Machinable and mechanical properties of sintered Al2O3-Ti3SiC2 composites

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Two-phase composites consisting of (1 – *x*) Al_2O_3 and xTi_3SiC_2 (*x* = 0–1) were prepared by spark plasma sintering (SPS). Sintered densities larger than 98% of theoretical density were achieved when the specimens were sintered at 1300◦C for 5 min (in vacuum, at pressure 30 MPa). When content of $Ti₃SiC₂$ increased up to 30 wt%, composites were found to be machinable—they could be drilled easily using conventional Fe-Mo-W drills or gravers. The mechanical properties of the $(1 - x)$ Al₂O₃-xTi₃SiC₂ composites were evaluated. The bending strength, Vickers hardness of the specimens had the following ranges: 428 ± 10.2 $(x = 0)$ to 673 \pm 15.4 Mpa $(x = 1)$ (bending strength at room temperature); 19.9 $(x = 0)$ to 4.0 GPa (*x* = 1) (Vickers hardness). ^C *²⁰⁰⁴ Kluwer Academic Publishers*

1. Introduction

Alumina (Al_2O_3) is a ceramic showing considerable promise for use in a number of engineering applications. It is widely used in areas where wear, chemical and/or heat resistance are required. However, some shortcomings such as poor machinability, and brittleness have to be overcome for its usage in industrial scale. Improving machinability of ceramic materials has become one of the attractive subjects for materials scientists since many years ago. Janet and Wu reported that the rare-earth phosphate composites as YPO_4/Al_2O_3 [1] and $LaPO_4/Al_2O_3$ [2] could be easily cut and drilled using conventional tungsten carbide metal-working tools.

Although the machinability of ceramics could be improved as mentioned above, many advantages of the ceramics such as strength, and other mechanical properties are partly sacrificed.

 $Ti₃SiC₂$ is a novel structural/functional material that combines the merits of both ceramics and metals. Briefly, it is electrically and thermally conductive, besides being easily machinable and resistant to thermal shock. It possesses high strength, high toughness, high melting point, low density and good thermal stability [3–9]. Especially, $Ti₃SiC₂$, a dramatic material with a plate, or layered shaped structure that has been deemed to have a good machinability recently attracted more attentions.

Present research aims to design and fabricate $(1 - x)$ Al₂O₃ x Ti₃SiC₂ ($x = 0$ –1) which could be machined using the conventional Fe-Mo-W drills while retain-

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ing the advantages such as the high strength could be remained.

2. Experimental

The raw materials used were Al_2O_3 (purity >99.9%, average size of 0.2 μ m) and Ti₃SiC₂ powder, which was synthesized directly from titanium (purity 99.5%, average size of 37 μ m), silicon(purity 99.8%, average size of 2 μ m), and graphite (purity 99%, average size of 15μ m) powders with solid-liquid reaction method [10]. Pure $Ti₃SiC₂$ powders were obtained [11] by removing TiSi2 using HF solution at room temperature, and then heating the powders in air at 500° C for 5 h, finally washing the powders with hot $(NH_4)_2SO_4 + H_2SO_4$ solution. The resulting powder had average particle size 10.0 μ m (purity higher than 99%). The powder mixtures of $(1 - x)$ Al₂O₃ *x*Ti₃SiC₂ ($x = 0$ to 1) were first blended by ball milling for 48 h. After milling, the slurry was dried with a rotary evaporator, then ground in mortar and pestle and sieving with 100 mesh. The sintering was performed by SPS at 1300° C in a 20 mm diameter graphite die. The pressure applied was 30 MPa. The vapor pressure during sintering was kept below 6 Pa.

The machinability of each specimen was tested using the conventional Fe-Mo-W drills. The drilling tests were done using a standard drill press operating 750 r.p.m. with a drop of water placed at the drill tip at the beginning of each run. The sintered specimens were tested by manually applying a fixed load of 39 N

Figure 1 XRD patterns of sintering specimen of $Al_2O_3/Ti_3SiC_2 = 3:7$ composites.

