

Properties of Fe₃Al matrix composites with Al₂O₃ particle dispersions

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(Received September 21, 1992; in final form December 8, 1992)

Abstract

Fe₃Al matrix composites reinforced with 0–13 vol.% fine alumina particles were fabricated by hot pressing powder mixtures of iron and aluminium. The microstructure and mechanical and several other properties of the alumina-reinforced composites were examined. As a result of the dispersion of alumina powder, the grain size of the matrix decreased. No third phase was observed between the matrix and the alumina by transmission electron microscopy. The matrix alloy showed quite good tensile strength (above 800 MPa) as well as good elongation (up to 5%) at room temperature. On increasing the volume fraction of alumina, the tensile strength increased slightly. The 0.2% proof stress was improved considerably more than the tensile strength and reached its maximum value at an alumina volume fraction of 2.5%. At 500 °C the strength of the composite was higher by 100 MPa than that of the monolithic alloy. The oxidation resistance of the Fe₃Al and its composites up to 1000 °C proved to be quite good.

1. Introduction

Intermetallic compounds are expected to be one of the key high temperature structural materials in the next century. They show excellent strength up to high temperatures as well as superior toughness to ceramic materials. Many studies on intermetallic compounds have been reported in the past decade, especially on nickel aluminides because they were found to be quite ductile even at room temperature (see *e.g.* ref. 1). Titanium aluminides such as TiAl also have the potential for use as components in aircraft and space vehicles because they possess light-weight character in addition to good mechanical properties (see *e.g.* ref. 2). However, these intermetallic compounds quickly lose their strength at elevated temperatures, so in order to replace the present high temperature structural materials such as nickel-based superalloys, their strength at elevated temperatures must be improved. Titanium aluminides have another problem restricting their practical use. Their poor ductility at room temperature is a critical disadvantage for structural uses. Recent works have been focused on improving the ductility of this alloy [2].

In contrast to the two aluminides mentioned above, only a few studies on iron aluminides have been reported in spite of their good mechanical properties [3–8]. Among several iron aluminides, Fe₃Al is one of the

most interesting since it has similar good ductility to nickel aluminides. In addition, it can have wide industrial applications because of the relative cheapness of the raw materials iron and aluminium. However, Fe₃Al has two major problems in being used at elevated temperatures. One is the transformation from the DO₃ phase to the B₂ phase at about 550 °C. This temperature sets one of the limits on the use of this material for high temperature applications, though the transformation temperature can be increased by adding various alloying elements [4]. For example, 10% of chromium addition can raise the transformation temperature by 50 °C. Further modification is required for practical use in a higher temperature range. The other problem with Fe₃Al is its low yield strength above 300 °C. The yield strength decreases from 700 MPa at room temperature to 200 MPa at 500 °C [5]. One promising way to improve the high temperature strength is by dispersing ceramic particles or fibres in the matrix. Because Al₂O₃ is chemically stable in most aluminides, it has the possibility of reinforcing intermetallic compounds as particles or fibres.

In the present work Fe₃Al matrix composites reinforced with fine Al₂O₃ particles were fabricated by powder metallurgy and their microstructure and mechanical and several other properties were examined.

2. Experimental procedures

2.1. Materials and sintering

Powders of Fe and Al were used as the starting materials for the matrix alloy. The chemical compositions and particle sizes are listed in Table 1. The powders were mixed in the precise atomic ratio of Fe:Al=3:1. Al_2O_3 powder was used as the reinforcement α phase Al_2O_3 , whose composition is also listed in Table 1.

Mixing of the powders was carried out by dry ball milling for 24 h in air. The powder mixture was put into a carbon mould for hot pressing. Sintering was carried out by hot pressing at 1200 °C for 30 min under a pressure of 20 MPa in a vacuum of about 5×10^{-4} Torr. Under these conditions all specimens were sintered to full density. The sintered body was about 50 mm in diameter and 4 mm thick.

2.2. Evaluation of mechanical properties

To evaluate the mechanical properties, tensile tests, Young modulus measurements and Vickers hardness tests were performed at room temperature. Tensile specimens had a square cross-section of 1.5 mm \times 1.5 mm and the gauge length was 10 mm. The strain rate was $8.3 \times 10^{-4} s^{-1}$. Tests were carried out at room temperature and 500 °C in air. The Young modulus was measured by the vibration resonance method using specimens in the form of bars of dimensions 3 mm \times 3 mm \times 40 mm. The Vickers hardness was measured on a polished surface.

