Microstructures and Mechanical Properties of Al-20Si- x Fe ($x=3, 5, 7$) Alloys Manufactured by Rapid Solidification Processing

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Rapidly solidified Al-20Si- x Fe ($x=3, 5$ and 7 mass%) powders were prepared by gas atomization. The microstructure consisted of needle shaped δ -Al₄FeSi₂ particles and primary Si embedded in Al-Si-Fe eutectic. With increasing Fe content, the size and volume fraction of primary Si decreased. After extrusion, the microstructures of the extruded bars showed a homogeneous distribution of two different sizes β -Al₃FeSi and Si particles embedded in the α -Al matrix. By increasing the Fe content from Al-20Si to Al-20Si-7Fe, the ultimate tensile strength (UTS) increased from 215 to 404 MPa at 300 K and from 122 MPa to 240 MPa at 473 K. Of these alloys, Al-20Si-5Fe alloy showed the best wear resistance. The UTS varied significantly with the initial powder size but wear resistance was independent of powder size. The effect of the particles distributed in the Al matrix upon the UTS was considered in terms of the cooling rate and particle distribution.

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I. Introduction

Al-Si alloys are used in many application area such as the automotive and electronic industries due to their good wear resistance and low thermal expansion coefficient. These properties are attributed to the high volume fraction of hard Si particles embedded in an alloy matrix. It is desirable to improve not only the wear resistance but also the high temperature strength for further applications under new demanding conditions. The Si content increase improves the wear resistance and mechanical strength. However, with increasing Si content above the eutectic composition causes the primary Si crystals to become coarser, resulting in deterioration in mechanical properties. Several different techniques such as modification treatment⁽¹⁾⁽²⁾, ternary alloying⁽³⁾ or rapid solidification technique⁽⁴⁾⁻⁽⁶⁾ have been applied to refine primary Si crystals. Due to recent developments in the technique for consolidation of Al alloy powders⁽⁷⁾, rapid solidification of hypereutectic Al-Si alloys seems to be the most promising route to improve the wear resistance and strength.

In this study, Fe was selected as a ternary alloying element for the Al-20Si alloy. It has been reported that Fe is also effective in improving wear resistance by forming an Al-Si-Fe intermetallic compounds⁽⁸⁾⁽⁹⁾ and improving high temperature strength by forming fine dispersoids in the Al matrix. It is necessary to consider the whole process from manufacturing of the raw alloys by the gas atomization through to determination of the properties of extrudates for industrial applications. In the present work, the effect of the structure and cooling rate by

varying both Fe addition and powder size on the microstructure and mechanical properties of rapidly solidified and extruded Al-20Si- x Fe ($x=3, 5$ and 7 mass%) alloys were investigated. This study was carried out by using a combination of optical microscopy, transmission electron microscopy (TEM), energy dispersive X-ray spectroscopy (EDX), X-ray diffractometry (XRD), tensile tests at room and high temperatures, and wear testing.

II. Experimental Procedure

In order to manufacture the rapidly solidified Al-20Si- x Fe ($x=3, 5$ and 7 mass%) alloy powder, the master alloy prepared by induction melting process was remelted and rapidly solidified using a N₂ gas atomizer with a melt delivery nozzle of 3 mm in diameter into an annulus operating with a pressure of 0.8 Pa. The melt flow rate, estimated from operating time and weight of atomized melt, was about 2×10^{-2} kg/s. The gas flow rate, calculated from gas consumption rate, was about 1.3×10^{-2} Nm³/s.

After atomization, the as solidified alloy powders were sieved into three groups depending on size: group A-powders smaller than 62 μ m; group B-powders larger than 62 and smaller than 120 μ m, and group C-powders larger than 120 and smaller than 210 μ m. Each group of alloy powders was consolidated by canning, degassing for 1 h at 673 K down to 0.13 Pa (10^{-3} Torr) and then hot extrusion after 1 h holding at 673 K with a reduction ratio of 25:1 into a bar with 10 mm in diameter. None of the extruded bars showed blistering or cracking on the surface.