Figure 2 Relationship between density and weight fraction of $Ti₃SiC₂$ in Al_2O_3 -Ti₃SiC₂ composite.

Figure 3 A hole drilled by a metallic tol on the disk ($x = 0.5$).

to the drill while measuring the time taken to drill holes of fixed depth (5 mm).

The density of sintered samples was measured accurately by the Archimede's method. The phase constitution was analyzed by X-ray diffraction (D/MAX-3B X-ray diffractometer) with Cu K_{α} radiation. The Vickers hardness was measured on the surfaces with a load of 98 N. Three-point bending tests were preformed

to determine the strength with size 2 mm \times 3 mm \times 12 mm and cross-head speed of 0.5 mm/min. The microstructure of the specimen was observed by SEM.

3. Results and discussion

Fig. 1 shows the XRD patterns of sintering specimen of Al_2O_3 -Ti₃SiC₂ composites. Ti₃SiC₂ and Al_2O_3 are

Figure 4 The relationship of the drilling rate and the amount of $Ti₃SiC₂$ on a fixed load.

Figure 5 Hardness vs. composition of Al₂O₃-Ti₃SiC₂ composites.

Figure 6 Bending strength vs. composition of $Al_2O_3-Ti_3SiC_2$ composites.

the main phases in sintered $Al_2O_3-Ti_3SiC_2$ composites besides very small peaks belonging to TiC. Since the heater and die of sintering furnace are made from graphite, the reaction (1) might take place in the last period of sintering period, same phenomena also was observed in the hot pressing [12] and no evidence of the reaction between Al_2O_3 and Ti_3SiC_2 is detected.

$$
Ti_3SiC_2 + C \rightarrow 3TiC + Si \tag{1}
$$

Fig. 2 shows that density of the sintered samples increases with the rise of $Ti₃SiC₂$ content, due to the higher density of Ti₃SiC₂ compared with that of Al_2O_3 ceramic. The density reaches its maximum value in the pure $Ti₃SiC₂$ material. The sintered density (%) (bulk density/true density \times 100%) of the composites is higher than 98% of theoretical density.

The dense Al_2O_3 ($x = 0$) ceramic was not machinable, while the dense Ti_3SiC_2 ($x = 1$) ceramic was machinable [6]. Ti₃SiC₂ containing Al₂O₃ ($x = 0.3$) to 1) dense ceramics sintered at 1300◦C could be machined using the conventional Fe-Mo-W drills. Fig. 3 shows a hole drilled by a metallic tool on the disk $(x = 0.5)$. The hole was cleanly drilled, with no evidence of large scale cracking or chipping. As shown in Fig. 4, the drilling rate decreased as the amount of Al_2O_3 increased for a fixed load. The reason that the composites were machinable might be due to the formation and linking of cracks at the weak interfaces between the two phases as discussed by David *et al*. [1]. But the single-phase $Ti₃SiC₂$ was also found to be machinable, interfacial debonding cannot be the only mechanism involved. Another possible mechanism is that associated with the deformation bands within individual grains of $Ti₃SiC₂$.

Fig. 5 shows the relationship between the Vickers hardness (Hv) and the content of $Ti₃SiC₂$. The softer $Ti₃SiC₂$ phase dominates the hardness of the composites, falling first abruptly between 0–20 wt% and then more gradually at higher content of the $Ti₃SiC₂$. The Vickers hardness of composites was from 19.9 ($x = 0$) to 4.0 GPa $(x = 1)$.

The bending strength of Al_2O_3 -Ti₃SiC₂ system raises with the increasing of $Ti₃SiC₂$ content (as shown in Fig. 6). This indicates that $Ti₃SiC₂$ additions strengthen Al_2O_3 ceramic. The strength of pure Ti₃SiC₂ material reached maximum about 673 ± 15.4 MPa $(x = 1)$. This value is higher than the $Ti₃SiC₂$ sintered using conventional hot-press method.