2.3. Microstructure determination

Optical microscopy (OM) and scanning electron microscopy (SEM) were used for the microstructure determination. After being polished to optical flatness, specimens were slightly etched by dilute H_2SO_4 solution before observation. Several specimens were examined by energy-dispersive X-ray (EDX) analysis to confirm the phase compositions. Transmission electron microscopy (TEM) was also used to examine the interface between the matrix and the Al_2O_3 particles. Thin specimens for TEM were prepared by electrolytic polishing followed by argon ion thinning. The microscope used

TABLE 1. Chemical composition of materials and particle sizes

Substance	Chemical compositions (wt.%)	Particle size (μm)
Fe	Fe > 99.5, Ni < 0.04, Mo < 0.01, C < 0.002, N < 0.05, O < 0.15	8
Al	Al: 99.41, Fe: 0.42, Cu: 0.02, Si: 0.12, Mn: 0.01, Ti: 0.01	< 44
Al_2O_3	Si: 0.027, Na: 0.003, Mg: 0.002, Cu: 0.002, Fe: 0.014, balance: Al_2O_3	0.6

was a Jeol JEM200CX operating at 200 kV. The phase constitution was also determined by X-ray diffraction.

2.4. Thermal expansion and oxidation resistance

The thermal expansion was measured up to 1000 °C at an increasing temperature rate of 5 °C min^{-1} . The oxidation rate was evaluated for the same specimens as those used for the Young modulus measurements. The bars were subjected to an oxidizing atmosphere at 500 and 1000 °C in air and the weight gains per unit area were measured at fixed intervals.

3. Results and discussion

3.1. Microstructure of Fe_3Al alloy and composites

The present fabrication process is a reactive sintering method in which the starting powder constituents react to form a compound during sintering [9]. The phase constitution after sintering was determined by X-ray diffraction. The profile from the sintered Fe_3Al alloy without any Al_2O_3 particles is shown in Fig. 1. It was found that the DO_3 phase was dominant in the monolithic alloy. In the case of the composites the DO_3 - Fe_3Al phase was also dominant under the present experiment conditions.

Figure 2 shows the density of the composites as a function of the volume fraction of Al_2O_3 particles. The density decreased linearly with increasing volume fraction and every measured value reached almost 100% of the pore-free calculated density. Figure 3 shows OM photographs of the typical grain structures of the monolithic Fe_3Al alloy and its composites. With increasing volume fraction of Al_2O_3 , the grain size of the matrix decreased. Figure 4 summarizes the change in grain size as a function of the Al_2O_3 volume fraction. With

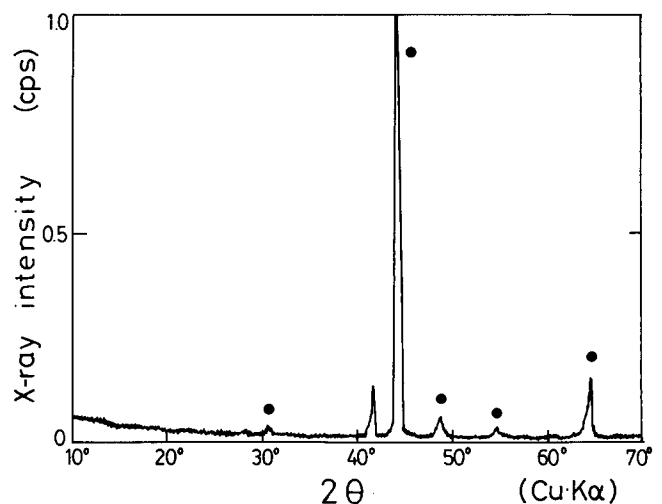


Fig. 1. X-ray diffraction profile of Fe_3Al . Solid circles show the peaks of DO_3 - Fe_3Al .

