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Microstructure, mechanical properties and machining performance of spark plasma sintered Al_2O_3–ZrO_2–TiCN nanocomposites

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Abstract

The effect of addition of nanocrystalline ZrO_2 and TiCN to ultrafine Al_2O_3 on mechanical properties and microstructure of the composites developed by spark plasma sintering (SPS) was investigated. The distribution of the nanoparticles was dependent on their overall concentration. Maximum hardness (21 GPa) and indentation toughness (5.5 MPa $m^{1/2}$) was obtained with 23 vol% nanoparticles, which was considered as the optimum composition. The Zener pinning criteria were also satisfied at this composition with grain size of the restraining nanoparticles ∼63–65 nm. Hardness of the composites follows the rule of mixtures; crack deflection and crack arrest by nanoparticles at grain boundaries along with mixed fracture mode led to high toughness in the nanocomposites. Cutting tool inserts were developed by SPS with the optimized composition and their machining performance was compared with commercial alumina based inserts. Increased toughness in the nanocomposite inserts reflects in the machining performance as the tool life improves drastically compared to that of the commercial inserts at high cutting speeds ≥500 m min^{-1}. This was attributed to differences in their failure modes; the commercial inserts fail catastrophically by fracture due to their low toughness whereas the nanocomposite inserts reach the tool failure criteria by crater wear at all machining conditions.

Keywords: Spark plasma sintering; Nanocomposite; Microstructure; Toughness; Alumina

1. Introduction

Alumina has been prepared by various sintering techniques over the decades;[1–3] however it possesses the intrinsic drawback of having low toughness (3.0–3.3 MPa $m^{1/2}$), restricting its use as structural materials such as cutting tools. Limiting grain size to submicron levels improves its hardness but toughness remains poor.[2] Various non-oxide and oxide additives such as TiC, SiC_w, and ZrO_2 have been added to alumina to overcome this limitation.[4,5]

Alumina–TiC composites are widely used as cutting tool inserts due to their ability to machine at higher speeds than cemented carbides and their superior hardness, toughness and strength compared to alumina. They are commonly known as ‘black ceramics’, having a composition of 70% alumina and 30% TiC.[4] Alumina–zirconia composites are also used as cutting tool inserts where zirconia toughens the alumina matrix by stress induced tetragonal to monoclinic martensitic phase transformation.[4] It is well known that addition of either oxide or non-oxide additives improve mechanical properties of alumina. In this context, addition of both oxide and non-oxide additives may be an attractive option as it may impart the beneficial effects of both the additives in the resulting composites. It becomes even more attractive if the oxides and non-oxides are nano sized as majority of the nano particles may remain at grain boundaries and interact with cracks leading to interesting features not observed in the conventional composites.

There are only a few reports on the addition of both an oxide and a non-oxide additive to alumina[6,7] and the resultant mechanical properties which leave ample opportunities to study this system in greater details. Sato et al.[6] have reported a relative density of 95%, with good hardness and fracture toughness in hot pressed Al_2O_3–TiC–ZrO_2 composite. The improvement in hardness was attributed to the dispersion of TiC in the Al_2O_3–ZrO_2 matrix and toughness to crack deflection. However, there was no microstructural evidence to substantiate the claims. Dong et al.[7] hot pressed Al_2O_3–TiC–ZrO_2 composites at 1923 K and

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observed some improvement in toughness over Al_2O_3–TiC. They reported a density of only ∼94% which is not sufficient for any structural applications. Machining tests were not carried out in these studies; hence the effect of improved hardness and toughness on the cutting performance of the materials could not be ascertained.

TiCN based ceramic composites are expected to exhibit better machining performances compared to TiC due to the presence of TiN in solid solution with TiC, possessing better thermal shock resistance essentially improving the tool life.[5] There is a report on the suitability of alumina–TiCN tools for machining hardened steel due to their superior flank wear resistance compared to other ceramic tools.[8] Recently, a study was carried out on the tool life and wear mechanism of Al_2O_3–TiCN tools in turning hardened alloy steels where crater wear is observed to be the dominant mode of tool wear.[9] However, a thorough structure–property–performance correlation was not carried out in any of the studies, nor was the additional role of zirconia explored.

Although there are several studies on the mechanical properties of alumina and alumina–zirconia in SPS,[3,10–15] there is only one report on SPS of Al_2O_3–TiC nanocomposites (excluding zirconia) where the authors have achieved good flexural strength but only a marginal improvement in toughness.[16] Table 1 summarizes the compositions, temperatures, grain sizes and mechanical properties of fully dense alumina and alumina based composites studied previously.

