Microstructure and mechanical properties of RSP/M AI-Fe-V-Si and AI-Fe-Ce alloys

U. PRAKASH, T. RAGHU, A. A. GOKHALE, S. V. KAMAT Defence Metallurgical Research Laboratory, Kanchanbagh, Hyderabad 500058, India

Two aluminium alloys with nominal compositions of Al-8Fe-4Ce and Al-8Fe-1V-2Si (all compositions in wt%) were rapidly solidified by ultrasonic gas atomization. The atomized powders with an average particle size (d_{50}) of 30 μ m were vacuum hot pressed and subsequently hot extruded. The P/M extrusion exhibited similar microstructure and elevated temperature tensile properties. The tensile and stress rupture samples of both the alloys exhibited ductile dimple failure. However, the Al-Fe-V-Si extrusion samples exhibited significantly better creep and stress rupture properties. The Al-Fe-Ce alloy was found to be more susceptible to cavitation at elevated temperatures which resulted in poor stress rupture properties. © 1999 Kluwer Academic Publishers

1. Introduction

Dispersion strengthened aluminium alloys based on Al-TM (TM = transition metal) type systems are being developed for elevated temperature structural applications in aerospace at temperatures up to 623 K as possible substitutes for the more expensive and refractory Ti alloys currently in use [1–3]. Alloy systems containing transition metals such as Fe, Ni, V, Cr, Zr, Mo and Ti with possible additions of Si and Ce (or misch metal) have been investigated. The elevated temperature strength of these alloys derives from the presence of a large (15 to 35%) volume fraction of fine and stable intermetallic dispersoids. Such a dispersion is usually achieved by employing rapid solidification (RS) techniques such as atomization or melt spinning followed by appropriate P/M processing [1–3].

P/M Al-Fe-Ce and Al-Fe-V-Si alloys are among the more promising alloys being developed. In Al-Fe-Ce alloys strengthening is primarily because of ternary dispersoids in an aluminium matrix [4] whereas in Al-Fe-V-Si alloys it is due to the presence of Al_{13} (Fe, V)₃Si dispersoids [5]. The objective of the present work is to compare the microstructure and mechanical properties of these two alloys prepared using identical processing routes.

2. Experimental

Alloy ingots with nominal compositions of Al-8Fe-4Ce and Al-8Fe-1V-2Si (all compositions in wt %) were prepared by air induction melting (AIM) under a halide flux cover in graphite crucibles followed by casting in cast iron moulds. The alloys were then remelted by AIM and atomized at 1523 K using a graphite nozzle of 3 mm diameter and atomization gas (argon) pressure of 60 kg/cm² in an ultrasonic gas atomization unit. The atomized powder was passed through a 100# mesh (BSS) sieve to eliminate coarse metal lumps and slag particles. The average particle size of the $-100# (<150 \,\mu\text{m})$

powder was determined in a Seishin SKC-2000S micron photosizer using ethylene glycol as the dispersing liquid. The atomized powders were consolidated by hot pressing at a temperature of 698 K in a split steel die in a 200 ton vacuum hot press. The vacuum hot pressed compacts were extruded at 698 K in a 2000 ton press equipped with a shear extrusion die using a 20:1 extrusion ratio and a ram speed of 5 mm/s.

Longitudinal sections of extruded rods were mounted in bakelite mounts and mechanically polished to $1 \,\mu m$ grade powder finish. Electron probe microanalysis (EPMA) studies were carried out on the polished sections in a CAMEBAX MICRO EPMA unit. Creep, stress rupture and tensile test samples were machined from the extruded rods and polished using 600 grit abrasive. The geometry and dimensions of samples used for tensile, creep and stress rupture tests are shown in Fig. 1a to c respectively. Tensile tests were carried out at 298, 473 and 573 K in a 100 kN Instron 1185 testing machine at a strain rate of 8×10^{-4} /s. Constant load creep and stress rupture tests were carried out in a 10 kN ESH creep testing machine. Tensile and stress rupture fracture surfaces were studied in a JEOL JSM-840 scanning electron microscope (SEM). Bulk hydrogen content of the extruded rods was determined using a LECO 402 hydrogen determinator. Young's modulus of the extruded rods was measured on cylindrical samples measuring $120 \text{ mm} \times 16 \text{ mm}$ diameter in an ultrasonic modulus measuring device.