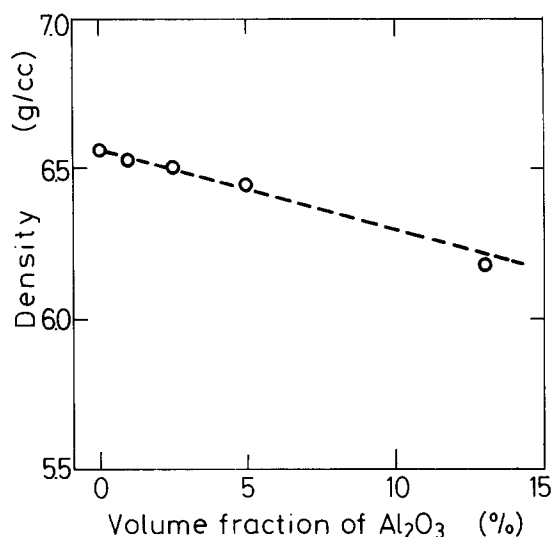


Fig. 2. Density of Fe_3Al matrix composites as a function of Al_2O_3 volume fraction.

increasing volume fraction of Al_2O_3 , the grain size decreased monotonously. Thus the Al_2O_3 particle addition to the alloy has the effect of grain refinement by preventing grain growth during sintering.

Figure 5 shows the typical dispersion structure of Al_2O_3 particles in the composites. The fine black particles are Al_2O_3 particles. The Al_2O_3 particles dispersed both within grains and along grain boundaries. At a volume fraction of 13% Al_2O_3 the aggregation of Al_2O_3 particles along grain boundaries became apparent.

Figure 6 shows a TEM photograph of an Al_2O_3 particle on a grain boundary. A dislocation structure was observed near the particle. No reaction between the Al_2O_3 and the matrix was evident. Figure 7 shows a high resolution transmission electron microscopy (HRTEM) photograph of the interface between the Al_2O_3 and the matrix. Both lattices are directly connected at the interface. Thus Al_2O_3 proved to be quite stable in the Fe_3Al matrix.

3.2. Influence of Al_2O_3 addition on mechanical properties

Figure 8 shows the influence of Al_2O_3 addition on the Vickers hardness. The Vickers hardness increased with increasing volume fraction of Al_2O_3 , though beyond 2.5 vol.% Al_2O_3 the increase was small. The increase in Young modulus is shown in Fig. 9 and is seen to be almost linear with increasing volume fraction of Al_2O_3 .

Figure 10 shows the tensile properties of the composites as a function of Al_2O_3 addition. The tensile strength did not change greatly with the addition of Al_2O_3 particles. The tensile strength of the monolithic Fe_3Al is quite high, about 800 MPa. Previous reports also revealed this high strength of Fe_3Al [5, 6]. The

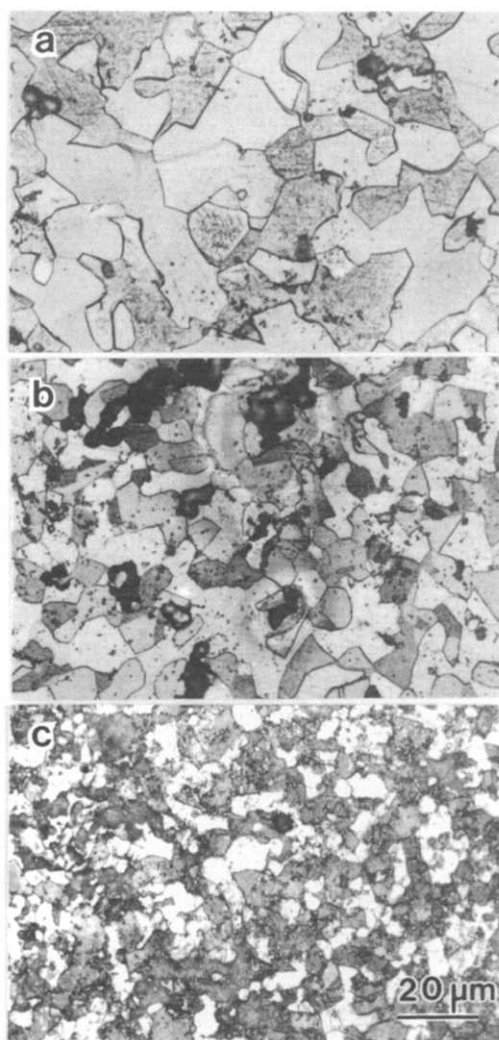


Fig. 3. OM photographs of Fe_3Al and its composites: (a) Fe_3Al , (b) Fe_3Al with 2.5 vol.% Al_2O_3 , (c) Fe_3Al with 13 vol.% Al_2O_3 .

highest strength reported was 800–900 MPa for an alloy fabricated by the powder metallurgical process [5]. The present alloy and its composites have the same strength level. This high strength is believed to be good enough for most structural applications. In addition, the alloy shows excellent elongation, reaching 5% at room temperature.

