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Microstructure and properties of in situ nanometric Al_2O_3 reinforced α-Fe(Al)–Fe_3Al-based composites

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ABSTRACT

The alpha phase Fe(Al)–Fe_3Al-based composites reinforced with 4–9 vol.% α-Al_2O_3 dispersions were fabricated by hot-pressing sintering. Nanometer-sized α-Al_2O_3 dispersed homogeneously in the submicron α-Fe(Al) and Fe_3Al matrix, with a 20% lattice-mismatch presented between α-Al_2O_3 and α-Fe(Al). Higher fracture toughness (39 MPa $m^{1/2}$) and bending strength (1260 MPa) were obtained for composites containing both intragranular and intergranular Al_2O_3 grains. The combination of fine grain strengthening with dispersion strengthening can account for the improvement of mechanical properties.

1. Introduction

The ordered nature of iron aluminides intermetallics leads to attractive properties such as high strength, improved hardness and excellent oxidation and sulphidation resistance at elevated temperature. These excellent properties are the results both of reduced dislocation motion and low diffusivities. Unfortunately, these factors also associated with low room temperature strength and fracture toughness, which limited their large-scale industrial applications [1]. Their room temperature strength and wear resistance can be obviously improved by adding carbides or borides with typical grain sizes of 5–10 μm [2–4]. Compared with carbides or borides, oxide grains have the advantages of high oxidation resistance, chemically inert and high electrical resistivity, which make them candidates for reinforcing iron aluminide. Liquid phase sintering is not suitable for fabricating these composites due to the poor wettability of liquid iron aluminides with oxides. Reaction sintering is one way to fabricate composites containing oxide grains, such as FeAl/Al_2O_3 [5], FeAl/ZrO_2 [6], and FeAl/TiC/Al_2O_3 [6]. For iron aluminides composites, most of the investigations have been focused on the reinforcement with micrometric oxide grains. Few investigations concern with the fabrication of the nanometric particulates reinforced iron aluminide matrix composites [7]. In this work, an α-Fe(Al)–Fe_3Al-based composites reinforced with in situ nanometric Al_2O_3 particulates were successfully fabricated, and the mechanical properties, microstructural characterization and strengthening mechanism were investigated.

2. Experimental procedures

A powder mixture of Fe–28 at% Al was mechanically ground in a planetary ball miller at a rate of 360 rpm (rotations per minute). The grinding was conducted for 50 h at room temperature in a high-purity argon atmosphere. The weight ratio of the ball to the powder was 7:1. The as-milled powder was then subjected to heat treatment in an ultra low pressure of 2×10^{-5} Torr at 800 °C for 1 h. After incorporating a small amount of appropriate propanetriol as plastic binding additives, the mixture was shaped into pellets under an uniaxial pressure of 180 MPa. The pellets were then hot-pressed uniaxially at 40 MPa in a high-temperature furnace at 1100–1300 °C for 30 min. The heating rate was 50 °C/min and the pressure in the furnace was 2×10^{-5} Torr. The sintered samples were furnace cooled for subsequent examinations. Fig. 1 shows the sintered samples before and after polishing and cutting. Microstructural investigations were performed by using scanning electron microscopy (SEM; S2500, Hitachi, Japan), transmission electron microscopy (TEM, JEM2000FX, JEOL, Japan) with a Link EDS system with ultra-thin detector window for local chemical analysis, and high-resolution transmission electron microscopy (HREM, JEM2010, JEOL, Japan). TEM and HREM specimens were mechanically ground to a 150-μm thickness, dimpled to 30 μm, before thinned to perforation by an argon-ion beam. The grain size was

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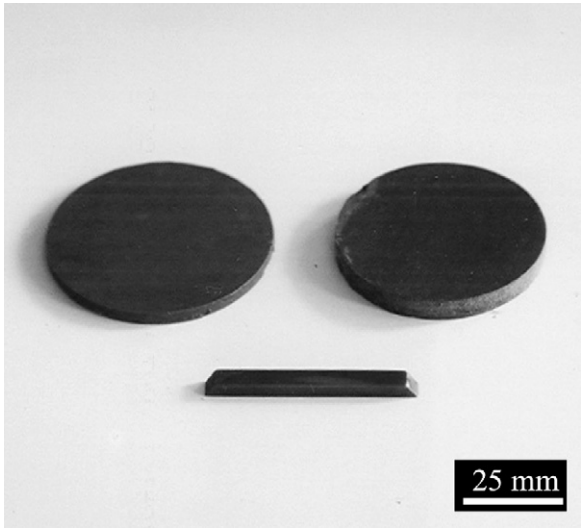

Fig. 1. Hot-pressed samples: before (discs) and after cutting and polishing (bar).