There are no reports available on SPS of Al_2O_3–TiCN–ZrO_2 nanocomposites, the influence of the nanoparticles on mechanical properties of the composites and the machining performance of cutting tool inserts developed from such nanocomposites. In this context, the present work was carried out with the specific objectives: (1) to synthesize Al_2O_3–TiCN–ZrO_2 nanocomposites by SPS for the first time, (2) optimize the composition that yields maximum hardness and toughness retaining a fine grain size, (3) develop standard cutting tool inserts with the optimum composition and (4) perform machining trials with the developed tool inserts and compare the wear and tool life with commercial inserts of similar compositions to justify the suitability of adding nanoparticles in cutting tool materials.

2. Experimental

2.1. Raw materials and powder preparation

This study involves commercial α-Al_2O_3 powders (Inframat Advanced Materials, Farmington, CT) with average particle size 150 nm, surface area ∼8.95 $m^2\,g^{-1}$, and purity 99.9%; TiCN powders (Impex Inc., Toronto, Canada) with particle size 50 nm, surface area ∼24.4 $m^2\,g^{-1}$ and purity 98.0% and ZrO_2 powders (Grade: TZ-3Y-E, Tosoh Corp., Japan) with nominal particle size 30 nm, surface area ∼15 $m^2\,g^{-1}$ and purity 99.7%.

Dispersion of nanopowders is difficult due to their tendency to agglomerate for minimizing their high surface energy. For homogeneous dispersion the following method was adopted[17]: slurries of the individual powders were prepared in distilled water with solid content ≤5%. The pH was maintained at 3 by adding concentrated nitric acid drop wise and stirring gently using a magnetic stirrer. The isoelectric point for alumina and zirconia are known to be ∼7 and the maximum value of zeta potential is ∼40 mV for pH values 2–4[18]; therefore a pH value of 3 was used. The composite powders were prepared by mixing the alumina slurry with that of ZrO_2 and TiCN having the requisite solid content to get the desired composition, sonicating them for ∼5 min and flocculating them by increasing the pH to 8 by adding ammonia solution. This procedure was also adopted for pure alumina to break the hard agglomerates. The composite slurry was then subjected to pressure filtration by pouring them in a Buckner funnel and pressurizing with continuous flow of Ar gas at high flow rate of 100 l min^{-1}. The water was filtered off and the cake subjected to drying in a vacuum oven at 373 K to get the dispersed composite powders of the desired compositions.

In a previous SPS study,[19] the zirconia content in alumina which led to maximum hardness and toughness was optimized using genetic algorithm. In the present work, this optimized zirconia content (9 wt%) was retained; nano TiCN was added in different weight percentages (15, 20 and 25 wt%) to make three compositions, one having the additive content almost similar to that of the commercially available 'black ceramic' insert, the second having a lower additive content and the third having a higher content. This was done to evaluate the effect of the

Table 1
Composition, temperature, density, and mechanical properties of fully dense alumina and alumina based composites reported previously.

Ref. no.	Composition	*T* (K)	*d* (μm)	HV_n(GPa)	Kc (MPa $m^{1/2}$)	Mode of sintering
1	Al_2O_3 + 0.25% MgO	1923	1.5	18 ± 0.5	3.3 ± 0.3	PS
2	Al_2O_3 + 0.1% MgO	1573	0.55	20 ± 0.5	–	HP
3	Al_2O_3	1423	0.6	21 ± 0.5	3.5 ± 0.5	SPS
7	Al_2O_3–TiC–ZrO_2	1923	0.5	94 HRC	5.3 ± 0.5	HP
8	Al_2O_3–TiCN	–	–	19.3 ± 0.5	4.5 ± 0.5	PS
10[a]	Al_2O_3	1473	0.5	–	–	SPS
11	Al_2O_3 + 0.125% MgO	1423	0.4	22 ± 0.4	4.5 ± 0.7	SPS
12	Al_2O_3–ZrO_2	1673	0.7	18 ± 0.4	5.1 ± 0.3	SPS
13	Al_2O_3–ZrO_2	1573	0.4	20.5 ± 0.5	3.6 ± 0.3	SPS
14	Al_2O_3–ZrO_2	1573	0.4	20.3 ± 0.3	–	SPS
15	Al_2O_3	1473	0.5–2.0	–	3–4.3 ± 0.3	SPS
16	Al_2O_3–TiC	1753	0.4	22 ± 0.3	3.9 ± 0.2	SPS

PS, pressureless sintering; HP, hot pressing.
[a] Bending strength of 800 MPa obtained at 1773 K.