Scanning electron micrographs of the fracture surface of composites for different $Ti₃SiC₂$ contents are shown in Fig. 7(a–d). The layered $Ti₃SiC₂$ grains can

Figure 7 The micrograph of composites with different ratio Al_2O_3 : Ti_3SiC_2 .

easily be identified in these micrographs. Besides the layered $Ti₃SiC₂$ grains, equiaxial $Al₂O₃$ grains can be seen. The size of Al_2O_3 grains was determined with the lineal intercept procedure. The average grain size for pure Al_2O_3 was about 6 μ m, while average grain size for Al_2O_3 on Al_2O_3 -10% Ti₃SiC₂ composite was only about 2 μ m. The grain growth of aluminum is clearly controlled by the presence of $Ti₃SiC₂$, presumably through a pinning mechanism.

It is interesting that $Ti₃SiC₂$ is layered structure like mica and graphite and has bonding of a metallic nature without strong in-plane Si-Si bonds. Analysis of the fracture surface, it is seen that pull-out and microplastic deformation of the layered $Ti₃SiC₂$ grains are evidence. Ti₃SiC₂ has a unique microstructure that contains many grains with micro-lamellae, as shown in Fig. 7d. Slip or shear deformation occurred at the planar boundaries between the lamellae. It is, therefore, conceivable that the inelastic deformation behavior originates from the slip or shear deformation that is operative even at ambient temperature [13]. From the above fractrographic analysis, we attribute drilled rate increased with the rise of $Ti₃SiC₂$ content to the layered structure, pull-out, and micro-plastic deformation of the $Ti₃SiC₂$ grains.

4. Summary

The machinable and mechanical properties of the dense Al_2O_3 -Ti₃SiC₂ composites were studied. The bending strength increases markedly with $Ti₃SiC₂$ content, Vickers hardness inverse ratio changes to the bending

strength. The machinability of composites was characterized using metallic drill. Ti₃SiC₂ containing Al_2O_3 $(x = 0.3 \text{ to } 1)$ dense ceramics sintered at 1300 \degree C could be machined using the conventional Fe-Mo-W drills. This could be attributed to morphology of $Ti₃SiC₂$.

References

- 1. B. JANET, B. D. MARSHALL, M. R. HOUSLEY and E. D. PETER MORGAN, *J. Amer. Ceram. Soc.* **⁸¹** (1998) 2169.
- 2. WU MIN, D. MIYAHARA, K. YOKOI, T. YAMAGUCHI, K. DAIMON, YASUO HIKICHI, T. MATSUBARA and T. OTA, *Mater. Res. Bull.* **36** (2001) 939.
- 3. J. J. NICKL, K. K. SCHWEITZER and P. LUXENBERG, *J. Less-Common Metals.* **26** (1972) 335.
- 4. R. PAMPUCH, J. LIS, L. STOBIERSKI and M. TYMKIEWICZ, *J. Eur. Ceram. Soc.* **5** (1989) 283.
- 5. T. EL-RAGHY, M. W. BARSOUM, A. ZAVALIANGOS and S . KALIDINDI, *J. Amer. Ceram. Soc.* **82** (1999) 2855.
- 6. M. W. BARSOUM and T. E L-RAGHY, *ibid.* **79** (1996) 1953. 7. T. EL-RAGHY, A. ZAVALIANGOS, M. W. BARSOUM
- and S . KALIDINDI, *ibid.* **80** (1997) 513.
- 8. I. M. LOW, S. K. LEE, B. LAWN and M. W. BARSOUM, *ibid.* **81** (1998) 225.
- 9. T. GOTO and T. HARAI, *Mater. Res. Bull.* **22** (1987) 1195.
- 10. S. ZHIMEI and YANCHUN ZHOU, *Scripta Mater.* 41 (1999) 61.
- 11. C. RACAULT, F. LANGLAIS and R. NASLAIN, *J. Mater. Sci.* **29** (1994) 3384.
- 12. T. E L-RAGHY and M. W. BARSOUM, *J. Appl. Phys.* **83** (1998) 112.
- 13. T. EL-RAGHY, P. BLAU and M. W. BARSOUM, *Wear* 238 (2000) 125.

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