The Effect of in-situ Formation of Al₂O₃ or Fe₃AlC on the Structural and Mechanical Properties of FeAl Intermetallic Alloys

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The effect of in-situ formation of reinforcing phase on the mechanical properties of FeAl intermetallic alloy was studied. The in-situ FeAl composites containing Al₂O₃ or Fe₃AlC were fabricated by mechanical alloying of elemental powders and mill scale powder followed by vacuum hot pressing. The starting materials were mixed in the appropriate ratio to synthesize 10 vol% of the reinforcing phase. FeAl + Al₂O₃ and FeAl + Fe₃AlC alloys showed high hardness value of HV813 and HV524, respectively. The effect of testing temperature on the 0.2% proof stress was evaluated at the temperature range from room to 1 273 K under compression. FeAl + Al₂O₃ and FeAl + Fe₃AlC alloy showed proof stress of 2 240 MPa and 1 380 MPa at room temperature, respectively. At higher temperatures above 773 K the proof stress decreased rapidly. The FeAl base in-situ composites including Fe₃AlC or Al₂O₃ showed the high stress exponent and activation energy in the temperatures range of 1 073 – 1 273 K.

Key Words: Composite Material, Sintered Materials, Material Testing, FeAl, in-situ Composite, Mechanical Alloying, Al₂O₃, Fe₃AlC, High Temperature Deformation

1. Introduction

The iron aluminide, monolithic FeAl, exhibits adequate oxidation and corrosion resistance, low density (≈ 6.0 Mg/m³), high strength up to about 800 K, and it can be prepared at small material cost. Therefore, it is often said that the FeAl intermetallic compound is particularly attractive for high temperature structural applications at intermediate temperature (up to 800 K) and a potential candidate to substitute stainless steels. However, its application has been limited because of the poor ductility and toughness at room temperature and the poor creep strength at high temperature. By improving high temperature strength, FeAl alloy might be able to be utilized at the temperature range over 800 K like Fe-base superalloy.

Dispersion strengthening may provide the better combination of high temperature strength and ductility at room temperature. Among the fabrication processes of composites, in-situ formation of matrix and reinforcement can create some advantages in comparison with the conventional process (by mixing alloy powders and hard particle, so called ex-situ composite), such as smaller grain size matrix and reinforcement, higher interface strength, better particle size distribution and so on.

Mechanical alloying (MA) is a useful process to produce high performance alloys, for example, amorphous, supersaturated solid solution and intermetallic compound with fine microstructure. However, it is important to add a small amount of hydrocarbons, alcohols or stearic acid, as a process control agent to prevent excessive adhesion of constituent elements to the vessel wall and the ball. Therefore, the process control agent often forms undesirable carbide during MA and following consolidation processes.

We have been investigating in-situ FeAl base oxide dispersion strengthened (ODS) alloy prepared by MA and vacuum hot pressing (VHP) process. This powder metallurgical technique can synthesize the matrix and the reinforcement (Al₂O₃) by a mechanochemical reaction, which is the reduction of Fe-oxide by Al. However, Fe₃AlC
phase was formed during fabrication process in addition to the Al2O3, because of the presence of the process control agent, stearic acid. The purpose of this investigation is to identify the in-situ formation of Al2O3 and Fe3AlC in the consolidated compacts and to evaluate the mechanical properties from room to elevated temperature. Therefore, we choose the composition of the compact to synthesize 10 vol% reinforcements in order to make the effect clear.

2. Experimental Procedure

The milled powder to fabricate FeAl + Al2O3 in-situ composite was prepared from Fe powder (particle size < 150 μm, 99.9% purity), Al powder (< 150 μm, 99.98%) and mill scale powder (< 10 μm, after clashing). And Fe, Al and C (< 20 μm, 99.9%) powders were used to produce FeAl + Fe3AlC in-situ composite.

The mill scale powder consists of FeO, Fe2O3 and Fe3O4, and the chemical composition is shown in Table 1. The mixing ratio of starting powders was calculated by assuming the Fe-40 mol%Al composition matrix with 10 vol% Al2O3 reinforcement, which are obtained after the ideal mechanochemical reaction, Fe + Al + Fe-oxide → FeAl + Al2O3. Therefore, the starting composition is Fe-26.4 mol%Al-3.3 mol%O for FeAl + 10 vol%Al2O3 composite.

