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Fabrication and mechanical properties of ultra-fine grained γ-Ni–20Fe/Al_2O_3 composites

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Abstract

High-dense (relative density $D \geq 98\%$) ultra-fine grained γ-Ni–20Fe/Al_2O_3 composites were fabricated by using a mechano-chemical process plus hot-pressing. Microstructure investigations revealed that fine γ-Ni–20Fe particles with the dimensions of several hundreds of nanometers dispersed homogeneously at the matrix grain boundaries. Mechanical tests indicate that fracture toughness increases monotonously from 4.7 MPa $m^{1/2}$ for monolithic alumina to 8.4 MPa $m^{1/2}$ for ~19 vol.% Ni–20Fe/Al_2O_3 composites. Fracture strength σ_f increased with addition of Ni–20Fe phase as Ni–20Fe content was low (≤5 vol.%). However, σ_f decreased with further increase of Ni–20Fe content, exhibiting a maximum strength of ~600 MPa at ~5 vol.% Ni–20Fe. The improvement of the mechanical properties could be ascribed to the microstructure refinement of the composites and increase of cracking resistance introduced by the dispersed Ni–Fe phase, which was supported by fracture characteristic observations.

Keywords: Hot-pressing; B. Composites; C. Mechanical properties; D. Al_2O_3

1. Introduction

Metal/ceramic-based ultra-fine grained composites or nanocomposites in which nanometre-sized particles having a diameter from a few to several hundreds of nanometers are incorporated into ceramic matrices [1] are attractive because of their great potentials in improving the toughness of monolithic ceramics. Many efforts have been made on these nanocomposites. For instance, Ni/Al_2O_3 nanocomposite [2] was reported to have improved mechanical properties as well as attractive magnetic properties, which suggested their talent functional applications. Later, Fe/Al_2O_3 [3] and Ni–Co/Al_2O_3 [4] nanocomposites were successfully synthesized and their mechanical properties were explored.

The present work focused on γ-Ni–*x*Fe/Al_2O_3 composite system. γ-Ni–*x*Fe alloys with *x* being in the range of 10–65 wt.%, so called Permalloy, are important soft magnetic materials [5–8], which have been widely used in industry for things such as recording heads, transformers or magnetic shielding materials [5]. Besides, γ-Ni–*x*Fe alloys possess f.c.c. crystallographic structure with good ductility, and their thermal expansion coefficient changes with *x* [5], which suggests that, as a disperse phase, Ni–*x*Fe alloys can thermally match ceramic matrices if their compositions are chosen properly. Therefore, once they are incorporated into alumina, they could not only improve the toughness of monolithic alumina, but also introduce soft magnetism into the composite system, realizing the combination of superior mechanical properties with excellent magnetic performance. In this report, γ-Ni–20Fe/Al_2O_3 ultra-fine grained composites or nanocomposite were fabricated and its mechanical properties were investigated. A study on magnetic properties of γ-Ni–

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20Fe/Al_2O_3 and the effects of different Ni–*x*Fe alloys (with different *x*) on properties of Ni–*x*Fe/Al_2O_3 will appear elsewhere [9].

2. Experimental procedures

Commercially available nanometer-sized alumina (purity ≥99.99%) was wet ball milled in 200 ml alcohol for 5 h with high-purity Al_2O_3 balls to break up the agglomerates in high-energy ball-milling machine. The ball-to-powder ratio was 6:1 and the rotation speed was 250 r/h. The ball-milled alumina was sieved in 200-items sieve as the source material after dried at 60 °C for 24 h. On the other hand, high-purity $Fe(NO_3)_3{\cdot}H_2O$, $Ni(NO_3)_2{\cdot}H_2O$ powders were used as a source material for Ni–Fe. The composition of Ni–Fe alloy was Ni–20 at.% Fe. The Ni–20Fe contents in four different composites are 5, 9, 15 and 19 vol.%, respectively. Weighted powders were initially dissolved in 200 ml alcohol and then mixed with the ball-milled Al_2O_3 for 5 h. After dried at 60 °C for 24 h, the mixture was sieved in 200-items sieve, and then calcined at 500 °C in air for 2 h. In order to obtain composites with homogeneously dispersed particles and crush soft agglomerates of the calcined powder, the calcined powders were wet ball milled again in 200 ml alcohol for 4 h, and then dried and sieved as above. The re-milled powders were subsequently reduced at 600 °C for 2 h to form nanometer sized γ-Ni–20Fe alloy (hereafter refer to it as Ni–Fe) in situ. Bulk samples with size of ϕ 60 mm × (∼) 3.5 mm were obtained by hot-pressing at 1400 °C for 40 min under applied stress of 30 MPa in high purity (5 N) argon atmosphere. For comparison, a monolithic alumina sample was prepared under the identical condition.