Although the tensile strength only showed a slight increase, the 0.2% proof stress increased by more than 100 MPa with the addition of 2.5 vol.% Al_2O_3 . Beyond 2.5 vol.%, however, the volume fraction of Al_2O_3 did not have great influence on the 0.2% proof stress. The elongation decreased monotonously with the addition of Al_2O_3 . At 2.5 vol.% Al_2O_3 it was about 2%. This decrease in elongation or reduction in ductility is a critical disadvantage of composite materials. Thus the addition of Al_2O_3 particles increased the strength but reduced the elongation. The optimum concentration of Al_2O_3 particles is about 2.5 vol.%, at which volume

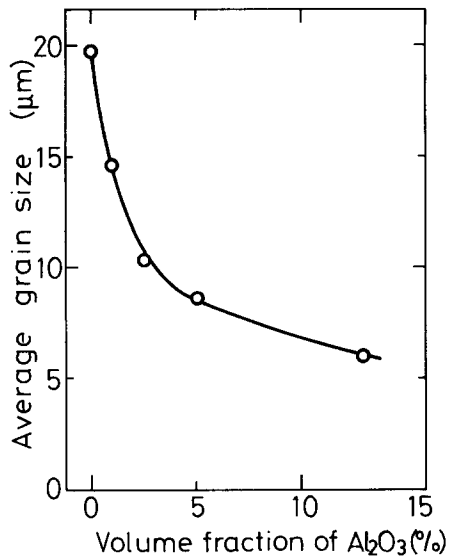


Fig. 4. Average grain size of Fe_3Al matrix composites as a function of Al_2O_3 volume fraction.

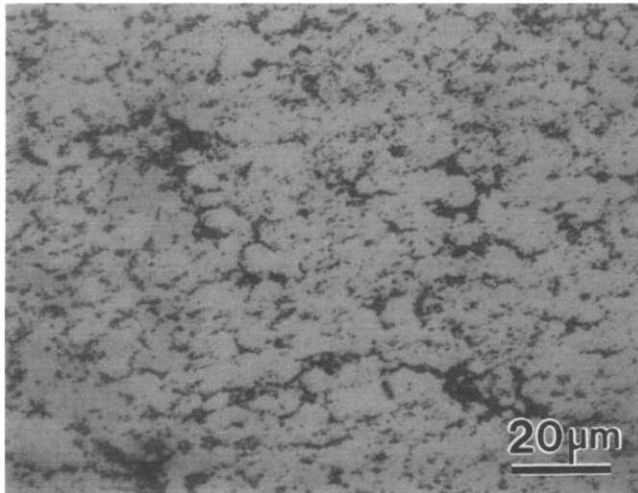


Fig. 5. Typical microstructure showing dispersion of Al_2O_3 particles in Fe_3Al composite with 13 vol.% Al_2O_3 .

percentage the 0.2% proof strength increases considerably and the decrease in elongation is limited to within a reasonable value.

Figure 11 shows typical fracture surfaces of the Fe_3Al alloy and the composite with 2.5 vol.% Al_2O_3 . The Fe_3Al alloy has a semi brittle fracture mode in which transgranular fracture was primarily observed but interface cracking was sometimes evident. The composite has a similar fracture surface to the monolithic alloy. Interface debonding between the particles and the matrix was frequently observed.