Since the accurate chemical composition of Fe3AlC phase was not fixed, a preliminary experiment was carried out to determine the appropriate carbon content of FeAl + 10 vol%Fe3AlC. The relationship between C content and volume fraction of Fe3AlC phase in the MA powder compact was determined.

Consolidation was carried out by vacuum hot pressing (VHP) at 1 273 K and 150 MPa for 3.6 ks. Cylindrical TZM alloy (Mo base heat resisting alloy) die having 12 mm inner diameter was used. The VHP pressure was applied first before heating, and kept constant during following heating and holding stage.

Microstructural investigations were performed on the milled powders and compacts using a scanning electron microscopy (SEM), energy dispersion X-ray spectrometry (EDS) and X-ray diffraction (XRD). The cross sections of the compacts were etched with an etchant consisting of 33%H2O + 33%HNO3 + 33%CH3COOH + 1%HF.

The effect of reinforcement (Al2O3 or Fe3AlC) formed by mechanochemical reaction in the compacts was evaluated by hardness test and compression test. The Vickers hardness tests were carried out at room temperature under a load of 300 gf for 15 sec. Results of hardness tests were the average of seven measurements.

Elevated temperature tests were carried out by constant crosshead speed compression testing under Ar atmosphere on the Shimadzu Autograph AG-IS testing machine equipped with an infrared radiation furnace. Only the room temperature testing was conducted in air. The dimension of the compression test specimen is rectangular with dimensions of 4 mm×4 mm×6 mm. Temperature dependence of 0.2% proof stress of each compact was tested using one specimen. The specimens were heated up at the rate of 0.33 Ks⁻¹ (20 Kmin⁻¹) and held for 10 min for homogenization at each testing temperature before loading. The temperatures employed for testing were in the range of 473 – 1 273 K at intervals of 100 K.

The dependence of strain rate on the flow stress was examined by strain rate change test at initial strain rate ranging from about 3×10⁻⁶ to 1×10⁻² s⁻¹. True compression stress, strain and strain rate were calculated from the recorded load-displacement data. The stress exponent (n value) and activation energy of high temperature deformation were discussed based on the power law equations:

\[ \dot{\varepsilon} = A\sigma^n \exp(-Q/RT) \]

where A is constants, n the stress exponent, Q the activation energy, R the gas constant and T the testing temperature.

And the equation based on the concept of threshold stress (σ₀) is

\[ \dot{\varepsilon} = A'(\sigma - \sigma_0)^n \exp(-Q/RT) \]

where A' is constant. Eqution (2) is used to examine the threshold stress of the in-situ composite.

3. Results and Discussion

3.1 Fabrication of in-situ composites

In order to study the effect of in-situ formation of reinforcements (oxide or carbide), MA process was chosen to prepare the alloyed powders and synthesize matrix and reinforcement. MA is the solid state in-situ process, so that it is beneficial method to produce high performance material having uniform and fine microstructure.

Fully milled powders, milled for 1 800 ks, were used in this investigation. Figure 1 shows the SEM micrographs of 1 800 ks milled powders showing difference in morphology, which relate to the chemical composition of the mixture of starting materials. The MA powder

Table 1 Chemical composition of mill scale powder (mass%)

| Element | Fe | C | Si | Mn | P | S | O |
|---------|----|---|----|----|---|---|---|
| Composition | bal. | 0.06 | 0.06 | 0.52 | 0.007 | 0.004 | 24.5 |
Fig. 1 SEM micrographs of 1800 ks milled powders. (a) FeAl+Al₂O₃ composition, (b) FeAl+Fe₃AlC composition