3. Results

The -100# atomized powders exhibited an average particle size (d_{50}) of 30 μ m. A bimodal particle size distribution (Fig. 2) was observed for both the alloy powders. The Al-Fe-V-Si extrusions had a bulk hydrogen content of 5.3 ppm while the Al-Fe-Ce extrusions exhibited a higher hydrogen content of 8.1 ppm. The Al-Fe-Ce extrusion had a modulus of 85 ± 1 GPa while





ENLARGED VIEW OF RIDGES



Figure 1 Showing design of (a) tensile, (b) creep and (c) stress rupture samples.



Figure 2 Showing typical particle size distribution in atomized powders.

a modulus of 89 ± 1 GPa was recorded for the Al-Fe-V-Si extrusion. EPMA revealed that for both the alloys, the extrusion microstructure (Figs 3a and b) comprised of alternate bands of regions containing fine and coarse dispersoids. The bands were parallel to the extrusion direction. EPMA X-ray mapping confirmed that the Al-Fe-Ce extrusions contained primarily ternary dispersoids and minor amounts of binary Al-Fe dispersoids. The Al-Fe-V-Si extrusions contained only quarternary dispersoids.

The results of tensile testing are summarised in Table I. At room temperature, the Al-Fe-Ce alloy



Figure 3 EPMA Backscattered electron image showing the microstructure in (a) Al-Fe-Ce and (b) Al-Fe-V-Si extrusions respectively.

TABLE I Tensile properties of Al-Fe-Ce and Al-Fe-V-Si alloys

Alloy	Test temperature (K)	0.2% Yield strength (MPa)	Tensile strength (MPa)	Total plastic elongation (%)	Uniform plastic elongation (%)
Al-Fe-Ce	298	486	560	2.1	2.1
	473	276	292	6.2	1.6
	573	209	209	4.4	0
Al-Fe-V-Si	298	358	420	9.2	2.3
	473	274	294	4.8	1.5
	573	208	208	4.2	0

exhibited a significantly higher yield and tensile strength as compared to the Al-Fe-V-Si alloy. Increasing the test temperature resulted in a relatively larger reduction in the yield and tensile strength of the Al-Fe-Ce alloy as compared to the Al-Fe-V-Si alloy. Thus at elevated temperatures, both the alloys exhibited similar strength. The uniform plastic elongation values were similar for both the alloys at all temperatures and decreased with increasing test temperature. The primary contribution to the total elongation changed from uniform elongation at room temperature to local elongation (necking) at room temperature.

Ductile fracture with a bimodal dimple distribution was observed in room temperature tensile samples of both the alloys (Fig. 4). No significant change in fracture characteristics was observed in elevated temperature tensile and stress rupture samples. The results of creep and stress rupture tests are summarised in Fig. 5a and b and Table II respectively. The Al-Fe-V-Si alloy exhibited a minimum creep rate of 7.5×10^{-6} per hour as compared to 7.2×10^{-4} per hour exhibited by the Al-Fe-Ce alloy when tested at 573 K and an initial creep stress of 100 MPa. Thus the Al-Fe-V-Si alloy exhibits vastly superior creep resistance. It also has a significantly longer stress rupture life as compared to the Al-Fe-Ce alloy under the test conditions used in this study. Cavitation was found to occur along



Figure 4 SEM fractograph showing ductile failure with a bimodal dimple distribution in tensile samples of Al-Fe-Ce (and Al-Fe-V-Si) extrusions.



