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# Microstructure and internal friction of spray deposited Al-3.3Fe-10.7Si alloy

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Al-3.3Fe-10.7Si alloy has been experimentally made with spray deposition technology. The internal friction of the alloy which was directly associated with the microstructures under spray deposited, extruted and heat treated conditions has been investigated using a low frequency inverted torsion pendulum over the temperature region of 10–300 °C. An internal friction peak was observed in the temperature range 50–250 °C in the present alloy. The $Q^{-1}$ peak decreased after extruted and in subsequent to the earliness of isothermal annealing, which was found to be directly attributed to the precipitation of $FeAl_2$ and Al—Fe—Si intermetallics from the supersaturated aluminium alloy matrix. We suggest that the internal friction peak in the alloy originates from grain boundary relaxation, but the grain boundary relaxation can also be affected by $FeAl_2$ and Al—Fe—Si intermetallics at the grain boundaries, which will impede grain boundary sliding.

## 1. Introduction

Recently, spray-deposited processing has received considerable attention for the results of several promising characteristics of the technique [1–4]. Firstly, improved properties can be obtained as compared with rapid solidification powder metallurgical (P/M) products. For instance, the technique eliminates the handling of fine reactive particulates, can apparently minimize oxidation of the alloys and reduce the manufacture cost. Then, spray-deposited materials have been reported to exhibit characteristics associated with rapid solidification, namely, fine-grained microstructures, increased solid solubility, nonequilibrium phases, and the absence of macrosegregation. Finally, the high damping particulate reinforced metal matrix composites can be made by means of the spray-deposited processing. The mechanical properties in the spray-deposited Al—Fe—Si alloy has been the subject of active investigation [5]. However, no internal friction studies associated with pricipitation of spray deposition Al—Fe—Si alloys are known. In this paper, we report on the internal friction results of Al-3.3Fe-10.7Si alloy with a discussion on the internal friction behaviour in terms of grain boundary relaxation.

## 2. Experimental procedure

### 2.1. Materials synthesis

The Al—Fe—Si alloy utilized in the present study was of commercial grade, nominally composed of 3.3 wt % Fe, 10.7 wt % Si and balance aluminium. A spray deposition processing variables is presented in Table I.

### 2.2. Hot extrusion and heat treatment

A 2.01-cm-diameter extrusion billet was removed from the spray-deposited materials and was hot extruted at 450 °C using a reduction ratio of 8 : 1. The specimens was subsequently preannealed at 360 °C for 1000 min.

### 2.3. Microstructural characterization

Microstructural characterization of the alloy was investigated in KYKY-AM-RAY 1000B scanning electron microscope (SEM) and H-800 transmission electron microscope (TEM)

### 2.4. Internal friction measurements

Internal friction was measured with a low frequency inverted torsion pendulum. Representative sections from the spray-deposited, extruted and preannealed samples were removed and machined into $1 \times 2.5 \times 35$ mm. All internal friction curves were obtained at a heating rate of $5\,^{\circ}\mathrm{C} \cdot \mathrm{min}^{-1}$, corresponding to a strain amplitude of $1 \times 10^{-6}$.

## 3. Results

### 3.1. Microstructure

Fig. 1 shows the microstructure of the as-deposited, as-extruted and heat treated Al—Fe—Si alloy. Fig. 1a reveals the presence of the silicon particles, with an average size of 4 $\mu$m. This agrees with previous work [6]. Micropores are observed in the as-deposited specimens. Fig. 1b,c allow the effect of hot extrusion and heat treatment on the spray deposited alloy microstructure to be observed. The silicon particles indicated by the arrows in Fig. 1 are seen to be slightly lessened over that found in the as-sprayed condition, which are broken up during extrusion. Additionally, it is interesting to note little needle phases and less size particles (Fig. 1b,c). It is shown that the needle phases are $FeAl_2$ and less size particles are Al—Fe—Si intermetallic compounds by means of X-ray energy dispersive spectromtry. Also, there is barely discernible changement in the silicon particle size after the heat treatment. Fig. 2 reveals the electron micrograph of Al—Fe—Si alloy in the different states. Inspection of Fig. 2 shows that intermetallic compound particles (mainly $FeAl_2$) with the bigger size are distributed mainly in grain boundaries in as-extruted alloys (A in Fig. 2a). During heat treatment, the size of the base alloy grains is basically a constant (Fig. 1c). Besides, intermetallic compound particles with the smaller size precipitate from Al alloy matrix (B in Fig. 2b).

