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Preparation and mechanical properties of Fe_3Al/Al_2O_3 nano-/micro-composite①

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Abstract: Al_2O_3 matrix composites reinforced with Fe_3Al nano-particles were fabricated by hot processing at 1 450－1 600 ℃. The effect of Fe_3Al content on the densification, mechanical properties and microstructure of the composites was investigated. The results show that some elongated Al_2O_3 grains are observed. Fe_3Al particles are mainly situated at grain boundaries of the matrix while smaller particles are trapped within the alumina grains. The addition of Fe_3Al nanoparticles improves the mechanical properties of alumina. The maximum strength and toughness of the Fe_3Al/Al_2O_3 nanocomposites are 832 MPa and 7.96 $MPa \cdot m^{1/2}$, respectively.

Key words: alumina; iron aluminide(Fe_3Al); composite
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1 INTRODUCTION

Alumina(Al_2O_3) ceramics are often considered for structural applications, due to their properties of high hardness, chemical and wear resistance and good mechanical properties at room and high temperature. The well known limitation for these ceramics, however, is the very low toughness. Recent studies have shown significant improvements in mechanical properties, including toughness, by adding ductile second phase particles, such as Ni, Al, Mo and Cu[1－4]. An increase of 80%－333% in the fracture toughness has been reported for Al_2O_3 with 20% Ni(volume fraction). The toughening mechanisms are crack bridging, crack deflection and network microstructure of Ni existed on Al_2O_3 grain boundary[1]. However, the presence of metallic particles degrades high temperature strength and oxidation resistance because of low melting point of metallic phases and poor wettability between metals and ceramics. Intermetallics have high melting point[5, 6]. They are also potential reinforcing phase of Al_2O_3, and have better chemical inertance and oxidation resistance than most metals have. Tuan et al[7, 8] researched the $NiAl/Al_2O_3$ composites containing 0－100% NiAl. In their work, the strength increased by 60% and the fracture toughness increased by 160% for the Al_2O_3 with 50% NiAl(volume fraction). ZHANG et al[9] observed an increase in strength and toughness by 39% and 42%, respectively, for Al_2O_3 with 40% Fe_3Al which has been used as cutting tool.

The mechanical properties of composites are known to be improved significantly when the size of dispersing particles is reduced from micrometer to nanometer. The most significant achievements with this approach have been reported by Niihara who firstly revealed that a dispersion of 5% SiC(volume fraction) nanoparticles into Al_2O_3 increased the room-temperature strength from 350 MPa to 1.0 GPa, accompanied with the fracture toughness increasing from 3.25 $MPa \cdot m^{1/2}$ to 4.7 $MPa \cdot m^{1/2}$. Proposed toughening mechanisms included crack deflection and microcracking induced by thermal expansion mismatch between the particles and the matrix grains[10].

The purpose of the present study is to fabricate Fe_3Al/Al_2O_3 nanocomposites. The mechanical properties and microstructure are also investigated.

2 EXPERIMENTAL

Commercial α-Al_2O_3 powder (Zhangjiakou East Special Ceramic Material Co, LTD, China) with an average grain size of 3 μm was used as matrix. Fe_3Al nano-powder was prepared by H_2-arc plasma technique, and mean particle diameter was 50 nm[11]. 5%－30% (mass fraction) of Fe_3Al nanoparticles were added to the starting Al_2O_3 powder. The mixture was ultrasonically dispersed and then ball-milled in ethanol for 1h. The slurry was dried in vacuum and then

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passed through sieves to get powders ⩽75 μm. The mixed powders were placed into a graphite die and hot pressed under 30 MPa in a flowing nitrogen atmosphere at 1 450 ℃–1 600 ℃ for 30min. For the sake of comparison, a monolithic alumina specimen was hot pressed at 1 530 ℃ for 30 min. The dimension of the hot pressed specimen was d42 mm × 5 mm.

