that bimodal microstructure that is thought to toughen α -SiAlON ceramics might not always bring better performance in cutting tool inserts as expected, maybe because the over-grown large elongated grains forming the bimodal microstructure cause premature fracture as critical flaws at the tool edge position. The optimal microstructure of α -SiAlON cutting tools should consist of thin grains with large length to avoid initiating catastrophic crack propagation.

IV. Conclusions

Fully dense Dy doped α -SiAlON ceramics could be prepared by hot pressing a powder mix of Si₃N₄, AlN, Al₂O₃ and Dy₂O₃ at 1900 °C under 30 MPa for 60 min.



Figure 10. Stereomicroscope images of wear mark at position of tool nose and edge on (A) Dy-0, (B) Dy-1, (C) Dy-2.5, (D) Dy-5 tool insert after turning Inconel 718 alloy at 300 m/min with a feed rate of 0.1 mm/rev - premature fracture and notch wear rather than attrition wear were the dominant failure mechanism



Figure 11. Stereomicroscope images of back flank wear mark on (A) Dy-0, (B) Dy-5 tool insert after turning Inconel 718 alloy at 300 m/min with a feed rate of 0.1 mm/rev. Fracture segmentation rather than attrition wear was observed.

The constituent phases were dominated by α -SiAlON, in combination with a trace concentrations of β -SiAlON and M' (Dy₂Si₃O₃N₄) phases. With excess addition of 0.5–5 wt.% Dy₂O₃ and using a Si₃N₄ powder containing a concentration of coarse α -Si₃N₄ particles, the coarse α -Si₃N₄ particles could nucleate growth of a few percent of large elongated α -SiAlON grains that are uniformly distributed on a fine-grained matrix to form selftoughening bimodal microstructures. The composition without excess Dy₂O₃ addition did not form such large elongated α -SiAlON grains and avoided formation of a significant bimodal microstructure.

Vickers hardness of the Dy- α -SiAlON ceramics showed a dependence on the indentation load to demonstrate a higher hardness value of 20.4–21.2 GPa at 1 kg in comparison with 18.7–19.7 GPa at 20 kg. The fracture toughness increased with the crack length (Vickers hardness indentation load) to illustrate an *R*-curve behaviour that was consistent with self-toughening effect of α -SiAlON ceramics. Fracture toughness values of 2.81–3.41 MPa·m^{1/2} were obtained at an indentation load of 1 kg and increased to 3.50–4.18 MPa·m^{1/2} at 20 kg.

Bending strength analysis identified over-growth of the large elongated grains in the bimodal microstructures as critical cracks to initiate premature fracture of the Dy- α -SiAlON ceramics. Fracture and notch wear rather than attrition wear at the tool nose and edge positions were the dominant reasons for tool failure when roughing Inconel 718 alloy at a speed of 300 m/min. Bimodal microstructures consisted of thinner grains with large aspect ratios that avoid large elongated grains as critical flaws to initiate catastrophic fracture at insert tips may benefit α -SiAlON cutting tools properties.

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