## 3.2. Internal friction

### 3.2.1. Internal friction of as-deposited and as-extruted Al—Fe—Si alloy

Internal friction variations with temperatures in the region of 10–300 °C for as-deposited and as-extruted Al—Fe—Si alloy are shown respectively in Fig. 3. A broad internal friction peak superimposed on a large high-temperature background was observed in the temperature region of 50–250 °C in the alloy. The internal friction peak height and background of as-extruted alloy are much lower than those of as-deposited alloy.

TABLE I Processing variables

| | |
|---|---|
| Atomization pressure | 3.0 MPa |
| Atomization gas | $N_2$ |
| Injection angle | 75° |
| Pouring temperature | 870 °C |
| Atomized droplet flight distance | 400 mm |

### 3.2.2. The effect of experimental frequency and annealing treatment on the internal friction

The effect of experimental frequency on internal friction is shown in Fig. 4a. Prior to the measurements, the specimen had been annealed *in situ* at 360 °C 1000 min to eliminate the instability of internal friction due to the precipitation of saturated solutes. The internal friction peak height varies very little with frequency, indicating an absence of any considerable change in the structure during the measurements. The shifting of the internal friction peak to a higher temperature with increasing frequency means that the origin of internal friction peak can be explained by anelastic relaxation. To determine the temperature of the internal friction peak, we assume the high-temperature background to be $Q_b^{-1} = Q_0^{-1} + A_{\exp}(-B/T)$ where the parameters, $A$ and $B$ can be obtained from the low and high temperature internal friction data, the value of $Q_0^{-1}$ is in the region between $Q^{-1} = 0$ and the internal friction value at room temperature (i.e. $Q_0^{-1} < Q_{RT}^{-1}$). Internal friction curves after subtraction of the background are shown

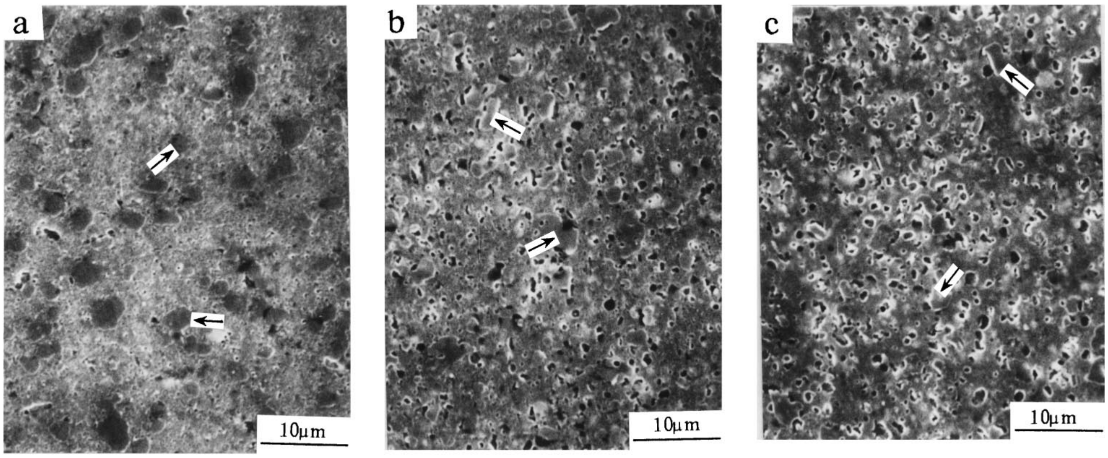

*Figure 1* Microstructures of spray deposited Al—Fe—Si alloy: (a) As-deposited; (b) as-extruted; (c) as-extruted + annealing.

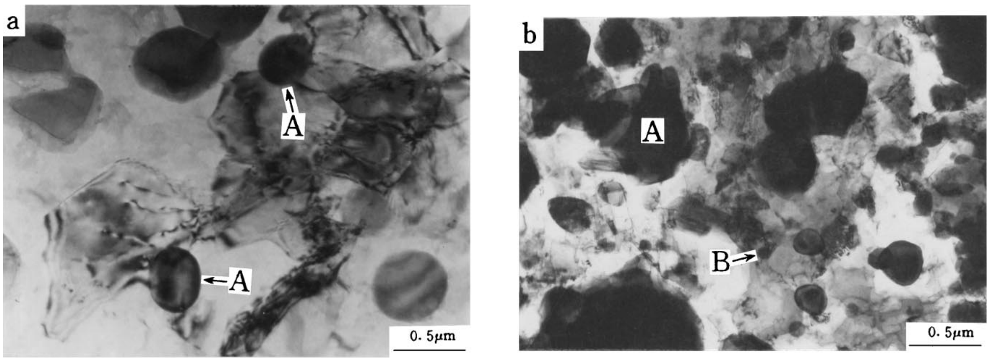

*Figure 2* Electron micrograph of Al—Fe—Si alloy, the intermetallic compound particles (A, B) precipitated are distributed mainly in grain boundaries: (a) As-extruted; (b) as-extruted + annealing (1000 min).

