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Si_3N_4–TiN–SiC three particle phase composites for wear applications

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Abstract

A three phase ceramic composite consisting of a Si_3N_4 matrix reinforced with TiN and SiC particles was prepared by hot pressing. The Si_3N_4–TiN–SiC composite material was investigated for microstructure and mechanical properties. Dry wear tests were carried out and the results compared with two phase Si_3N_4–TiN and Si_3N_4–SiC composites. The Si_3N_4–TiN–SiC was found to have an interesting combination of high abrasive wear resistance, low coefficient of friction and high hardness, which could lead to its use in very interesting wear applications.

Keywords: B. Composites; C. Wear resistance; D. Si_3N_4; E. Cutting tools

1. Introduction

Ceramic materials for cutting tools and high wear resistance applications in demanding environments have seen significant commercial and scientific interest in the last 25 years. Initially commercial products based on alumina (Al_2O_3), silicon nitride (Si_3N_4) and sialon were developed and these have recently been complimented by particle reinforced composite and layered materials for machining a range of cast irons, steels and other metals. Of these, Al_2O_3–titanium carbide (TiC) composite tools are commercially available, in addition Al_2O_3–TiC composite tools with titanium nitride (TiN) coatings are also available, e.g. SPK ceramic inserts from Kyocera (Japan), Ingersoll (Germany). Furthermore, Si_3N_4 based tools with TiN–Al_2O_3 and titanium carbon nitride (TiCN)–TiN coatings are available from companies including NGK/NTK (Japan) and Kyocera.

Si_3N_4 ceramics reinforced with TiN particles have led to an interesting group of ceramic composite materials. The addition of the TiN particles (nano or micron sized) can lead to two main effects. Firstly, if enough TiN is introduced then a percolating network is introduced in the electrically insulating Si_3N_4 matrix and a conductive ceramic which can be electro-discharge machined (EDM) is created, which can lead to a reduction of diamond grinding costs when preparing components and cutting tips [1,2]. The second effect is that the introduction of TiN into the Si_3N_4 matrix can lead to a ceramic with improved mechanical properties including strength, fracture toughness and Young's modulus [3]. Si_3N_4–TiN composites have also been shown to have improved wear resistance over monolithic Si_3N_4 especially during dry sliding wear tests [4–6]. This has made Si_3N_4–TiN composites of particular interest for the application of cutting tool materials for irons, steels and other metals. Si_3N_4–TiN composites can be made by different methods including the use of TiN, TiO_2 or Ti starting powders using hot pressing and spark plasma sintering for the densification processes and by SHS (self-propagating high-temperature synthesis).

An alternative reinforcing particle used for Si_3N_4 ceramics is silicon carbide (SiC). SiC has a high hardness (typically HV=22–32 GPa), therefore SiC particles can lead in particular to an increase in hardness over the base matrix, thus improving

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the wear resistance and can also lead to an increase in the thermal conductivity when incorporated into a Si_3N_4 matrix [7–9]. Si_3N_4/SiC composites have been produced by different methods including co-mixing with SiC particles [10,11]. Compared to TiN the electrical conductivity is lower for SiC. The addition of SiC particles can also lead to improvements in other properties, e.g. fracture toughness and strength. Si_3N_4/SiC composites are also of interest for cutting tool applications. Recently Si_3N_4/SiC composites had been developed for cutting tips with a microstructure specifically designed for the high speed industrial machining of wood [12,13].

There has been recent interest in multi-phase ceramic composites, with some ceramic composites being developed with up to five different particle phases [14–17]. These multi-phase ceramic composites (e.g. B_4C–SiC–Si–TiB_2, ZrB_2–SiC–B_4C, Al_2O_3–ZrB_2–ZrO_2, Al_2O_3–TiC–ZrO_2, etc.) are investigated for a range of mechanical properties and applications at different temperatures [15–18].

In the current work we produce a composite by co-mixing three different starting powders (Si_3N_4, TiN and SiC) as the main constituents. To date we have not come across such a particle reinforced composite in the literature. These composites were sintered by hot pressing using rare earth oxides as sintering additives. The microstructure, mechanical properties and wear behaviour is compared against two phase Si_3N_4/SiC and Si_3N_4/TiN composites. The goal is to see the combined effect of the high hardness particles (SiC) and the self-lubricating particles (TiN) on the wear properties of the Si_3N_4/SiC/TiN composites compared to the two phase composites, and therefore, to make a material suitable for use as a cutting tool.