Density measure of the as-sintered samples was using the Archimedes' method. Phase identification was performed by X-ray diffraction analysis (XRD, D/max-rA, Japan) with CuK_α radiation. Rockwell hardness (HRA) was tested. Microstructure of the composites and crack propagation behavior were observed by scanning electron microscopy (SEM, Hitachi S-2500, Japan). Microstructural characteristics were further examined by transmission electron microscopy (TEM, Hitachi S-800, Japan). Grain size was estimated by line intercept method. The sintered specimens were cut with a diamond saw and ground into 2 mm × 4 mm × 36 mm and 3 mm × 4 mm × 36 mm for determination of fracture toughness and bending strength, respectively. The fracture toughness, K_{IC} was measured by single-edge notch beam (SENB, the notch width of 0.25 mm and the depth of 2 mm) method. Bending strength was measured by three point bend method. The cross head speed was 0.5 mm/min with a span of 20 mm. The bending strength and toughness reported for each sample were the average values of 6–8 specimens.

3 RESULTS AND DISCUSSION

The XRD pattern for an Fe_3Al/Al_2O_3 composite after hot pressing is shown in Fig. 1. The XRD analysis indicates that no phase other than Fe_3Al and α-Al_2O_3 is produced after the hot pressing.

Fig. 2 and Fig. 3 show the SEM photograph of polished surface and the TEM micrograph of 5% Fe_3Al/Al_2O_3 composite hot pressed at 1 530 ℃, respectively.

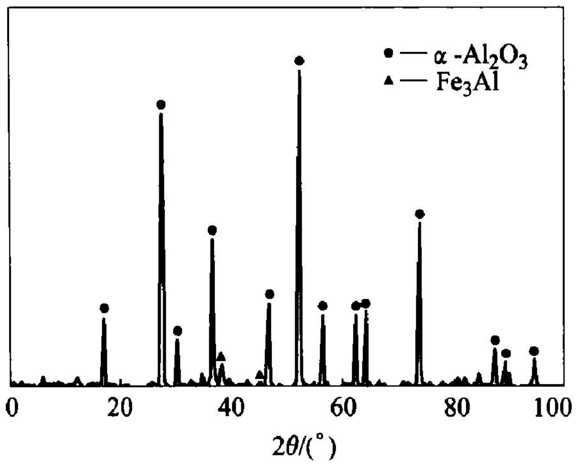

Fig. 1 XRD pattern for 5% Fe_3Al/Al_2O_3 composite after hot pressing at 1 530 ℃

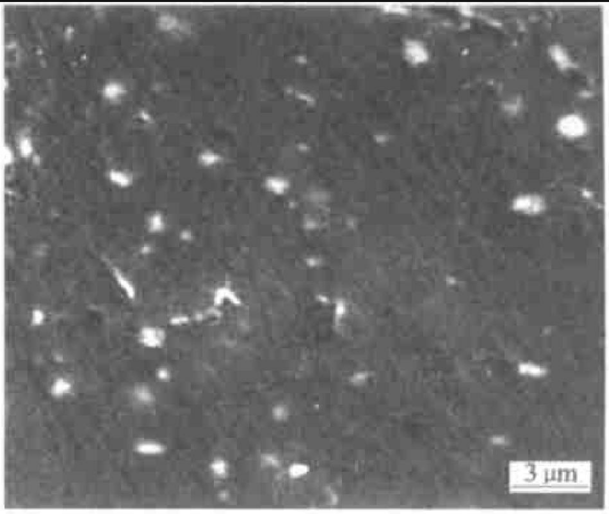

Fig. 2 SEM morphology of polished surface of 5% Fe_3Al/Al_2O_3 composite sintered at 1 530 ℃

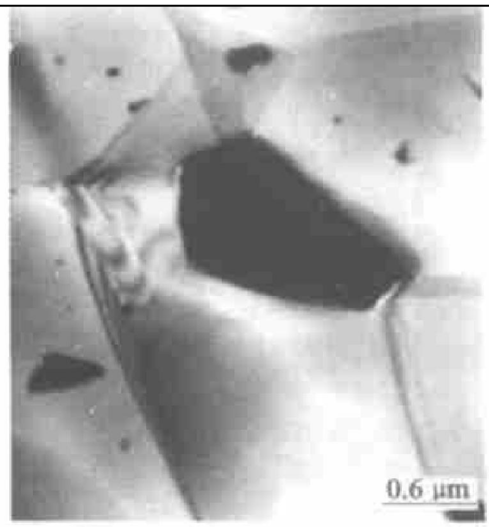

Fig. 3 TEM micrograph of 5% Fe_3Al/Al_2O_3 composite sintered at 1 530 ℃

Fe_3Al particles appear a white contrast in SEM micrograph, but dark contrast in TEM micrograph. In the composite, the Fe_3Al particles were uniformly dispersed in Al_2O_3 matrix. Many large ones of 600 nm in average size were observed at grain boundaries, and some nano-Fe_3Al particles were found to be embedded in the matrix grains. Grain size of the intragranular particles varied from several to several hundred nanometers.