3.3. High temperature properties

The maximum temperature of use of Fe_3Al seems to be 500 °C because of the transformation at around

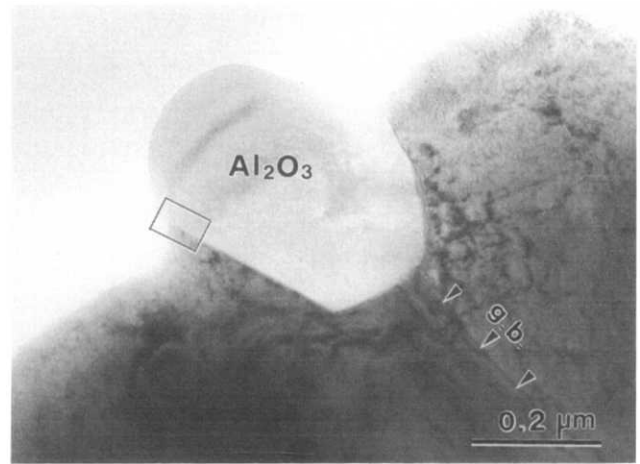


Fig. 6. TEM photograph of an Al_2O_3 particle on a grain boundary.

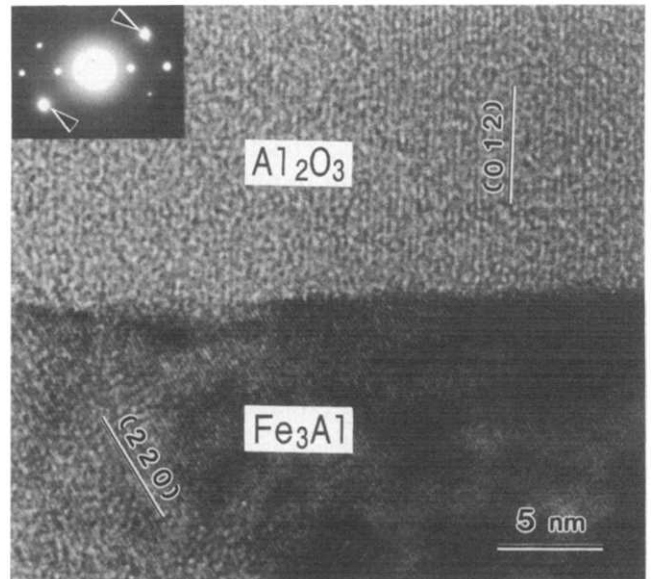


Fig. 7. HRTEM photograph of interface between Al_2O_3 and Fe_3Al matrix. The arrows on the electron diffraction pattern (inset) show the reflections from the (220) plane of Fe_3Al .

550 °C. Thus the strength at 500 °C was measured in air for two of the composites. Table 2 summarizes the 0.2% proof strength and tensile strength. The 0.2% proof strength of the monolithic Fe_3Al alloy decreased from 700 MPa at room temperature to 440 MPa at 500 °C. In contrast, the two composites with Al_2O_3 reinforcement maintained good strength. The 0.2% proof strength was higher than 620 MPa at 500 °C. Thus Al_2O_3 particle reinforcement proved to be effective in maintaining the strength at a high level also at elevated temperatures.

Figure 12 shows the average thermal expansion coefficient from room temperature to 1000 °C as a function of the volume fraction of Al_2O_3 . The thermal expansion

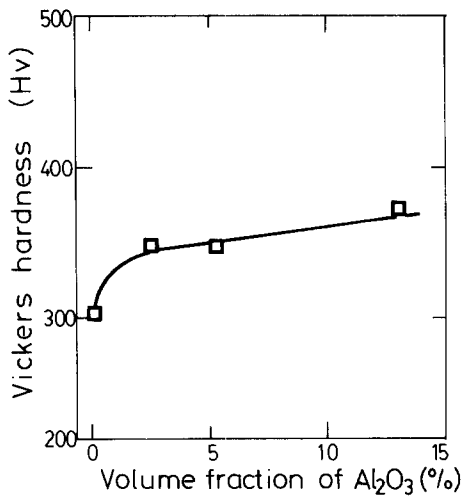


Fig. 8. Vickers hardness as a function of Al₂O₃ volume fraction.

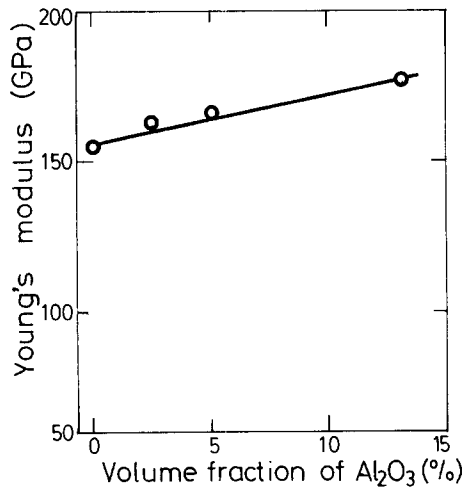


Fig. 9. Young modulus as a function of Al₂O₃ volume fraction.