Fig. 2 X-ray diffraction patterns of the compacts. (a) FeAl+Al₂O₃ composition, (b) FeAl+Fe₃AlC composition

having FeAl+Al₂O₃ composition consists of agglomerated particles in Fig. 1 (a), the size of which is less than 1 µm. The FeAl+Fe₃AlC composition powder shows larger size about 5 µm in Fig. 1 (b). The XRD, thermal analysis (DTA) and Mössbauer spectroscopic study indicate that the FeAl phase can be synthesized from elemental powders only by MA⁴,⁷. Figure 2 shows the XRD profiles of the compacts. According to the XRD analyses of milled powders and compacts as shown in Fig. 2, matrix FeAl phase can be synthesized mostly by MA process, and the reinforcement, Al₂O₃ or Fe₃AlC, are synthesized during consolidation process up to 1273 K. After 1800 ks milling, the volume fractions of FeAl phase of both MA powders were about 70%, which were obtained by the rough estimation using the height ratio of the major peak of α-Fe and FeAl. Both 1800 ks milled powders show small crystallite size about 20 nm calculated from XRD line broadening, regardless of the composition of starting material⁴.

Figure 3 (a) shows SEM backscattered electron image (BEI) on the cross section of FeAl+Al₂O₃ compact (as-polished condition) and (b) shows SEM micrograph of Fe+Fe₃AlC compact (etched). Microstructure of FeAl+Al₂O₃ compact is divided into two areas, network like bright region and dark region with small darker spots. The contrast difference in the BEI corresponds to the fraction of Fe and Al elements. From the mixing ratio of starting materials and XRD results as shown in Fig. 2, the small spots in the dark contrast region in Fig. 3 (a) seem to be fine Al₂O₃ precipitations. Grain size of the synthesized Al₂O₃ precipitation is about 20 nm obtained from TEM observation. Therefore, the FeAl+Al₂O₃ compact consists of Al₂O₃ concentrated area and Al₂O₃ free area. The very fine pores, indicated by white arrows in the figure, are observed only in the FeAl+Al₂O₃ compact. The relative density measured by area analysis of the cross section of the compact was about 99.9% (5.420 Mgm⁻³ by Archimedes method) of the theoretical. FeAl+Fe₃AlC compact showed high density, almost 100% (5.935 Mgm⁻³). The ratio of Fe and Al content of the precipitation was 70 to 30 by SEM-EDS analyses. In addition to the XRD result as shown in Fig. 2, the precipitation in Fig. 3 (b) seems to be the Fe₃AlC phase. The average particle size of Fe₃AlC precipitation is 0.55 µm.
and the volume fraction is about 8.9%, which is close to the expected value of the reinforcement, 10 vol%.

Even after the VHP runs at 1 273 K for 3.6 ks, these two composites reveal still fine matrix, 50 nm obtained from XRD line broadening.

FeAl + Al2O3 and FeAl + Fe3AlC in-situ composite were obtained through MA and VHP process, and both composites have high density and consist of very fine microstructure.

### 3.2 Mechanical properties of in-situ composites

#### 3.2.1 Hardness test

Table 2 summarizes the results of Vickers hardness tests of the in-situ composites. The diagonals of indentations were about 33 µm for FeAl + Fe3AlC composite and about 26 µm for FeAl + Al2O3 composite. These sizes were larger than about 0.4 µm in the diameter of each MA powder and interparticle separation of Fe3AlC precipitation. Therefore, hardness obtained here seems to represent the property of the compact itself. Additionally, the hardness of FeAl (Fe-40 mol%Al composition monolithic compact, coarse grain) and Fe3AlC phase are shown in the table. The composites fabricated in this investigation show higher hardness values of HV524 and HV813 than HV440 of the FeAl monolithic compact. In the case of FeAl + Fe3AlC composite, HV524 is much higher than the estimated value calculated by the rule of mixture of FeAl and 8.9 vol%Fe3AlC. Hardness of FeAl intermetallic compound increases with increasing Al content\(^{(1)}\). Additionally, it is strongly affected by the presence of thermal vacancy whose concentration increases by increasing cooling rate after heat treatment. For example, Fe-40 mol%Al material cooled by water from 1 273 K showed about HV400, which is higher than HV180\(^{(1)}\) of the material annealed under 673 K-118 ks to remove excess thermal vacancy. Although all of the in-situ composites, obtained here, were furnace cooled after VHP runs, hardness value HV440 of FeAl monolithic compact shows almost same value as the material quenched by water. Therefore, higher hardness values of the in-situ composites seem to be the effect of relatively fine microstructure as well as Hall-Petch effect due to synthesized hard particles.

