Microstructures and Mechanical Properties of AI-Fe Composites Produced by Plasma Synthesis Method

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Abstract

The present work was undertaken to highlight a novel in-situ process in which plasma spraying techniques plus electromagnetic stirring were used to produce $Al/Al_{13}Fe_4$ composites consisted of angular and needle-like $Al_{13}Fe_4$ intermetallic compounds of about 10-20µm in size. The microstructures of the fabricated materials depend mainly on the temperature of the melt and the plasma spraying conditions. The formation of intermetallic compounds in the matrix by the plasma synthesis method was examined. In addition, the mechanical properties of the $Al/Al_{13}Fe_4$ composites were examined.

1. Introduction

Particulate-reinforced aluminum based metal matrix composites (MMCs) have a high potential for advanced structural applications due to their higher stiffness, superior strength, good elevated temperature resistance and improved resistance to wear [1-2]. MMCs have been synthesized by a number of techniques such as powder metallurgy, mechanical alloying, liquid metal infiltration, squeeze casting, spray codeposition, stir-casting and in-situ fabrication method [3]. Among the many techniques, an in-situ fabrication method has attracted great attention due to their strong interfacial bonding between the matrix and reinforcements [4-6].

The plasma synthesis method (PSM) is a kind of in-situ formation methods of intermetallic compounds in the matrix and it involves incorporation of metallic particles into a molten metal using a plasma jet. In the process, the particles are heated and accelerated in the plasma arc, which helps the incorporation of the particles into the molten metal and further accelerates the in-situ formation of intermetallic compounds in the melt [6].

In the present work, in-situ $AI/AI_{13}Fe_4$ composites were produced by the PSM and the microstructures and mechanical properties of the fabricated composites were investigated.

2. Experimental

The fabrication processing was carried out by a ValuPlazTM Plasma Spray System. Details of the experimental equipment were given elsewhere [7]. Aluminum ingots of 1000g placed in a clay-graphite crucible with argon flux cover were heated by a high frequency induction furnace. The crucible with the molten aluminum was taken out from the chamber in the high frequency induction furnace, and was put into the chamber under the plasma spraying nozzle. As the spray processing began, the electromagnetic stirring equipment was switched on to distribute particles homogeneously in the molten aluminum. After the injection of iron particles reached a required value, the melt was poured into a steel mould with 85mm in diameter. The main processing variables such as plasma spraying conditions and the temperature of the melt are summarized in Table 1. The as-cast Al/Al₁₃Fe₄ composites were swaged at room temperature with a 4:1 swaging ratio.

Standard metallographic techniques were employed to examine the microstructures of the samples. The microstructures were characterized using an optical microscope, a scanning electron microscope (JSM 5800) equipped with an energy dispersive X-ray spectrometer (EDS). The identification of phases was carried out using a Rigaku X-ray diffractometer with Cu K α radiation at 40kV and 30mA. The mechanical properties of the samples were evaluated by means of the compression tests.

	Processing variables				Volume fraction of Al ₁₃ Fe ₄ (%)		
Specimen	Input current (A)	Gas Flow rate (I/min)	Powder Feeding rate (g/min)	Temp. of melt (°C)	Total	Angular	Needle
P13	300	65	40	840	13.7	10.5	3.2
P14	300	65	60	820	14.3	11.9	2.4
P16	300	80	40	830	16.0	13.2	2.8

Table 1: Processing variables for test materials and their resultant volume fraction of Al₁₃Fe₄.