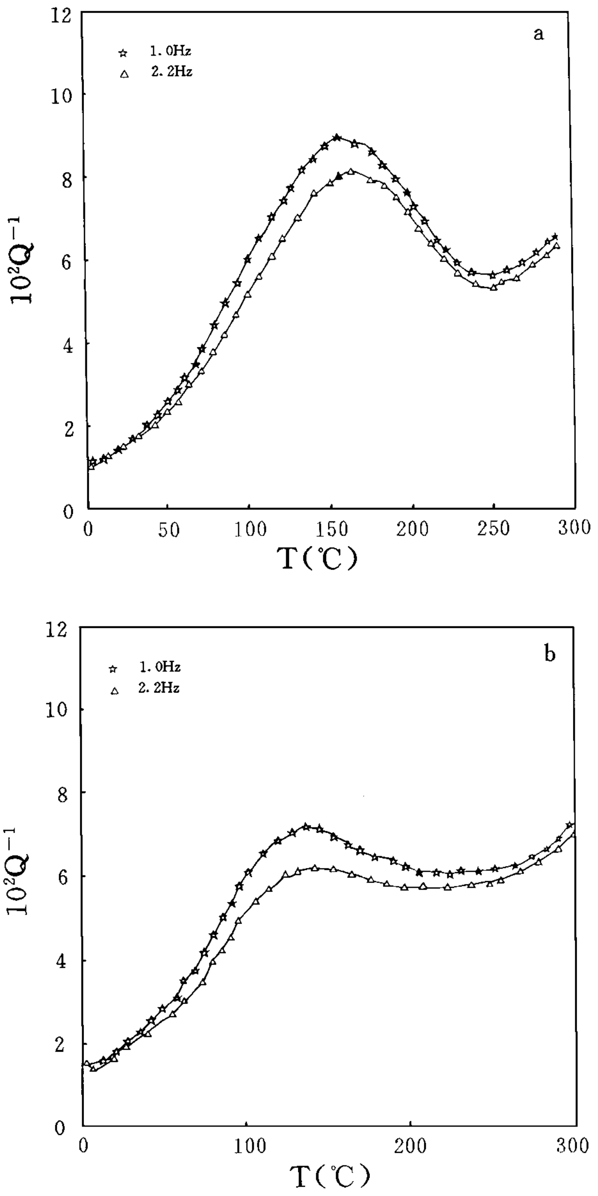

*Figure 3* Internal friction vs. temperature of the alloy: (a) As-deposited; (b) as-extruted.