The relative density of monolithic Al_2O_3 sintered at 1 530 ℃ is 98.3%, which notes that the ceramic has compacted. Fig. 4 shows the relative density of the composites containing 5%, 10%, 20% and 30% nano-Fe_3Al particles (noted as FA5, FA10, FA20, FA30) changed with the sintering temperature. The relative density increased with the increase of sintering temperature, but decreased with the increase of Fe_3Al contents. At 1 560 ℃ the densities of FA5, FA10 and FA30 were 99.4%, 98.3% and 88%, respectively. At

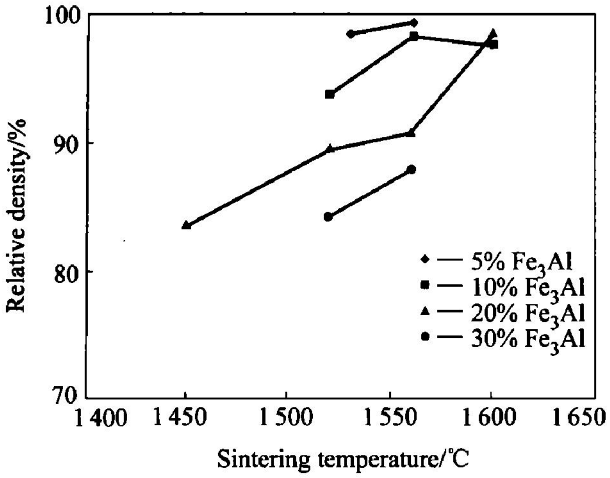

Fig. 4 Relative densities of Fe_3Al/Al_2O_3 composites with different Fe_3Al contents at different sintering temperatures

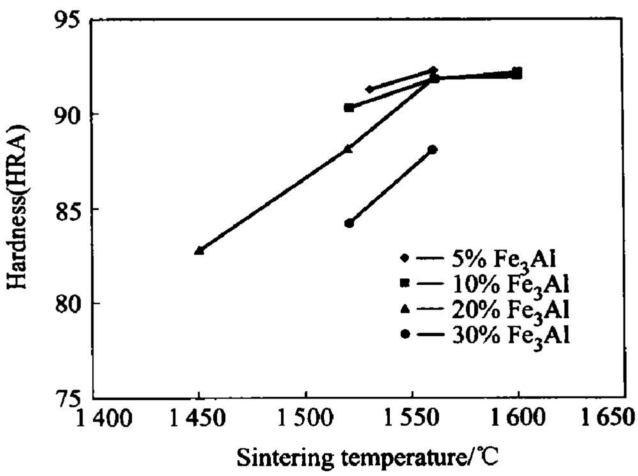

Fig. 5 Variation of hardness of Fe_3Al/Al_2O_3 nanocomposites with variable Fe_3Al contents at different sintering temperatures

1 530 ℃, the density of FA5 was 98.6%. To get the same density, FA10 and FA20 needed to be sintered at 1 560 ℃ and 1 600 ℃. The second phase of nano-Fe_3Al inhibits the densification of Al_2O_3 matrix.

As shown in Fig. 5, the Rockwell hardness (HRA) of the composite is the function of Fe_3Al contents and sintering temperature. Compared with Fig. 4, the variation of the hardness with Fe_3Al content and sintering temperature is similar to that of the relative density of the composite, implying the dependence of hardness on the densification of Al_2O_3 matrix. The highest hardness value of the composites is HRA 92.