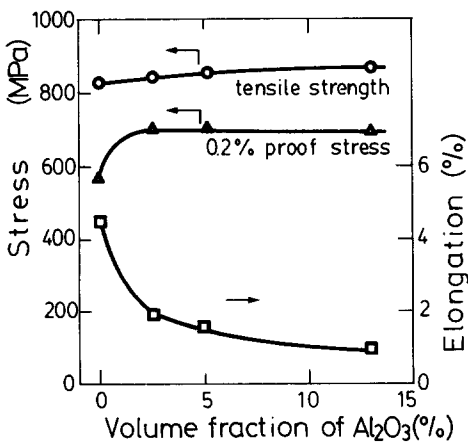


Fig. 10. Tensile strength, 0.2% proof stress and elongation at room temperature as functions of Al₂O₃ volume fraction.

coefficient decreased monotonously with increasing volume fraction of Al₂O₃, though the changes were not very large.

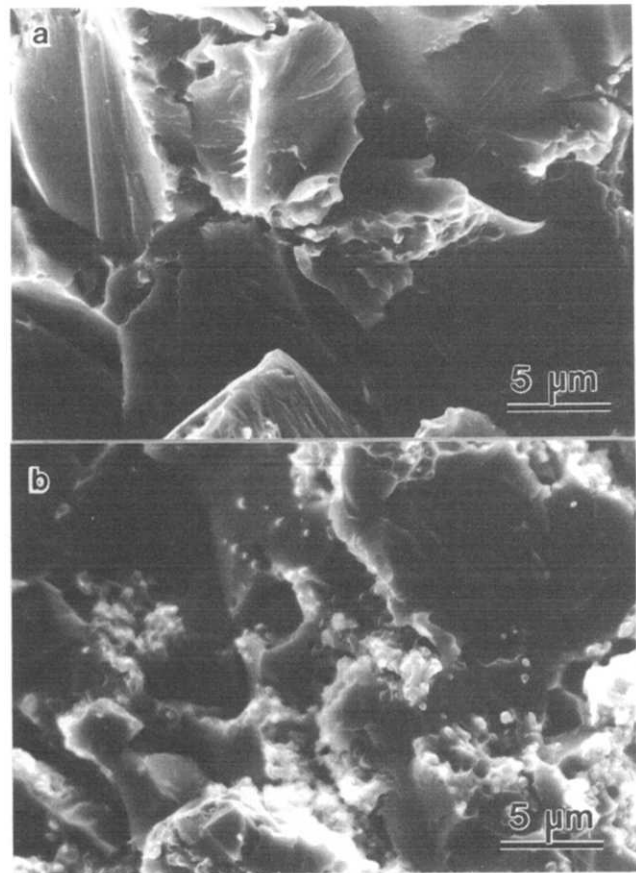


Fig. 11. Fracture surfaces of (a) Fe₃Al and (b) Fe₃Al with 2.5 vol.% Al₂O₃ at room temperature.

TABLE 2. Values of 0.2% proof stress, tensile strength and elongation of composites at 500 °C

Material	0.2% proof stress (MPa)	Tensile strength (MPa)	Elongation (%)
Fe ₃ Al	441	594	16.3
Fe ₃ Al-2.5 vol.% Al ₂ O ₃	619	664	12.2
Fe ₃ Al-5 vol.% Al ₂ O ₃	636	689	10.0

At 500 °C no significant increase in weight gain due to oxidation was evident. Figure 13 shows the weight gain at 1000 °C. For comparison, the figure also shows the weight gain of Inconel 713 alloy at 1000 °C, which is a representative high temperature nickel-based superalloy. All the composites increased their weight up to 1 h after heating but did not during further oxidation. The Inconel 713 alloy increased in weight monotonously. The oxidation of the Inconel alloy obeys the diffusion process of cations and anions in the protective oxide film such as Cr₂O₃ formed on the surface. In contrast, Fe₃Al seems to form Al₂O₃ on the surface as the oxide film. The diffusion of Al and O atoms in Al₂O₃ is much slower than that of Cr and O atoms in Cr₂O₃. The diffusion constants of the former two are 10⁻¹⁷ cm²