#### 3.2.2 Compression tests

The effect of reinforcements on the mechanical properties, especially high temperature properties, was evaluated under compressive load at the temperature from room temperature to 1 273 K. The variation of 0.2% proof stress with testing temperature was determined at an initial strain rate of about \(2.8 \times 10^{-4} \text{s}^{-1}\) as shown in Fig. 4. In addition, the results taken from literature references\(^{(8),(9)}\) are also shown in the figure. Our samples were tested under as-VHP condition, of which cooling rate is estimated between the furnace cooling and air cooling from 1 273 K as mentioned in the previous section. FeAl + Al2O3 and FeAl + Fe3AlC composite show very high proof stress at lower temperature range with 2 240 MPa and 1 380 MPa, respectively. And the proof stress remains almost constant up to 700 K. Those are very high values in comparison with the literature data of FeAl monolithic large grained alloy\(^{(9)}\) (under compression) and FeAl-1 mass%Y2O3 alloy\(^{(8)}\) as shown in Fig. 4. Furthermore, the precipitated reinforcement, Al2O3 or Fe3AlC, is effective to increase the proof stress at all testing temperature range. The proof stress of all of the FeAl alloy depicted in Fig. 4, more precisely B2 type FeAl alloy\(^{(1)}\), begins to decrease rapidly when the temperature exceeds around 700 – 800 K, regardless of the existence of high strength reinforcements. Some models have been suggested to explain the relationship between the proof stress and the testing temperature of FeAl, for example cross-slip/pinning, anti-phase boundary mechanism, dislocation decomposition, vacancy hardening mechanism, and so on\(^{(1)}\). The precipitated reinforcement, Al2O3 or Fe3AlC, can increase the proof stress, however, it drops at the temperature almost same as FeAl monolithic compound. It appears that the temperature dependence of the matrix (FeAl) affects the proof stress of the composites. The FeAl + Al2O3 composite shows high strength in comparison with the FeAl + Fe3AlC. Furthermore, as the in-situ composites have fine matrix, it contributes to enhance the proof stress at lower temperature range due to the Hall-Petch relation.

The examples of the stress-strain curve of FeAl + Al2O3 composite at 473 K and FeAl + Fe3AlC composite at room temperature are shown in Fig. 5. FeAl intermetallic compound usually shows peak stress and plateau after yielding at lower testing temperature associated with dislocation locking mechanism\(^{(9)}\). The FeAl + Fe3AlC com-

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**Table 2** Results of hardness tests

| Material | FeAl | Fe3AlC | FeAl + 8.9vol%Fe3AlC | FeAl + Al2O3 |
|----------|------|--------|----------------------|-------------|
| HV       | 440  | 646    | 524                  | 813         |

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**Fig. 4** Variation of 0.2% proof stress with testing temperature for FeAl base alloys.
posite shows peak stress under compression test as well as the FeAl monolithic compound\(^{(9),(10)}\). FeAl + Al\(_2\)O\(_3\) composite showed brittle fracture just after yielding at lower temperature regime up to 573 K. On the other hand, when the testing temperature increased above 773 K, FeAl + Al\(_2\)O\(_3\) composite began to show compressive ductility. As described above, effect of the Al\(_2\)O\(_3\) reinforcement on the strength is more remarkable than Fe\(_3\)AlC. In addition, our previous data of FeAl + 10 vol%Al\(_2\)O\(_3\) + 7.6 vol%Fe\(_3\)AlC composite showed hardness of HV798\(^{(4)}\), which is close to HV813 of the FeAl + 10 vol%Al\(_2\)O\(_3\) composite obtained here. In this case, 7.6 vol%Fe\(_3\)AlC was not effective on the hardness of FeAl + 10 vol%Al\(_2\)O\(_3\) composites. Al\(_2\)O\(_3\) can make a higher contribution to the hardness of FeAl at room temperature than Fe\(_3\)AlC phase, as well as the high temperature strength already shown in Fig. 4.