The microstructures of the Al-20Si- x Fe powders and bars were examined by optical microscopy after polishing

and etching in Keller's reagent. In order to identify the primary Si size variation with the cooling rate, each 30 powders of 30–40, 45–55, 80–90 and 170–180 μm were freely chosen and the in Si sizes were measured. For transmission electron microscopy, thin foil specimens were prepared by ion beam milling after mechanical grinding of extrudates. The microstructures of the specimens were also observed by using a CM20 transmission electron microscope operating at 200 KeV.

The crystal structures of the constituent phases in the alloy powders were determined by X-ray diffractometry (XRD). XRD traces were obtained with monochromatic Cu-K α radiation over a 2θ range of 10° – 70° . Mechanical properties of the alloy bars were evaluated by tensile and wear tests. Alloy bars were machined into small tensile specimens in accordance with ASTM-A370 and tensile testing was performed at 300 K and 473 K with a cross-head speed of 0.2 mm/min by using an Instron 4206 machine. For wear testing, plate type specimens were polished and mounted on an Ohgoshi-type wear testing machine. In Ohgoshi wear testing, ring-shaped material (AISI 1045) is pressed and rotated against a specimen surface, forming a wear track. During the test, the sliding distance and load were kept constant at 100 m and 2.1 kg respectively.

III. Results and Discussion

1. Microstructure of powder and extrudate

The size of Al-20Si-xFe ($x=3, 5$ and 7) powders

manufactured by N₂ gas atomization varied from 10 to 210 μm with a mean powder size of about 75 μm . The mean powder size was almost independent of alloy composition for a given atomization condition in the study. The smaller the powder sizes, the more spherical in the shape. With increasing powder size, the shape changes to tear drop and then to an irregular shape. Also many satellites were observed on the surface of alloy powder.

Al-20Si alloy powder showed a microstructure consisting of primary Si particles embedded in the Al-Si eutectic structure. The microstructural scale decreased with decreasing powder size. **Figures 1(a)(b)(c)** and **(d)(b)(e)** show optical micrographs of Al-20Si-xFe ($x=3, 5$ and 7) about 75 μm in diameter and Al-20Si-5Fe alloy powders with three different sizes of about (d) 30 μm , (b) 75 μm and (e) 170 μm , respectively. For a given powder size of 75 μm in the horizontal direction, the volume fraction and size of the needle shaped particles increased whereas but the size of primary Si decreased with increasing Fe content as shown in **Fig. 2**. The figures show that the Si size was reduced from about 5 to 2.5 μm in the composition of Al-20Si-5Fe with decreasing powder size from 70–180 to 30–40 μm . With increasing amount of Fe addition from 3 to 7 mass%, the Si size decreased to 3 from 5 μm . The composition and crystal structure of the needle shaped particles in the alloy powder was analyzed by EDX and XRD experiments. An XRD trace in **Fig. 3** obtained from Al-20Si-5Fe powders shows that all the diffraction peaks could be analyzed as a mixture of fcc α -Al, diamond cubic Si and tetragonal δ -Al₄FeSi₂ intermetallic compound phases⁽¹⁰⁾.

Fig. 1 Optical micrographs of rapidly solidified Al-20Si-xFe ($x=3, 5$ and 7) powders. (a) $x=3$, 75 μm , (b) $x=5$, 75 μm , (c) $x=7$, 75 μm (d) $x=5$, 30 μm , (e) $x=5$, 170 μm .

Optical micrographs of Al-20Si-5Fe powders in Figs. 1(d), (b) and (e) arrayed vertically show that the microstructure of the coarse alloy powders again consists of needle shaped δ -Al₄FeSi₂ particles, primary Si crystals and Al-Fe-Si eutectic. With decreasing powder size, the microstructural scale and fractions of δ -Al₄FeSi₂ and primary Si decreased, as shown in Fig. 1. Al-20Si-5Fe powders smaller than 30 μ m often revealed a microstructure of α -Al dendrites, as shown in Fig. 1(d).

