

MICROSTRUCTURAL FEATURES OF RS Al-BASED THERMAL-STRENGTHENED ALLOYS^①

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ABSTRACT To develop high temperature aluminium alloys for commercial and military aircraft, the as-quenched microstructural features of rapidly solidified Al-Fe-V-Si alloys were examined with XRD, TEM and Mössbauer spectroscopy. The results show that the microstructure of the alloy consists of microcellular α -Al and nearly spherical α -Al₁₃(Fe, V)₃Si nanoscale particles. There are three kinds of Fe atomic configurations in as-quenched state. The values of matrix lattice parameter decreases with the increasing of Fe solution concentration. The structure of α -Al₁₃(Fe, V)₃Si particles is *bcc* and its a_0 is of 1.260 nm.

Key words Al-Fe-V-Si alloys dispersive particles atomic configuration lattice parameter

1 INTRODUCTION

In recent years there has been considerable effort in the high-efficiency, high-performance and high-specific-strength material for commercial and military aircraft to develop high temperature aluminium alloys capable of competing with titanium alloys. Such alloys are typically double-strengthened by superfine grains and dispersive intermetallic compounds. So far, the rapid solidification P/M processing route has led to the development of several ternary or quaternary alloy systems based on the Al-Fe system by the addition of transition and rare earth elements (Co, Ce, Mo, Ti, Zr, V, Si, etc)^[1-3]. Simultaneously, the emphases of these studies have been devoted on the microstructures and properties of materials. The microstructural evolution of alloy ribbons, particularly, Fe atomic configurations is seldom reported, so in this paper the analysis is mainly put on the microstructural features in RS thermal-strengthened Al-Fe-V-Si alloy ribbons.

2 EXPERIMENTAL

Three quaternary Al_{97.6-x}Fe_{4.3}V_{0.7}Si_{1.7} ($x = 4.3\%$, 5.4% , 6.4%) alloy ribbons were

prepared by the melt spinning technique. The continuous ribbons were typically about 30 μm thick and 10 mm wide. The as-quenched microstructure and composition of fine precipitates were investigated in Philips EM420 transmission electron microscopy (TEM). Room temperature ⁵⁷Fe transmission Mössbauer effect spectra were obtained using a computer controlled FH-198 spectrometer operating in the constant-acceleration mode. The source was Pd ⁵⁷Co and the velocity was calibrated with a 12.7 μm thick Fe foil.

Individual specimens were placed with high purified powdered silicon as a standard in a *D/max-rB* X-ray diffractometer using CuK α radiation at 40 kV and 40 mA. The precision of 2θ is about 0.001° . A scan rate of $2\theta = 1^\circ/\text{min}^{-1}$ was used to measure Al peak height for matrix lattice parameter analysis of alloy ribbons. By means of the combination of $\cos^2\theta$ function and least square method the accurate values of matrix lattice parameter was given by extrapolated plots.

3 RESULTS AND DISCUSSION

The as-quenched microstructures of the cen-

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tral region of the quaternary Al-Fe-V-Si alloy ribbons were shown in Fig. 1. These regions were characterized by the microcellular structure and the random orientations and high volume fractions of very fine spherical dispersoids (less than 50 nm in diameter), which were inconsistent with typical cellular solidification structures where the size of the intercellular phase scales with the cell size and spacing. With the increase of Fe concentration, the trend of α -Al cellulation decreased, in the meantime, the particle further fining and the formation of particle cluster were observed, as shown in Fig. 1(b) and (c). It is suggested that the dispersoids are considered to be formed prior to the Al cells, i. e., as a primary phase. Probably a high nucleation rate of the intermetallic particles combined with a low growth rate and little growth time result in a very fine dispersion of particles in the undercooled liquid.

Based on the results of Jones *et al*^[3-4], the

as-quenched microstructures of RS Al-Fe binary or Al-Fe-Ni(Ce) ternary alloys contained typical eutectic or cellular structure designated as A and B regions. The A region was composed of cellular α -Al subgrains and Al₆Fe or Al₃Ni intermetallics precipitated along subgrain boundaries; the B region corresponded to the eutectic groups of α -Al and hypoeutectic phase. However, there are different microstructural characterization of RS Al-Fe-V-Si alloy, the deviation is mainly attributed to the action of component elements. Since vanadium element has lower diffusion rate in α -Al and it can easily form the peritectic microstructure, the liquidus temperature could increase by adding a small amount of vanadium (< 2%), thus the relative supercooled degree of molten alloy may correspondingly be promoted at the same cooling rate.

Although the function of silicon element preventing the formation of eutectic and primary phase is not distinct, it can cause significant