The effect of the strain rate on the flow stress was examined at high temperature regime from 1073 to 1273 K where the steady state deformation occurred. The experiments were carried out over the strain rate range from \(3.4 \times 10^{-6}\) to \(1.3 \times 10^{-3}\) s\(^{-1}\) under compression by using strain rate change technique. The relationship between the true flow stress and the true strain rate can be fitted to the power law equation (1) under constant temperature condition. The apparent stress exponents (\(n\) value) were estimated by fitting the experimental data to Eq. (1) as shown in Fig. 6. In both composites, the flow stress increases gradually with increasing strain rate in a linear function within the experimental regime of this investigation. Although the in-situ composites obtained here have really fine microstructure about 50 nm of matrix diameter, the large stress exponents were obtained over the testing temperature range. The FeAl + Fe\(_3\)AlC composite shows almost same stress exponent about 7.6 regardless of testing temperature. The FeAl + Al\(_2\)O\(_3\) composite shows higher stress exponent more than 10 and it decreases with in-
creasing testing temperature. In addition, the stress exponent tends to decrease with decreasing strain rate only at 1273 K. The stress exponent becomes 4.2 at the strain rate lower than $3 \times 10^{-5}$ s$^{-1}$, namely the strain rate sensitivity exponent $m$ value defined as $m = 1/n$ increases from 0.01 to 0.24. Materials having fine and equiaxed microstructure like these in-situ composites, even intermetallic compound or ceramic, shows superplastic deformation quite frequently with indicating $m$ value larger than 0.3. Therefore, FeAl + Al$_2$O$_3$ composite might show the superplasticity, but it seems to be necessary higher temperature or lower strain rate to deform superplastically.

Next, we discuss the threshold stress of high temperature deformation according to the Eq. (2) by extrapolation of the testing data to lower strain rates. The threshold stress can be estimated from a linear extrapolation of the flow stress to zero strain rate at the data under constant testing temperature. The data of Fig. 6 is replotted in Fig. 7 as $\dot{\varepsilon}^{1/n}$ against the flow stress using the $n$ value of stress exponents obtained from the relationship between strain rate and flow stress as indicated in Fig. 6. Figure 7 shows linear relationships at all testing temperatures except for FeAl + Al$_2$O$_3$ composite at 1273 K. The stresses are approaching zero at zero strain rate as far as the stress exponents are assumed to be constant at the strain rate range smaller than $\dot{\varepsilon} = 10^{-6}$ s$^{-1}$. In this investigation, we could not find the threshold stresses of in-situ composites. If we can examine the relationship between strain rate and flow stress at lower strain rate, it is expected that the stress exponent would increase with decreasing strain rate and the threshold stress could be found.

Furthermore, we discuss the temperature dependence of flow stress by using Eq. (1) without considering threshold stress. Under the assumption that the deformation mechanism is identical for the temperature from 1073 K to 1273 K, $n$ values used for calculation of the apparent activation energy were determined to be the average value of 11.7 for FeAl + Al$_2$O$_3$ and 7.6 for FeAl-Fe$_3$AlC composite. Figure 8 (a) and (b) show Arrhenius plots of the flow stress against the reciprocal testing temperature, where straight lines can be drawn through the data points and the apparent activation energies are calculated from the slope using average $n$ values over the testing temperature range. The apparent activation energy was determined to be about 675 kJmol$^{-1}$ for FeAl + Al$_2$O$_3$ and 520 kJmol$^{-1}$ for FeAl+Fe$_3$AlC composite, respectively.

The stress exponent and activation energy of FeAl intermetallic compound were reviewed$^{(1)}$. All data presented below were calculated using experimental raw data without considering the existence of threshold stress, like this investigation. The powder metallurgical Fe-40 mol%Al showed apparent activation energy of about 450 kJmol$^{-1}$ under compressive stress at temperature range from 1100 K to 1400 K, as well as this investigation, and stress exponent is about $n = 6$ corresponding with the steady state deformation condition or $n = 3$ during work hardening condition$^{(13)}$. The large grained Fe-48 mol%Al showed $n \approx 5$ at 1073 K and $n \approx 3.5$ above 1123 K, and the apparent activation energy of 395 kJmol$^{-1}$ at temperature from 1073 K to 1123 K$^{(12)}$. These stress exponent and activation energy are somewhat smaller than those of our results. In this investigation, high stress exponent and activation energy are found even at high temperature range from 1073 K to