During gas atomization of the Al-20Si-*x*Fe alloy, a liquid stream of molten metal was disintegrated into small particles by a high velocity gas jet and then solidified rap-

idly by a surrounding gas. Cooling rate increased with decreasing powder size. We can see that the Al dendrites are formed only at the powder smaller than 30 μ m as shown in Fig. 1(d). Therefore, the solidification behavior of Al-20Si-5Fe alloy powders may be divided into two groups depending on their size. For powders larger than about 30 μ m, liquid undercooling is not so high and the nucleation of an intermetallic compound or primary Si cannot be suppressed. As a result we can suppose that the solidification occurs in the following sequence: (1) nucleation and growth of metastable δ -Al₄FeSi₂ particles, (2) nucleation and growth of primary Si and finally and (3) eutectic solidification of the remaining liquid. Due to the formation of δ -Al₄FeSi₂, the size and fraction of the primary Si crystal decreased with increasing Fe content in Al-20Si-*x*Fe alloys. The solidification sequence can be proven by the DTA experiment, which shows that one endothermic peak at 863 K and two exothermic peaks at 734 and 628 K. The microstructures of the materials quenched from the two different exothermic peaks demonstrated that the intermetallic compound of δ -Al₄FeSi₂ and primary Si were formed of the high and low temperature peaks, respectively, indicating the formation of the phases in the order of δ -Al₄FeSi₂ and Si. In addition, the increase in volume fraction of δ -Al₄FeSi₂ and the decrease in Si with addition of Fe are another evidence for the solidification sequence, as shown in Fig. 2.

If the powder size was smaller than 30 μ m (*i.e.* high cooling rate), as shown in Fig. 1(d), the nucleation and growth of δ -Al₄FeSi₂ and Si particles may be suppressed due to the high cooling rate in the liquid state and the supersaturated α -Al phase nucleates first and starts to grow into the surrounding undercooled liquid. Due to the la-

Fig. 2 Variation in primary Si size with Fe content and the powder size of Al-20Si-*x*Fe (*x*=3, 5 and 7) alloys.

Fig. 3 XRD profiles of (a) the as-solidified Al-20Si-5Fe powder and (b) the extruded bar.

tent heat evolution at the growing solid liquid interface, the α -Al/liquid interface temperature increases, the growth rate of the α -Al phase decreases as the interface moves, and finally eutectic solidification occurs. Indeed we can see in Fig. 1(d) that dendrite arm spacing is increasing as the interface moves from the nucleation point, which indicates that the growth rate decreased as the α -Al phase grows. The α -Al phase solidifies into a cellular or dendritic structure because the planar solid/liquid interface is unstable when the solid grows into an undercooled liquid with a velocity lower than that given by the absolute stability theory. At the later stage of solidification, the growth rate is limited by the heat balance between the latent heat of solidification and the cooling heat flux of the powder.

Figures 4(a) and (b) show optical micrographs of hot extruded Al-20Si-xFe ($x=3$ and 7) alloy bars. The micrographs of the bars were selected from (a) central and (b) edge part of transverse part and (c) parallel part to extruding direction. XRD experiments in Fig. 3(b) showed that the δ -Al₄FeSi₂ phase had transformed into an equilibrium β -Al₅FeSi phase with a monoclinic crystal structure, during degassing and hot extrusion⁽¹⁰⁾. The microstructure of Al-20Si-xFe alloy bars showed β -Al₅FeSi and Si particles distributed homogeneously in an α -Al matrix. It can be seen that the size of primary Si was not changed, whereas the eutectic microstructure coarsened in order to reduce the specific surface area during the hot degassing and extrusion process. It should be noticed that the β -Al₅FeSi which was transformed from δ -Al₄FeSi₂ during hot consolidating processes was fragmented into small particles, due to severe plastic deformation of the α -Al matrix. An extra atom of Si in the transformation, δ -Al₄FeSi₂ $\rightarrow$ β -Al₅FeSi + Si, was thought to be incorporated into the eutectic Si coarsening during the hot consolidation process⁽¹⁰⁾. In addition, it was shown that the microstructure became more homogeneous and refined with decreasing initial alloy powder size in Al-20Si-5Fe alloy bars.