Figure 5 Showing creep curves obtained at 573 K and a stress of 100 MPa from (a) Al-Fe-Ce and (b) Al-Fe-V-Si alloy samples.

 $\label{eq:TABLE_II} \begin{array}{c} \mbox{TABLE II Stress rupture properties of Al-Fe-Ce and Al-Fe-V-Si alloys} \end{array}$

Alloy	Stress (MPa)	Temperature (K)	Rupture life (h)
Al-Fe-Ce	100	573	41.6
	100	623	0.5
	80	623	5.6
Al-Fe-V-Si	150	573	4.6
	100	573	>330
	100	623	52.4
	80	623	520

extrusion bands (Fig. 6) in elevated temperature tensile and stress rupture samples of both the alloys. The severity of cavitation increased with increasing temperature with linkage occurring across the bands at higher temperatures.

4. Discussion

The two alloy extrusions exhibit similar microstructure in SEM. The formation of bands of material alternately containing coarse and fine dispersoids observed in the present work has previously been reported [6] for P/M extrusions of alloys prepared from atomized alloy powders or melt spun ribbons. Microstructure of both powders and ribbons depends on the local rapid solidification conditions and is rarely uniform [4, 7]. The bands observed (Fig 3a and b) form during extrusion of the vacuum hot pressed powder preforms. However, the reasons for the segregation of coarse and fine dispersoids into different bands are not immediately obvious.

The ternary dispersoids in the Al-Fe-Ce alloy have been reported to be incoherent with the aluminium matrix [4]. The dispersoids in Al-Fe-V-Si alloy may be coherent up to a size of 100 nm above which they tend to lose their coherency [5, 6]. In the present work, a



Figure 6 Showing cavitation along extrusion bands in elevated temperature Al-Fe-V-Si (and Al-Fe-Ce) tensile samples tested at 573 K. Some linkage across the bands is also observed.

significant fraction of the dispersoids, particularly those in the bands containing coarse precipitates, are likely to be incoherent. Their volume fraction may be brought down by working with finer powder sizes which are subjected to higher cooling rates and thus are likely to have finer dispersoids. It is, however, unlikely that coarse dispersoids can be eliminated completely.

In the past, the Al-Fe-Ce alloy has generally been processed by gas atomization while the Al-Fe-V-Si alloy has been rapidly solidified by melt spinning [8, 9]. The creep properties of the Al-Fe-Ce alloy observed in this work are comparable to those reported for this alloy [8]. However, the creep properties of the Al-Fe-V-Si alloy are slightly inferior to those reported earlier [9]. The elevated temperature properties of the two alloys appear to be determined by the extent of cavitation. The Al-Fe-Ce alloy has a shorter stress rupture life and inferior creep properties. The onset of cavitation in Al-Fe-V-Si is evidently delayed during creep and stress rupture tests. The susceptibility of Al-Fe-Ce alloys to cavitation is not evident from the tensile test results probably because during tensile testing the time available for cavitation to occur is limited and thus both the alloys exhibit similar tensile properties at elevated temperatures. A high hydrogen content may lead to increased cavitation [4], and the Al-Fe-Ce alloy which shows greater susceptibility to cavitation indeed has a higher hydrogen content. A weak precipitate/matrix interface may also lead to an increase in susceptibility to cavitation. However, it is difficult to separate the effect of strengthening precipitate on cavitation from that of hydrogen because the extrusions have different hydrogen contents.

5. Conclusions

The Al-Fe-Ce and Al-Fe-V-Si extrusions studied had similar microstructure and elevated temperature tensile properties. However, the Al-Fe-V-Si alloys exhibited significantly longer stress rupture life and lower minimum creep rate as compared to the Al-Fe-Ce alloy. The poor stress rupture properties of the Al-Fe-Ce alloys have been related to a greater susceptibility to cavitation in the Al-Fe-Ce alloy samples at elevated temperature.

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