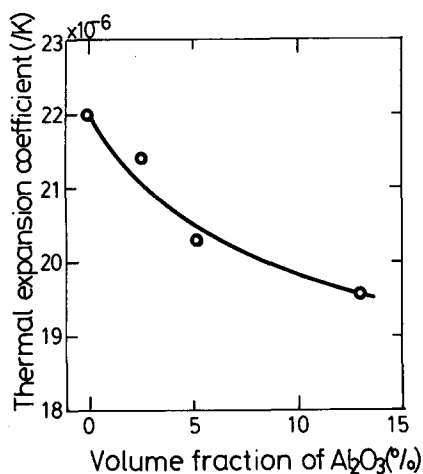


Fig. 12. Thermal expansion coefficient between room temperature and 1000 °C as a function of Al_2O_3 volume fraction.

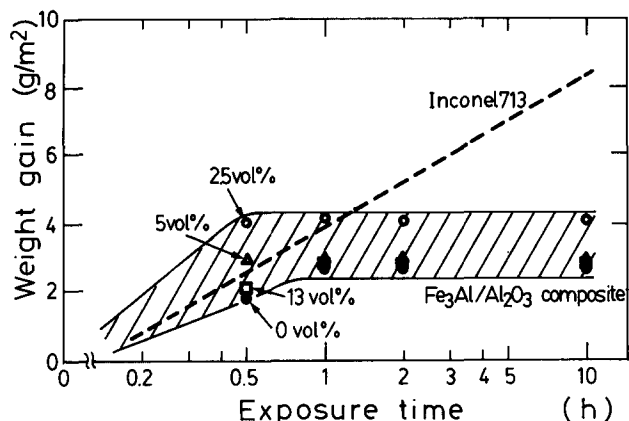


Fig. 13. Weight gain of Fe_3Al and its composites as a function of exposure time at 1000 °C. The dashed line represents Inconel 713 alloy.

s^{-1} at 1000 °C while those of the latter two are 2×10^{-15} and $10^{-16} \text{ cm}^2 \text{ s}^{-1}$ respectively [10]. Therefore once a dense Al_2O_3 layer has formed on the surface, it will act as a good protective layer. This is the reason why Fe_3Al and its composites possess good oxidation resistance.

4. Conclusions

The present work has examined several properties of Fe_3Al matrix composites reinforced with fine Al_2O_3 particle dispersions. The Fe_3Al alloy and its composites were fabricated by the reactive sintering process from powder mixtures. Several important results were obtained as follows.

(1) At the Fe_3Al - Al_2O_3 interface no third phase was observed. Both phases were connected directly on an atomic scale.

(2) The grain size decreased monotonously with increasing volume fraction of Al_2O_3 particles added.

(3) The hardness and Young modulus increased slightly with the addition of Al_2O_3 .

(4) The 0.2% proof stress and tensile strength were improved by the addition of Al_2O_3 particles. At 2.5 vol.% Al_2O_3 addition the 0.2% proof stress increased by 100 MPa at room temperature, but above this volume fraction little further improvement was obtained. The increase in tensile strength was less than that in 0.2% proof stress.

(5) The elongation decreased monotonously with increasing Al_2O_3 volume fraction. The elongation of Fe_3Al with 2.5 vol.% Al_2O_3 was 2% while that of Fe_3Al was 5% at room temperature.

(6) At 500 °C the composites had higher strength, by about 100 MPa, than the monolithic alloy.

Thus the best improvement obtained with the dispersion of Al_2O_3 particles was the increase in strength at room temperature to elevated temperature. However, accompanying the increment in strength, the elongation decreased, which is always the case in composite materials. Therefore the optimum choice for the Fe_3Al matrix composite will be in the range 2.5–5 vol.% Al_2O_3 particle addition.

Acknowledgments

The author thanks Mr. K. Matsunaga for his help with the experiments. The work was partly supported by the Research and Development Fund of the AMADA Foundation in 1991.

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