The hot-pressed samples were ground, polished and finally cut into rectangular shaped specimens with 4 mm × 3 mm × 35 mm in size for bending tests. Sample phases were analysed with X-ray diffraction. Microstructures of powders were examined with transmission electron microscopy (TEM) and the Brunauer-Emmett-Teller (BET) method with nitrogen absorption. To reveal the grain morphologies and dispersion states of the composites, Ni–20Fe/Al_2O_3 and monolithic alumina specimens were thermally etched for 15 min at 1200 and 1400 °C, respectively. The morphology of thermally etched surfaces and fractography of bulk specimens were observed with field-emission-scanning electron microscopy (FE-SEM). Sample compositions were confirmed with energy dispersive X-ray spectroscopy (EDS). The densities of the hot-pressed specimens were measured based on Archimedes principle in alcohol with the accuracy of ±0.5%. Vickers indentation tester was used to measure the hardness (load: 1.96 N; loading time: 30 s) and fracture toughness of the specimens (load: 98 N; loading time: 15 s), for which at least six indentations were conducted. Fracture strength was obtained by three-point bending tests (span: 30 mm) of, at least, five specimens with a cross-head speed of 0.5 mm/min.

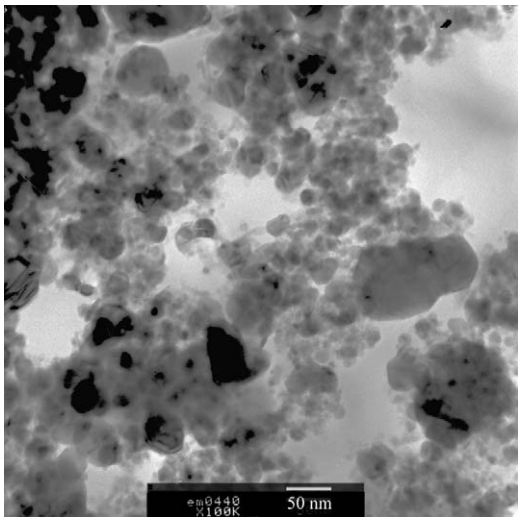

Fig. 1. TEM micrograph for the 15 vol.% Ni–20Fe/Al_2O_3 nanocomposite powder.

3. Results

3.1. Microstructures

The average particle sizes for powder samples with different Ni–Fe contents, after hydrogen reduction, were determined to be 13–50 nm. Fig. 1 gives a TEM micrograph that shows the particle morphologies for the 15% Ni–20Fe/Al_2O_3 powder. One can see that most of particles have sizes in the range from ∼10 to ∼40 nm with light agglomeration, which is beneficial to densification during subsequent hot-pressing. Table 1 lists the densities and compositions of the bulk samples. One can see that the relative densities of all samples are ≥98%. Although sample density differs a little for samples with different Ni–Fe contents, no clear relationship between density and the composition was found.

Fig. 2 shows XRD patterns for S1 and S2. As compared to the pattern of S1, there are some additional reflection peaks in the pattern of S2 that are consistent exactly with those peaks originating from γ-Ni–Fe alloy (as marked in the figure), indicating that there are only two phases in S2: one is γ-Ni–Fe and the other is α-Al_2O_3. In fact, apart from the two constituent phases, no other phase such as Fe, Ni or corresponding metal oxides was observed for all the composite samples with different compositions. This result indicates that Ni- and Fe-nitrates have been completely

Table 1
The Ni–20Fe content (*C*), absolute density (ρ), and relative density (*D*) for different samples after hot-pressing

	Sample				
	S1	S2	S3	S4	S5
C (vol.%)	0	5	9	15	19
ρ (g/cm^3)	3.91	4.17	4.36	4.66	4.86
D (%)	98.3	98.7	99.0	98.4	98.0