Fig. 6 shows SEM fractographs of the Al_2O_3 and the Fe_3Al/Al_2O_3 nanocomposites. The microstructure of monolithic Al_2O_3 sintered at 1 530 ℃ is characterized by platelet-shaped grains with an

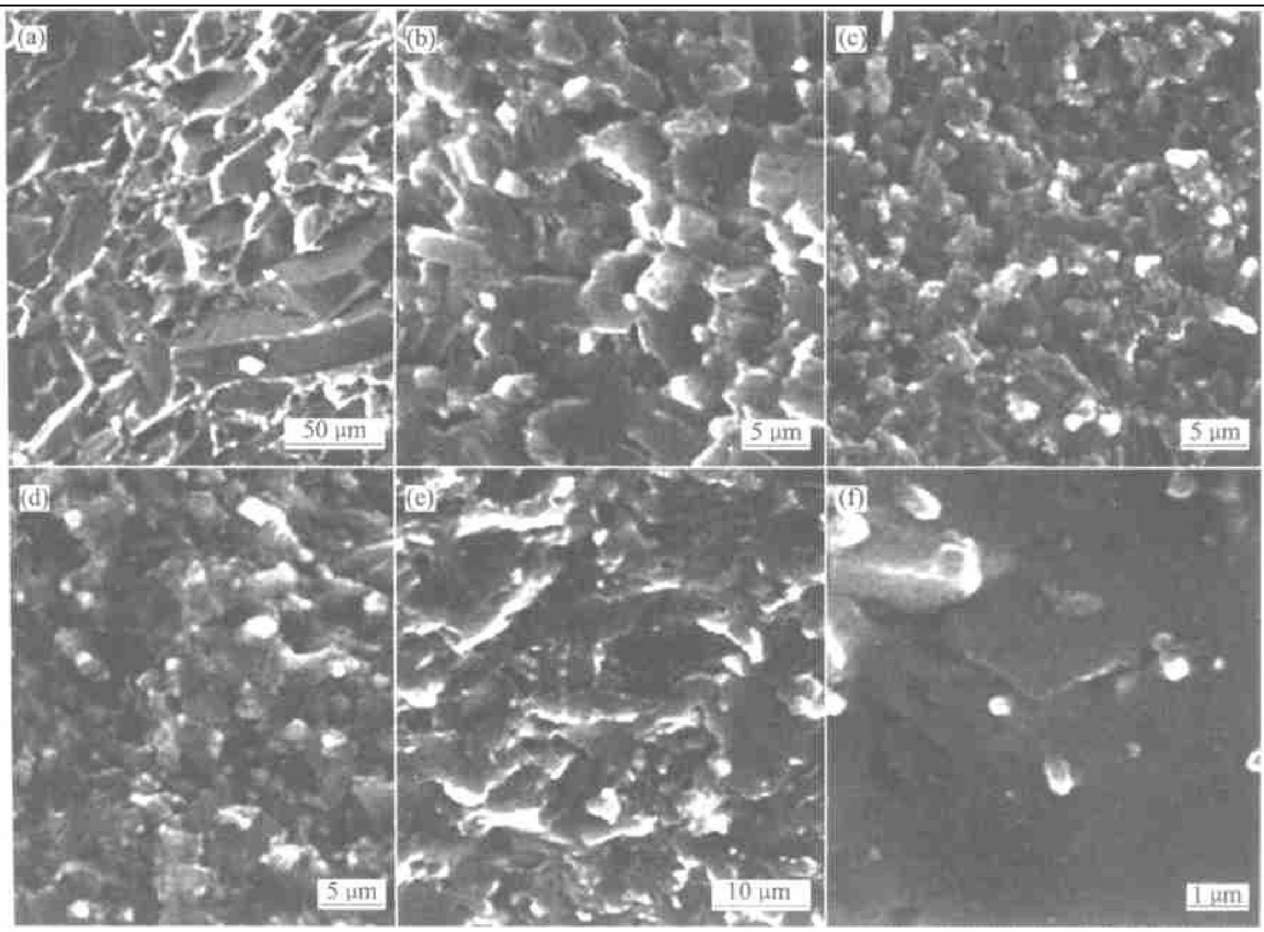

Fig. 6 SEM fractographs of monolithic Al_2O_3 and Fe_3Al/Al_2O_3 composites sintered at different temperatures
(a) —Monolithic Al_2O_3, 1 530 ℃; (b) —5% Fe_3Al-Al_2O_3, 1 530 ℃; (c) — 10% Fe_3Al-Al_2O_3, 1 520 ℃; (d) —20% Fe_3Al-Al_2O_3, 1 520 ℃; (e) and (f) —10% Fe_3Al-Al_2O_3, 1 600 ℃

aspect ratio of 4.2 and with average size of 91 μm. The grains of the nanocomposites are smaller and less anisotropic, with an average size of about 8 μm. The phenomenon of the preferential growth of Al_2O_3 grains has been well investigated[11]. The nonuniformly distributed aluminosilicate glassy phase in Al_2O_3 grain boundaries, caused by small amounts of impurities of the original material, attributed to the anisotropic morphology[11].