![Fig. 7](image-url)  
**Fig. 7** Relationship between $\dot{\varepsilon}^{1/n}$ and flow stress for estimation of threshold stress. (a) FeAl + Al$_2$O$_3$ composition, (b) FeAl + Fe$_3$AlC composition

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1 273 K in comparisons with the literature values described above. Furthermore, apparent activation energies described above are larger than the interdiffusion activation energy of FeAl (Fe-40 mol% Al) which has been obtained about 260 kJmol$^{-1}$ and 280 – 310 kJmol$^{-1}$. In comparison with the high temperature deformation characteristics of the FeAl intermetallic compound in the literature, it is thought that the synthesized fine hard particle of Al$_2$O$_3$ or Fe$_3$AlC increases the stress exponent and apparent activation energy. The stress exponent and activation energy of FeAl base composite were not reported so far.

The superplastic deformation of FeAl intermetallic compound has been reported in many investigations. Fe-40 mol% Al alloy fabricated by hot extrusion shows $m = 0.3$ and the apparent activation energy of 375 kJmol$^{-1}$ under compression test at the temperatures above 1 373 K and strain rate less than 0.1 s$^{-1}$. The large grained material having the composition of Fe-36.5 mol% Al showed superplastic deformation at 1 173 – 1 273 K and 10$^{-4}$ – 10$^{-2}$ s$^{-1}$, caused by grain refinement by dynamic recovery and recrystallization. And the apparent activation energy of superplastic deformation was showed to be 370 kJmol$^{-1}$. These results show same activation energy under superplastic deformation about 370 kJmol$^{-1}$, which value is somewhat higher than the interdiffusion activation energy of FeAl, but smaller than the case of non-superplastic deformation described above. In addition, it has been reported that the MMC showed superplastic deformation by the mechanism of internal stress superplasticity at higher strain rate over 10$^{-2}$ s$^{-1}$, which was faster than that of the superplastic material having the same composition as the MMC.

The matrix itself of the FeAl + Al$_2$O$_3$ and FeAl + Fe$_3$AlC in-situ composites is expected to show the superplastic deformation, because of their really fine microstructure. Therefore, higher temperature than 1 273 K or higher strain rate over 10$^{-3}$ s$^{-1}$ at the temperature below 1 273 K seems to be necessary for superplastic deformation.

As mentioned above, despite the fact that the in-situ composites obtained here have very fine microstructure, they do not show the superplastic deformation. As compared with experimental condition and structural characteristics of superplastic FeAl alloys, it seems that higher temperature and strain rate than that of this investigation is necessary to develop superplastic deformation in FeAl in-situ composites studied here.

4. Conclusions

Mechanical properties of in-situ FeAl composites prepared by MA and VHP process were studied. Reinforcements, such as Al$_2$O$_3$ or Fe$_3$AlC, are formed by the reduction of Fe oxide by Al or mechanochemical reaction between elemental powders. The crystallite size of matrix remains nano size, about 50 nm, after consolidation by VHP process. The reinforcement of Al$_2$O$_3$ is used for the size of about 20 nm, and Fe$_3$AlC is 0.55 µm. FeAl + Fe$_3$AlC and FeAl + Al$_2$O$_3$ in-situ composites show high hardness value of HV524 and HV813 at room temperature, respectively. Synthesized reinforcement can provide the improved proof stress at lower temperature range up to about 700 K. The proof stress begins to decrease rapidly when the testing temperature exceeds about 700 – 800 K, regardless of the existence of high strength reinforcement. In-situ composites show high stress exponent and activation energy, such as $n = 7.6$ and $Q = 520$ kJmol$^{-1}$ for
FeAl + Fe₃AlC alloy and \( n = 10 \to 14 \) and \( Q = 675 \text{kJmol}^{-1} \) for FeAl + Al₁₂O₃ composite, respectively.

The in-situ precipitation, especially Al₂O₃, can increase the strength at high temperature compared to the monolithic FeAl intermetallic compound. The high temperature strength of in-situ FeAl ODS alloy seems to be comparable with the commercial Fe base ODS alloy like MA754 or MA6000.

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