2. Experimental

Starting powders consisting of α-Si_3N_4 (grade M11, H.C. Starck, Germany), α-SiC (grade UF25, H.C. Starck, Germany) and TiN (grade C, H.C. Starck, Germany) were prepared with a sintering additive system containing Al_2O_3 (CT3000 SG, Alcoa), $La(OH)_3$ (Auer Remy GmbH, Germany) and Y_2O_3 (grade C, H.C. Starck, Germany). The properties of the starting powders are listed in Table 1. Four different compositions were prepared, two Si_3N_4–TiN composites with 20 and 30 wt % TiN, one Si_3N_4–SiC composite and one Si_3N_4–TiN–SiC composite. The starting compositions of the four composites are presented in Table 2 in volume%.

Table 1
Starting properties of raw powders and the sintering additives.

Powder	Average d_{50} [μm]	Spec. surface area [m^2/g]	Density [g/cm^3]
Si_3N_4	0. 6	12.71	3.02
SiC	0.22	27.33	3.05
TiN	1.7	3.2	5.33
Al_2O_3	0.7	7.4	3.86
$La(OH)_3$	0.45	9.85	4.23
Y_2O_3	0.8	14.7	4.52

Table 2
Starting compositions of the different composites in volume%.

Composition	Si_3N_4	TiN	SiC	Al_2O_3	Y_2O_3	$La(OH)_3$
Si_3N_4–20TiN	83.5	11.8	0	1.9	2.8	0
Si_3N_4–30TiN	76.9	18.7	0	1.8	2.6	0
Si_3N_4–SiC	66.7	0	27.5	0.7	1.6	3.5
Si_3N_4–TiN–SiC	69.0	8.9	16.3	0.4	1.7	3.7

A water based slurry was prepared and the powders were wet milled for 48 h on a roller mill in a PET bottle with 3 mm Si_3N_4 balls. After 48 h the average particle size of the slurry was measured using laser light diffractometry (LS230, Beckman Coulter, Germany). PEG 20000 was added as a binder and the slurry milled for a further 30 min. The slip was then sieved through a 63 μm mesh and spray dried into granulates. Granulation was performed using a Minor Hi-Tec spray dryer (Niro S/A Denmark). The density of the powders initially and after spray drying was measured by He-pycnometry.

The granulated powder was die pressed into two different disc sizes (20 mm and 50 mm diameter with heights of 3 and 5 mm respectively). These were subsequently hot pressed in BN coated graphite dies. The smaller discs of the Si_3N_4–TiN–SiC ceramic were hot pressed between 1750 and 1820 °C in N_2 with a pressure of 30 MPa and a dwell time of 30 min. The small discs were used to determine the optimum sintering temperatures and to prepare polished specimens for SEM, XRD and hardness measurements. The hot pressing conditions for the Si_3N_4–TiN and Si_3N_4–SiC had been previously determined [13,19]. Samples for XRD, SEM and micro hardness were prepared by diamond grinding and final polishing using 1 μm diamond paste. XRD was performed with a PAN analytical XPert Pro diffractometer from Philips, SEM was carried out on a HR-SEM (Hitachi S-4800, Japan). Vickers hardness was performed using a Leitz Wetzlar mini-load tester (Germany).

From the large discs which were hot pressed at 1800 °C and with a pressure of 35 MPa, bars of $3 \times 4 \times 45$ mm were prepared for mechanical testing. The bars were diamond ground as specified in EN843-1 with 45° chamfers being ground on the edges [20]. Four point bending strength tests were carried out with a 20/40 mm load span and fracture toughness (K_{Ic}) was measured using the single edge v-notch beam (SEVNB) method [21]. Young's modulus (E) was also measured on these test bars by pulse excitation using a Grindo-Sonic Mark 5 (Lemmens, Belgium).

Dry friction wear tests were performed using a ball-on-flat specimen testing configuration (SRV from Optimol, Munich, Germany) with linear reciprocal sliding based on ASTM G133 [22]. The upper oscillating specimen was a 6 mm diameter Si_3N_4 bearing ball with a specified $R_a = 7$ nm and a Vickers (HV10) hardness of 1600 (grade Cerbec SN-101C, Coorstek, Connecticut, USA), as 100Cr6 steel bearing ball was previously found to be too soft and adherent. The ball acts on the bottom specimen (block) at a preselected oscillation frequency (10 Hz), stroke (2 mm) and normal load (10 N), the setup has