3. Results and Discussions

3.1 Microstructures

Figure 1 show the microstructures of as-cast and as-swaged Al/Fe composites (P16). The as-cast microstructure consists of angular and needle-like iron aluminide intermetallic compounds embedded in the α -Al matrix. From the X-ray diffraction patterns, iron aluminide is identified as Al₁₃Fe₄ and no other phases such as AlFe, Al₂Fe, Al₆Fe and AlFe₃ are identified. It is confirmed that the angular Al₁₃Fe₄ originates from the reaction of the Al melt and Fe particles, and needle-like Al₁₃Fe₄ originates from the crystallization of Al-Fe following the dissolution of Fe particle into the Al melt [7]. Furthermore, small amount of Al₅Fe₂ is detected in the X-ray diffraction pattern. This phase is observed only in the interfacial layer of the partially reacted Fe.

The size distribution of $AI_{13}Fe_4$ intermetallic compounds was measured using the image analyzer for as-cast samples and it was confirmed that more than 90% of $AI_{13}Fe_4$ is in the range from 5 to 15μ m. The $AI_{13}Fe_4$ became finer and the distribution of particles became uniform when the specimen is cold swaged. This is due to the occurrence of particle fracture during cold deformation [8]. The same microstructural features were obtained for other samples except for total volume fraction of $AI_{13}Fe_4$ and the ratio of volume fraction for angular and needle-like morphologies, indicated in Table 1.



Figure 1: Optical micrographs of (a) as-cast and (b) as-swaged specimen of Al/ $Al_{13}Fe_4$ (P16). 3.2 Formation of Intermetallic Compounds

The analysis of the partially reacted particles can give good insights for the formation of intermetallic compounds in the matrix by the PSM. Figure 2 shows an optical micrograph of the area around a partially reacted Fe particle. The intermetallic compound formed around the partially reacted Fe particle is composed of two morphologies: one is a tongue-like morphology and this phase grows inward, and the other one is agglomeration of small compounds and this phase grows outward. On the basis of the SEM/EDS analysis, the reaction zone could be divided into five regions; fully reacted and detached intermetallic compounds (A), fully reacted and attached intermetallic compounds (B), partially reacted intermetallic compounds (C), the Fe particle (Fe) and the matrix (M). The chemical compositions are different for each region (Table 2). The central region is confirmed as pure Fe and the inner region of the partially reacted intermetallic compounds is confirmed as Al₅Fe₂. The fully reacted intermetallic compounds (A and B) contain less Fe composition compared with the partially reacted intermetallic compounds (C) and the range of composition well corresponds to $AI_{13}Fe_4$. Thus, it can be said that the intermetallic compounds formed in the matrix is Al₁₃Fe₄ and the partially reacted particle is composed of Al/Al₁₃Fe₄/Al₅Fe₂/Fe layer.

It is reported that the effective free energy of formation concept well predicts the first and the subsequent phase between the solid/liquid interface [9-10]. The free energy of formation value at 1073K was replotted as a function of concentration for AI-Fe system in Figure 4. In an AI-Fe system, AI₁₃Fe₄ gives the most negative effective free energy of formation value at the composition of lowest liquidus. Thus, it can be predicted that Al₁₃Fe₄ would be the first phase that forms in the layer between liquid Al and solid Fe. Furthermore, Al₅Fe₂ gives the lowest effective free energy of formation between the Al₁₃Fe₄ phase and Fe. Thus, Al₅Fe₂ can be formed between the reaction of Al₁₃Fe₄ and Fe. Based on the above explanations, the sequence of the formation of intermetallic compounds in AI-Fe system by the PSM is as follows; i) iron dissolves into the aluminum melt when the iron concentration reaches to its maximum solubility at the processing temperature. The dissolved iron crystallizes to form needle-like Al₁₃Fe₄ during solidification as verified in [7], ii) the Al₁₃Fe₄ phase forms first in the layer of liquid Al/solid Fe, iii) the Al₁₃Fe₄ phase decomposes to form Al₅Fe₂ in the layer of Al₁₃Fe₄/Fe, and Al₅Fe₂ grows toward the Fe particle due to higher growth velocity of Al₅Fe₂ than that of Al₁₃Fe₄, as verified in [11], iv) the Al₁₃Fe₄ phase separates from the layer of Al₁₃Fe₄/Al₅Fe₂ owing to heat extraction caused by the reaction and MHD stirring force, v) new Al₁₃Fe₄ forms in the layer of fresh Al/Al₅Fe₂, and vi) the steps of iv) and v) repeat to react the Fe particle completely to form $AI_{13}Fe_4$ in the matrix.