In the composite containing 5% Fe_3Al sintered at 1 530 ℃, the elongated Al_2O_3 grains with an average size of 12 μm and approximated aspect ratio of 3.2(Fig. 6(b)) are smaller than those of monolithic Al_2O_3. The fracture mode includes intergranular and transgranular. With the increase of Fe_3Al nano-particles, the grain size and aspect ratio of the matrix decreased. In 20% Fe_3Al/Al_2O_3 composite sintered at 1 520 ℃, the elongated grains were hardly to be found (Fig. 6(d)). The composite had high porosity, showing intergranular fracture. This indicated that the addition of Fe_3Al nano-particles result in grain-growth inhibition of the Al_2O_3 matrix. At higher Fe_3Al contents and sintering temperatures, slight grain growth of Al_2O_3 and the coalescence of Fe_3Al particles at the Al_2O_3 grain boundaries were observed.

The bending strength and fracture toughness of the monolithic Al_2O_3 sintered at 1 530 ℃ are 359 MPa and 4.6 $MPa \cdot m^{1/2}$, respectively. Fig. 7 illustrates K_{IC} and bending strength of the Fe_3Al/Al_2O_3 nanocomposite versus the Fe_3Al content and hot pressing temperature. On the whole, these mechanical properties were increased by addition of Fe_3Al particles. Bending strength of the 5% Fe_3Al/Al_2O_3 composite sintered at 1 530 ℃ was 666 MPa, which was 1.8 times higher than that of the monolithic Al_2O_3 prepared under the same conditions. When the hot pressing temperature was increased to 1 560 ℃, the bending strength of FA5 increased to a maximum value of 832 MPa, whereas the strengths of FA10 and FA20 were 722 MPa and 701 MPa, respectively. Consequently, strengthening of the composite was assumed to be attributed to the fine microstructure and the densification of the composite. At 1 560 ℃, the fracture toughness of the composite with 5% Fe_3Al was 7.34 $MPa \cdot m^{1/2}$, and those of FA10 and FA20 were slightly higher. When hot pressing temperature was elevated to 1 600 ℃, the fracture toughness of FA10 increased to 7.96 $MPa \cdot m^{1/2}$, but the value of FA20 decreased to 5.87 $MPa \cdot m^{1/2}$.

The crack propagation behavior around the Vickers indentation for the 5% Fe_3Al/Al_2O_3 composite sintered at 1 560 ℃ is shown in Fig. 8. The cracks propagated with large deflection around the Fe_3Al particles and the in-situ formed platelet Al_2O_3 grains. The pull-out of the elongated grains

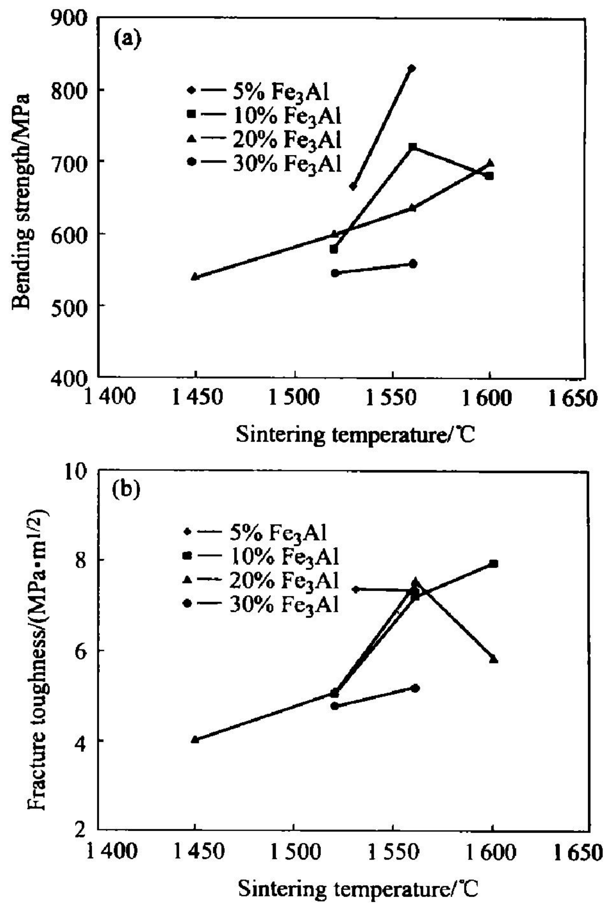

Fig. 7 Variation of bending strength(a) and fracture toughness(K_{Ic}, b) of Fe_3Al/Al_2O_3 nanocomposite with sintering temperature and Fe_3Al content