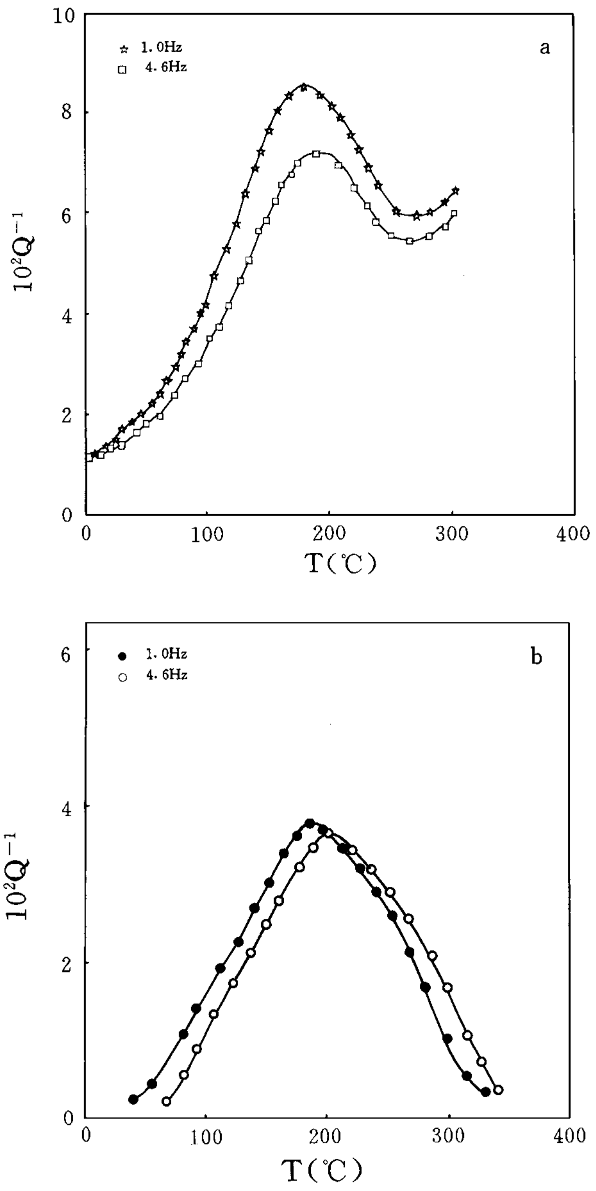

*Figure 4* The effect of frequency on the internal friction of Al—Fe—Si alloy preannealed at 360 °C for 1000 min: (a) Original data and background; (b) after subtraction of the background.

in Fig. 4b. The activation energy for the relaxation process is found to be 41 Kcal $\text{mol}^{-1}$ from the shift of the peak temperature with frequency, and is slightly larger than that of a pure polycrystalline aluminium (34 Kcal $\text{mol}^{-1}$) [7]. The preexponential factor $\tau_0$ is found to be $3.1 \times 10^{-21}$ s, and is much lower than the value of $10^{-14}$ s for a pure polycrystalline aluminium [8]. The effect of isothermal annealing on the internal friction of Al—Fe—Si alloy is shown in Fig. 5. The height of the internal friction peak decreased slightly and then unchanged with the increase of annealing time.

## 4. Discussion

It is well known that spray-atomized and deposited materials can exhibit characteristics associated with rapid solidification, namely, fine-grained microstructures, increased solid solubility. Because of heating and subsequent air-cooling when hot-extruted, supersaturated phases in deposited specimens precipitate partially (Fig. 1b). During heat treatment, precipitation

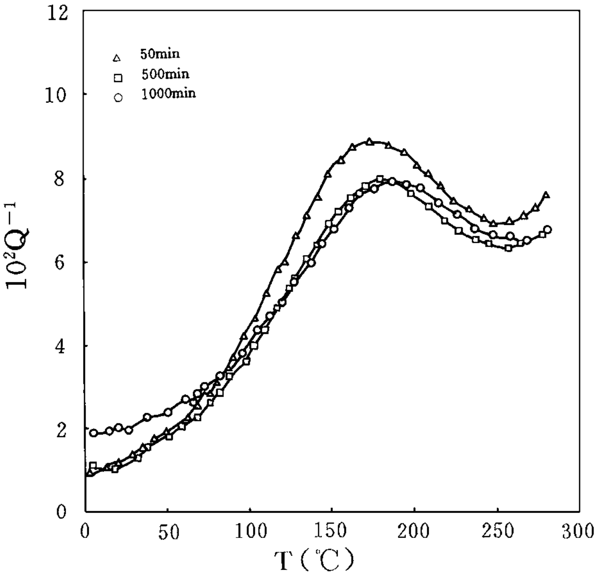

*Figure 5* Internal friction vs. temperature of the Al—Fe—Si alloy preannealed at 360 °C for different times ($f = 1.0$ $H_2$).

happen completely (Fig. 2b). These results don't agree with that of reference [9], which specimens are obtained by means of P/M process. The reason is that the precipitation for a given alloy is mainly controlled by the degree of supersaturation, the cooling rate of atomized powder is higher 10–100 times than that of spray deposited materials, as a result, alloys from rapidly solidified powder have the bigger saturated degree, precipitation tends to happen completely during hot extrusion and subsequent air cooling.

The reason why the coarsening of the base alloy grains is not very obvious during heat treatment at 360 °C is associated with the addition of iron [9]. The iron-bearing intermetallic particles with a fairly high heat resistance block the migration of grain boundaries, modify the recrystallization behaviour of the present alloy and inhibit the coarsening of the grains. In addition, after the annealing, barely discernible change in the silicon particle size is also related to iron in two ways: (1) it is probable that the activity of silicon is reduced due to the interaction between the silicon particles and Al—Fe—Si intermetallics or even the incorporation of iron in the slilicon particles; (2) the coarsening of silicon particles proceeds through the diffusion of silicon atoms over a long distance from one particle to another, occuring preferentially along grain and subgrain boundaries because the activation energy for pipe diffusion is lower than for volume diffusion. Al—Fe—Si dispersed intermetallic phases, which are broken up during extrusion and redistributed on a fine scale at grain and subgrain boundaries, as well as within grains, may act effectively as sinks for vacancies and obstacles to the diffusion of silicon atoms over a long distance, particularly along the grain and subgrain boundaries with which the intermetallic particles interact. As a result, the growth of silicon particles becomes difficult and their coarsening rate is relatively slowed down.