Figure 5 showed a bright field TEM micrograph of the Al-20Si-5Fe alloy bar and a selected area diffraction pattern (SADP) taken from a small Al-Fe-Si intermetallic compound. The SADP could be analyzed into a monoclinic β -Al₅FeSi crystal structure with lattice constants of $a=0.612$ nm, $c=4.15$ nm and $\beta=91^\circ$ ⁽¹⁰⁾ in agreement with the XRD results. The streaks in the [001] direction indicate the presence of planar defects in {001} of β -Al₅FeSi particle, as marked by arrows.

It can be seen that these types of planar defects and the cracking of the Al-Si-Fe intermetallics during hot processes may promote the transformation reaction from the δ to β phase, since the thermal reaction activated along the increased surface area by the crack formation promoted diffusion of Si and Al. Two different sizes of β -Al₅FeSi particles appeared in the extruded bar: large particles 2–3 μ m long, and a smaller sized particle about 0.1–0.5 μ m. The large ones were considered to form by fragmentation of the needle-shaped coarse δ -Al₄FeSi₂ particles and the small β -Al₅FeSi particles were from δ -

Fig. 4 Optical micrographs of the rapidly solidified and extruded Al-20Si-xFe ($x=3$ and 7) alloy bars. (a) $x=7$ and (b) $x=3$.

Al₄FeSi₂ particles in the Al-Fe-Si eutectic. α -Al showed an equiaxed grain 2–3 μ m in size. Si particles 0.5–1 μ m in size appeared at α -Al grain boundaries, indicating coarsening of Si in the Al-Fe-Si during degassing and hot extrusion.

2. Mechanical properties

The room and high temperature tensile properties of the various extrudates were summarized in Table 1. By adding 7 mass%Fe to an Al-20Si alloy, the ultimate ten-

Fig. 5 TEM microstructure taken from the Al-20Si-5Fe alloy bar.

sile strength (UTS) increased from 215 to 404 MPa at 300 K and from 122 to 240 MPa at 473 K. Also the UTS of the Al-20Si-5Fe alloy at room temperature increased from 329 to 390 MPa with decreasing initial alloy powder size from group C to group A. The increase of the UTS with increasing Fe content may result from a homogeneous distribution of thermally stable β -Al₅FeSi particles and reduced primary Si particles as shown in Fig. 4. Also the increase in the UTS with decreasing initial alloy powder size indicates that the fine distribution of β -Al₅FeSi and Si particles was attributed to the large amount of undercooling which was important for further increasing UTS in the Fe-containing alloys. It can be supported from the microstructure showing that the sizes of the β -Al₅FeSi and primary Si particles in the extruded bars of groups C and A measured 4–5 and 2–3 μ m, respectively. As a result, one can say that increasing the fraction of β -Al₅FeSi in the Al matrix by Fe additions and increasing cooling rate by powder size both lead to improvements in UTS.

The work hardening exponent, n , is usually calculated from true stress (σ)-true strain (ϵ) relation, described as $\sigma = K\epsilon^n$. The value of n was calculated to be 0.17, indicating minimal work hardening. This was confirmed by stress-strain curves which showed little difference in the

Fig. 6 Variation in specific wear properties of Al-20Si- x Fe alloy bars as a function of sliding speed.

values between yield strength (YS) and UTS. The TEM micrograph as shown in Fig. 5 also supported this theory. We can see that the large volume fraction of intermetallics and small subgrain boundaries which act as a dislocation sinks.