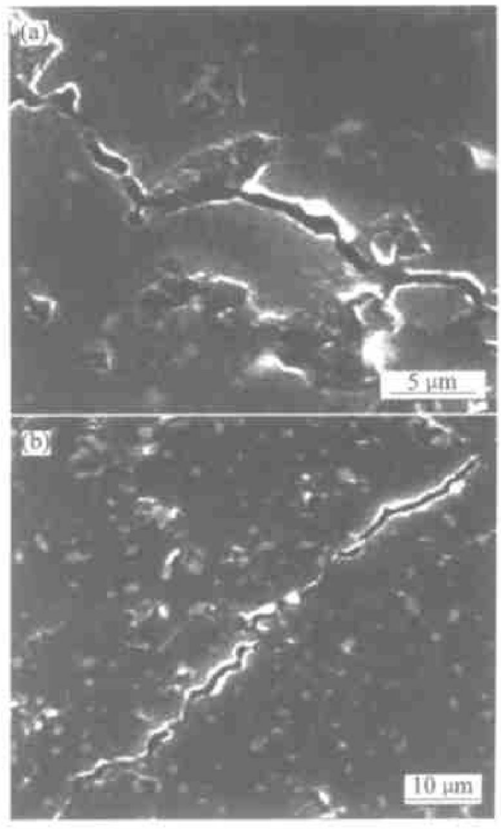

Fig. 8 SEM photographs of indentation crack propagation of nano-composites
(a) —5% Fe_3Al-Al_2O_3, 1 530 ℃;
(b) —10% Fe_3Al-Al_2O_3, 1 600 ℃

was found. In addition, crack penetration through the coalesced Fe_3Al particles and crack-bridging by the elongated Al_2O_3 grains were observed. The crack path is thus erratic, and the toughness of the composites is therefore enhanced. The crack path was almost straight in FA10 sintered at 1 600 ℃, even though the crack deflection and bridging of the Fe_3Al intermetallic particles were found. From the SEM fractograph of the composite, no elongated grains was found(Fig. 6(e)). The high fracture toughness likely results from fine grain. At a higher magnification the fracture surface of FA10 shows many Fe_3Al nano-particles trapped in the grains of the matrix(Fig. 6(f)). This microstructure was believed to be helpful to strengthening grain boundaries[11]. Thus, the cracks are forced to deflect into Al_2O_3 grains. Transgranular fracture is the main reason for its high toughness.

4 CONCLUSIONS

1) 5%−30% (mass fraction) Fe_3Al/Al_2O_3 nanocomposites are successfully fabricated by hot pressing under N_2 atmosphere. In these composites, nanometer Fe_3Al particles are dispersed within the Al_2O_3 grains, and relatively large Fe_3Al particles with about 600nm, are found at the grain boundary.

2) The addition of Fe_3Al particles result in the grain growth inhibition of the Al_2O_3 matrix and make the densification of the composite be difficult. The platelet Al_2O_3 grains are found in the composites. With the increase of Fe_3Al content, the aspect ratio and grain size of the elongated grains decrease.

3) The highest value of bending strength and fracture toughness of Fe_3Al/Al_2O_3 nanocomposites are 832 MPa and 7.96 $MPa\cdot m^{1/2}$, respectively. The strengthening mechanism is attributed to the fine microstructure and densification of the composites. The enhancement of toughness of the composite containing 5% Fe_3Al is due to the crack deflection and bridging effect of Fe_3Al as well as the pull-out effect of the elongated Al_2O_3 grains. In addition, the transgranular fracture caused by fine grain microstructure is the main reason for the high toughness of 10% Fe_3Al/Al_2O_3 nanocomposite.

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