It is also well known that an internal friction peak is observed at 290 °C in polycrystalline aluminium, and that this peak results from grain boundary relaxation [7], which may be impeded by the precipitate phases at the grain boundaries [8]. We suggest that the internal friction peak of the Al—Fe—Si alloys with a-height much lower than that of the pure polycrystalline aluminium and a position at a lower temperature is also related to grain boundary relaxation. This peak is affected by the $FeAl_2$ particles and Al—Fe—Si intermetallics at the grain boundary, which block grain boundary sliding and, as a result, the internal friction peak is reduced and shifted to a lower temperature. Al—Fe—Si intermetallics in as-deposited alloy is thermodynamically metastable and precipitates at grain boundaries or inside the aluminium grains during hot-extrusion. Therefore, the internal friction peak should be lowered after hot extrusion.

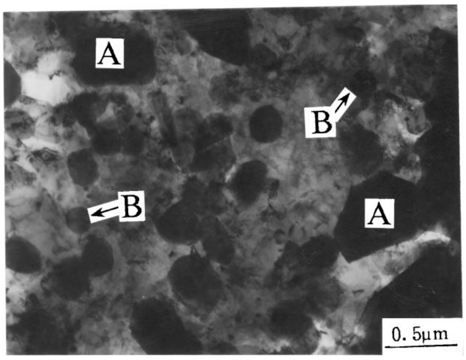

*Figure 6* Electron micrograph of Al—Fe—Si alloy. The intermetallic compound particles (A, B) precipitated are distributed mainly in grain boundaries. As-extruted + annealing (500 min).

The effect of isothermal annealing on the internal friction behaviour of the Al—Fe—Si alloy can also be discussed in terms of the precipitates impeding grain boundary relaxation. In early time of isothermal annealing, the incomplete precipitating Al—Fe—Si saturated solution precipitates at grain boundaries, the number of the precipitates at the grain boundary should increase, as a result, the internal friction peak is reduced. After end of the precipitation, the coarsening of the matrix grains and silicon particles is impeded by iron-bearing intermetallics during isothermal annealing, namely, the size of the grains tends to be constant. On the other hand, the results from electron micrograph of the alloy (Fig. 6) preannealed at 360 °C for 500 min and 1000 min show that the size and the number of the precipitates tends to be the same for two different annealing time. i.e., precipitation has basically finished when the alloy is preannealed at 360 °C for 500 min, therefore, the height of the internal friction peak in Fig. 5 is basically changeless with isothermal annealing.

The pre-exponential factor $\tau_0$ is related to the internal friction peak temperature and a large decrease of $\tau_0$ will result in a lower peak temperature if the activation energy for this process is constant. The increase of the number of the precipitates can cause the peak temperature to shift to a lower temperature. Therefore, one may assume that the very small value of $\tau_0$ for the Al—Fe—Si alloy, in comparison with that of the pure polycrystalline aluminium, can mainly be attributed to the presence of the precipitates.

## 5. Conclusion

(1) The grain size of the spray deposited Al-3.3Fe-10.7Si lessens after hot extrusion or subsequent to heat treatment. This is because the alloy forms a volume fraction of thermally stable intermetallic phases at the grain boundary, which inhibit the coarsening of the grains.

(2) An internal friction peak was observed in the temperature range of 50–250 °C in spray deposited Al-3.3Fe-10.7Si alloy, whose height of the internal friction peak is much higher than that of hot extrusion condition and isothermal annealing condition.

(3) It is suggested that the origin of the internal friction peaks observed in the spray deposited Al—Fe—Si alloy can be explained by grain boundary relaxation, which is affected by $FeAl_2$ particles and Al—Fe—Si intermetallic particles at the grain boundaries.

(4) The decrease of the height of internal friction peak in as-extruted Al—Fe—Si alloy and in subsequent to the earliness of isothermal annealing may be

attributed to the precipitation of $FeAl_2$ and Al—Fe—Si intermetallic particles from the supersaturated aluminium alloy matrix, which block grain boundary sliding. The unchangeableness of the internal friction peak height with annealing time may be related to the iron-bearing intermetallic particles inhibition to the coarsening of the grains at the grain boundaries.

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