Figure 2: Optical micrographs showing the partial-reacted particle region composed of tongue-like morphology (C) and agglomeration of small compounds (B).

Table 2: Results of EDS analysis of the partially reacted particle in Figure 2.							
Position	Composition	Bomarka					
	Experimental Nominal range		Itenidiks				
Fe	99.9	-	Fe				
А	35.9-39.3	38.7-41.5	Al ₁₃ Fe ₄				
В	37.4-37.8	38.7-41.5	Al ₁₃ Fe ₄				
С	45.3-45.8	43.6-47.0	Al ₅ Fe ₂				
М	0.3-0.4	-	AI				



Figure 3: Effective free energy of formation diagram for AI-Fe at 1073K.

3.3 Mechanical Properties

The yield strength determined for $Al/Al_{13}Fe_4$ composites through the compression test is plotted in Figure 4. It is demonstrated that the formation of $Al_{13}Fe_4$ increases the yield strength from about 60MPa to more than 150MPa.

The strengthening mechanism which may operate in particle reinforced MMCs has been considered in several publications [12-13], and the behavior has been modeled mathematically. The models may be grouped into the continuum mechanics and micromechanics approaches. It is reported that the micromechanics approach well predicts the strength for particle reinforced MMCs [1].



Figure 4: Changes in yield strength with volume fraction of Al₁₃Fe₄.

In the micromechanics approach, the principal strengthening mechanism for particle reinforced MMCs would include dislocation strengthening, $\Delta\sigma_{dis}$, sub-grain strengthening, $\Delta\sigma_{gb}$, work hardening, $\Delta\sigma_{wh}$, and solid solution strengthening, $\Delta\sigma_{solute}$. Linear summation of such terms is often used to predict yield strength of MMCs. Thus, the predicted yield strength in this study was calculated as follows:

$$\sigma_{c} = \sigma_{m} + \Delta \sigma_{dis} + \Delta \sigma_{gb} + \Delta \sigma_{wh1} + \Delta \sigma_{wh2} + \Delta \sigma_{solute} \tag{1}$$

The detailed expression of each term in equation (1) and parameters used in the calculation are given in the reference [14]. Furthermore, in the calculation, the particle size is fixed as 5μ m (upper boundary) and 15μ m (lower boundary). It can be seen that the measured values well agree with the predicted ones. To compare the properties of Al/Al₁₃Fe₄ fabricated by the PSM and conventional melting and casting (CMC) process, the Al-Fe alloys were fabricated by the conventional gravity casting. In the case of Al-Fe by the CMC process, the yield strength decreases sharply beyond about 12vol% Al₁₃Fe₄ and thus this composition is thought to be the limit for the addition of Fe by the CMC process. On the contrary, Al/Al₁₃Fe₄ fabricated by the PSM exhibits good properties even up to 16vol% Al₁₃Fe₄. This is due to the unique microstructural features obtained by the PSM.

4. Conclusions

Based on the analyses of the Al/Fe in-situ composites fabricated by the plasma synthesis method, the following conclusions can be drawn.

- (1) The as-cast microstructures of Al/Fe consist of angular and needle-like $AI_{13}Fe_4$ intermetallic compounds embedded in the α -Al matrix. Small amount of AI_5Fe_2 is detected and this phase is observed only in the interfacial layer that is composed of $AI/AI_{13}Fe_4/AI_5Fe_2/Fe$.
- (2) The yield strength of Al/Al₁₃Fe₄ composites increase with volume fraction of Al₁₃Fe₄ and well agree with the predicted models.

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