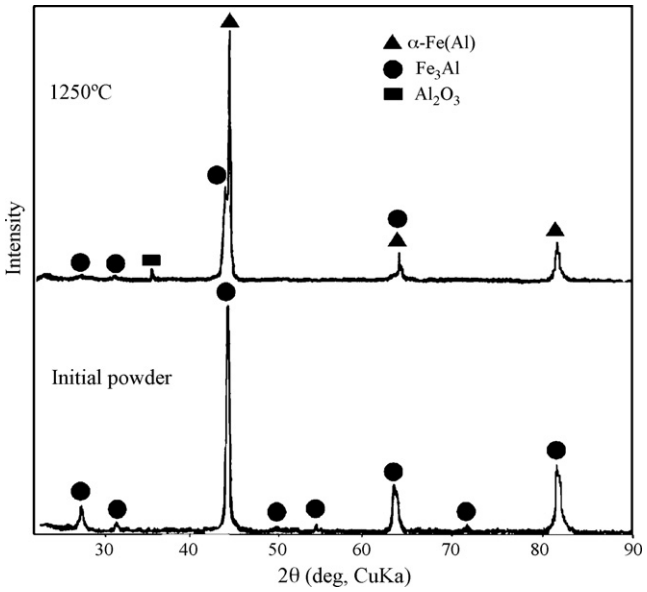

Fig. 2. X-ray diffraction patterns of (a) the initial powder and (b) the sample hot pressed at 1250 °C for 30 min.

measured at each microstructural state by image analysis and quantification software both in the SEM and TEM. Average sizes were determined as the equivalent circular diameter of grains, counting typically about fifty grains per state. Phase distribution was determined by X-ray powder diffraction (XRD; D/max-ra, Rigaku, Japan). Densities of the specimens were measured by the Archimedes immersion technique. Vickers indentations were made on the polished specimens with a maximum load of 296 N. The indenter was held for 15 s, to allow observation of crack propagation.

The mechanical properties of the samples were measured on their polished surfaces. Fracture-toughness tests were performed by the single-edge notched-beam (SENB) method (on specimens 2 mm × 4 mm × 36 mm in size, at a loading rate of 0.5 mm/min, with a span of 20 mm). A straight-through notch with a relative length, a/W = 0.5 was introduced at the center of the specimens by a diamond blade. The bending strength was measured by the three-point bend test (on specimens 3 mm × 4 mm × 36 mm in size). An average value was obtained from six individual measurements. HD-187.5 Brinell tester was used to measure the hardness of the samples.

3. Results and discussion

3.1. Microstructural characterization

Fig. 2 shows the XRD pattern of the sample hot-pressed at 1250 °C for 30 min. The XRD pattern of the initial powder mixture before sintering was also shown for comparison. The XRD data for initial powder and sintered sample are presented in Table 1. All peaks for initial powder can be well indexed to Fe_3Al of DO3 structure (PDF card #06-0695). It can be observed that the full width at half-maximum of peak at 44.6° decreased by 36.4% after sintering, indicating the grain growth and the relaxation of internal strains. Moreover, the peak at $d = 0.2047$ nm had split into two peaks after sintering. The peak with higher intensity ($d = 2.031$ nm) attributed to the cubic structure α-Fe(Al) solid solution (PDF card #06-0696), while that with lower intensity ($d = 2.047$ nm) attributed to the Fe_3Al of DO_3 structure. Peaks of α-Al_2O_3 (PDF card # 82-1468) were also observed in the XRD pattern of hot-pressed sample. Because the amount of α-Al_2O_3 was very low, only one characteristic peak (1 0 4) can be observed. Further EDS analysis was made to confirm the formation of α-Al_2O_3 (see the next paragraph). Although the milling had been carried out in a high purity argon and heat treatment and hot pressing in 2×10^{-5} Torr atmosphere, it is hard to avoid exposing to air as the initial powders were putting into the furnace. In addition, the milled powders were nanometer-sized, and they can trap more oxygen than coarse powders due to the high activity and specific surface area [8]. Similar phenomenon had occurred in NiAl fabricated by MA and hot-pressing sintering, in which α-Al_2O_3 formed in NiAl matrix during sintering [9]. The formation of the Al_2O_3 grains would cause the deviation of Al content from the initial powder, thus leading to form α-Fe(Al) solid solution and Fe_3Al, rather than the pure Fe_3Al phase. It was interesting to note that this result was different from that obtained by Schicker and Garcia [10]. In their work on Fe–Al/Al_2O_3, Fe and Al_2O_3 were the final phases as Al content <10 vol.%, whereas FeAl and Al_2O_3 would be formed as Al content >10 vol.%. Such a difference might result from different sintering conditions. In this work, the hot pressing procedures were carried out at temperature between 1100 °C and 1300 °C, while that of

Table 1
Experimental results of XRD.

d (nm)	*hkl*			*d* (nm)	*hkl*		
Initial powder	Fe_3Al	α-Fe(Al)	α-Al_2O_3	Sample sintered at 1250 °C	Fe_3Al	α-Fe(Al)	α-Al_2O_3
0.3356	1 1 1	–	–	0.3341	1 1 1	–	–
0.2801	2 0 0	–	–	0.2891	2 0 0	–	–
0.2047	2 2 0	–	–	0.2551	–	–	1 0 4
0.1786	3 1 1	–	–	0.2047	2 2 0	–	–
0.1673	2 2 2	–	–	0.2031	–	1 1 0	–
0.1445	4 0 0	–	–	0.1434	4 0 0	2 0 0	–
0.1292	4 2 0	–	–	0.1170	2 1 1	–	–
0.1182	4 2 2	–	–				