In order to examine the strengthening mechanism of the rapidly solidified Al-Si-Fe alloys, based on Figs. 1 and 4, the relationship between UTS and inter-particle distances of primary Si, $d^{1/2}$ and d was examined. Here, symbols $d^{1/2}$ and d indicate the dispersion strengthening and particle strengthening effects, respectively. The linear relations between UTS and both types of the inter-particle distances indicated that the highly improved mechanical properties of the rapidly solidified hypereutectic Al-Si alloys could be attributed to a combination of dispersion strengthening and particle strengthening mechanism⁽¹¹⁾.

Figure 6 showed a variation of specific wear behavior of Al-20Si- x Fe alloy bars as a function of sliding speeds from 0.5 to 3.6 m/s. With increasing Fe content to 5%, the specific wear decreased significantly throughout the sliding speed range. For Al-20Si and Al-20Si-3Fe alloys, the specific wear increased gradually with increasing

Table 1 Summary of tensile properties of Al-20Si- x Fe alloy bars.

Alloys	UTS at 300 K (MPa)	Elong. at 300 K (%)	UTS at 473 K (MPa)	Elong. at 473 K (%)
Al-20Si**	215	12.2	122	16.7
Al-20Si-3Fe**	301	8.4	179	12.5
Al-20Si-5Fe*	390	1.3	231	3.5
Al-20Si-5Fe**	359	4.3	206	4.1
Al-20Si-5Fe***	329	5.6	194	5.6
Al-20Si-7Fe**	404	2.2	240	3.4
Al-20Si-5Fe-2N{9}	424	1.0		

*from group A powders **from group B powders ***from group C powders.

sliding speed, indicating that the Si and Al-Si-Fe intermetallics contribute to improve wear properties simultaneously. However, the specific wear of Al-20Si-5Fe and Al-20Si-7Fe alloys increased slowly, reached a maximum at a sliding speed of 2.4 m/s, and finally decreased with a further increase of the sliding speed.

The reversed behavior of the wear properties compared to the UTS between Al-20Si-5Fe and Al-20Si-7Fe alloys should be considered. One can say that the UTS was certain to be improved by the refined and densified distribution of the Si and Fe intermetallic compound, especially arranged in the direction of the tensile force. Also the Fe intermetallic compounds are well known to improve the room and elevated temperature strength. The poorer wear property, however, may result from the lack of Si particles, the main wear resistant particles, in the Al-20Si-7Fe alloy, due to the consumption of Si by forming Fe intermetallics. It means the important contributions of Fe intermetallic and Si, to UTS and wear resistance respectively, in the rapid solidification process. However, we can note that any effect of the powder size on the wear property was not found in Al-20Si-5Fe powder extrudates. It can be thought that once the wear value reached a maximum by a combination of composition, particle distribution and particle size, it is unnecessary to reduce the particle size for the further improvement. Therefore, one can say that it is desirable to combine those conditions in balance.

IV. Conclusions

As the solidified Al-20Si-xFe powder showed microstructures consisting of needle shaped δ -Al₄FeSi₂ and primary Si particles embedded in an Al-Fe-Si eutectic. With increasing Fe content, the size and volume fraction of the δ -Al₄FeSi₂ increased but the size and volume fraction

of primary Si decreased. During degassing and hot extrusion, δ -Al₄FeSi₂ transformed into an equilibrium β -Al₅FeSi phase and fragmented into small particles. The microstructures of the extruded bars showed a homogeneous distribution of two different sizes of β -Al₅FeSi and Si particles embedded in the α -Al matrix. With increasing Fe content from Al-20Si to Al-20Si-7Fe, the UTS increased from 215 to 404 MPa at 300 K and from 122 MPa to 240 MPa at 473 K. A combination of dispersion strengthening and particle strengthening effects was identified as the main strengthening mechanism of this alloy. The variation of cooling rate and Fe intermetallic particle density plays an important role for the UTS of the materials. Al-20Si-5Fe showed the best wear resistance. The UTS varied significantly with the initial powder size, whereas wear resistance was independent of the initial alloy powder size.

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