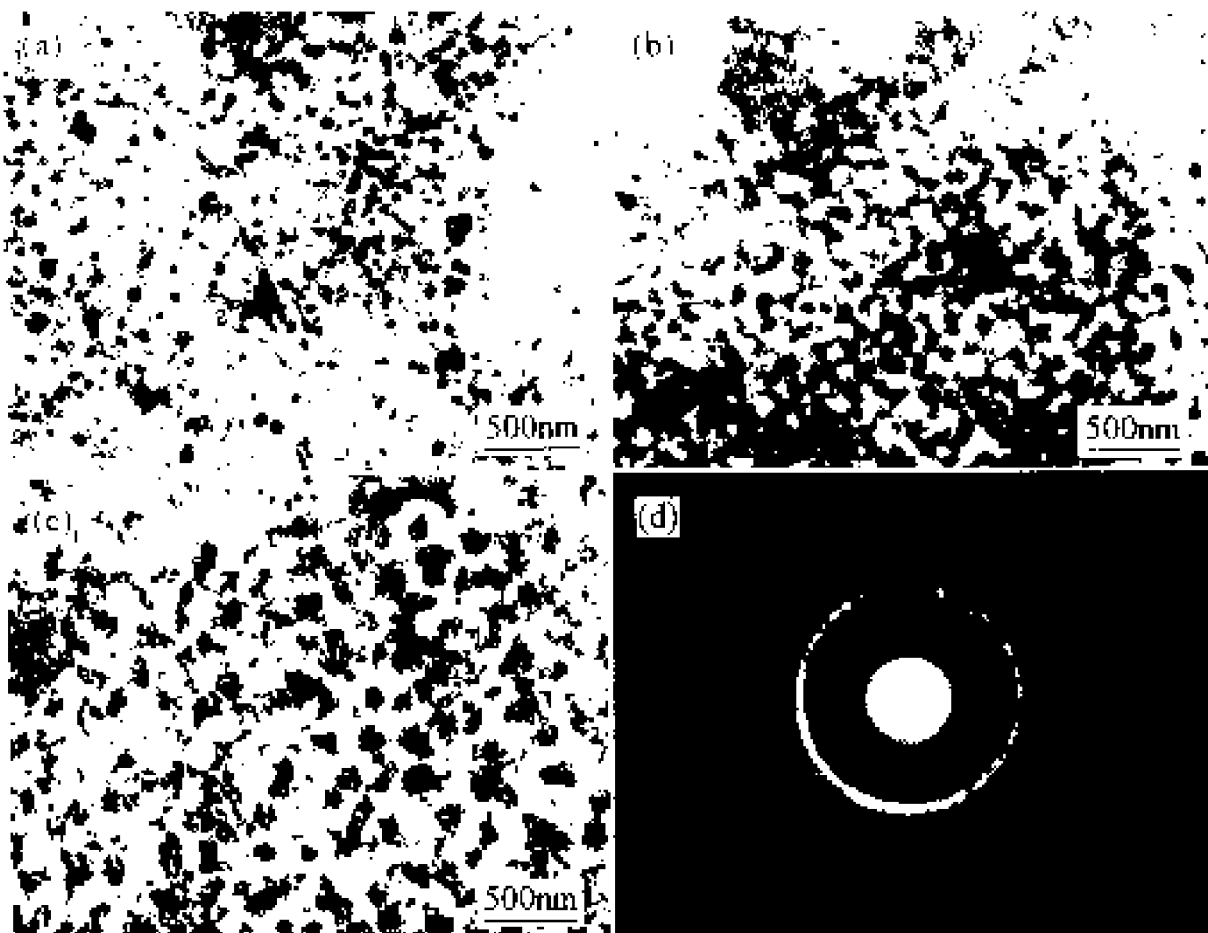


Fig. 1 TEM microstructures of rapidly solidified Al_{97.6-x}Fe_xV_{0.7}Si_{1.7} alloys

(a) $x = 4.3$; (b) $x = 5.4$; (c) $x = 6.4$; (d) —SAED

effect on the component's phase structure and thermal stability^[5]. In addition, the shear viscosity of the molten alloy and the interaction between Fe and Al atoms enhance with the increment of Fe content, which is detrimental to atomic diffusion or cooperative arrangement of several atoms, resulting in a high resistance to nucleation and growth of α -Al and the enhancement of alloy disordering.

The selected area electron diffraction (SAED) (Fig. 1(d)) and X-ray energy spectrum (EDX) studies demonstrated that the multigrain rings appeared and the stoichiometry of dispersive dispersoids was close to α -Al₁₃(Fe, V)₃Si. The as-quenched XRD results of three Al-Fe-V-Si alloys showed the intensity of main peaks corresponding to dispersive particles enhanced with the increase of Fe concentration, as shown in Fig. 2(a) ~ (c). It indicates that the raising tendency of the number of second phase precipitation exists.

In view of the existence of large supersaturation degree in as-quenched state directly influencing the accurate determination of phase structure, typical XRD pattern showing the variation of intensity as a function of 2θ was presented in Fig. 2(d) for the 4.3% Fe alloy aged at 400 °C for 1 h. The results of peak position, intensity, indexing and d -spacing of the various reflections were summarized in Table 1. All the precipitate reflections could be consistently indexed on the basis of a bcc lattice ($h + k + l = 2n$) with $a_0 = 1.260 \pm 0.005$ nm, which closely matches the value of 1.256 nm for the bcc α -AlFeSi compound^[6]. So it is suggested that α -Al₁₃(Fe, V)₃Si particles are the extensive phase of α -AlFeSi compound. Furthermore, it should be noted that stronger reflections appeared in (530), (532) and (600) high indexed planes due to the particular structure factors of complex unit cell having 138 atoms. Recently, detailed calculation of space structure and valence electron structure of α -Al₁₃(Fe, V)₃Si and α -AlFeSi intermetallic compounds indicated that $\langle 532 \rangle$, $\langle 530 \rangle$ and $\langle 600 \rangle$ belong to the strong bond directions and the strongest bond direction $\langle 530 \rangle$ is exactly parallel to the five-fold axis of i -

AlFeSi quasicrystalline phase^[7]. So some extent structural connection exists between α -Al₁₃(Fe, V)₃Si particle and quasicrystalline phase. Above relationship were experimentally proved by the appearance of partly quasicrystalline phase using different RS technologies in Al-Fe-V-Si alloys^[8].

Mössbauer spectroscopy offers a unique opportunity to obtain the nuclear level scale microstructural information by means of the hyperfine interactions between atomic nucleus and local electric-field and magnetic-field surroundings. Due to different phases having various characteristics of Mössbauer spectroscopy, the use of the corresponding hyperfine parameter

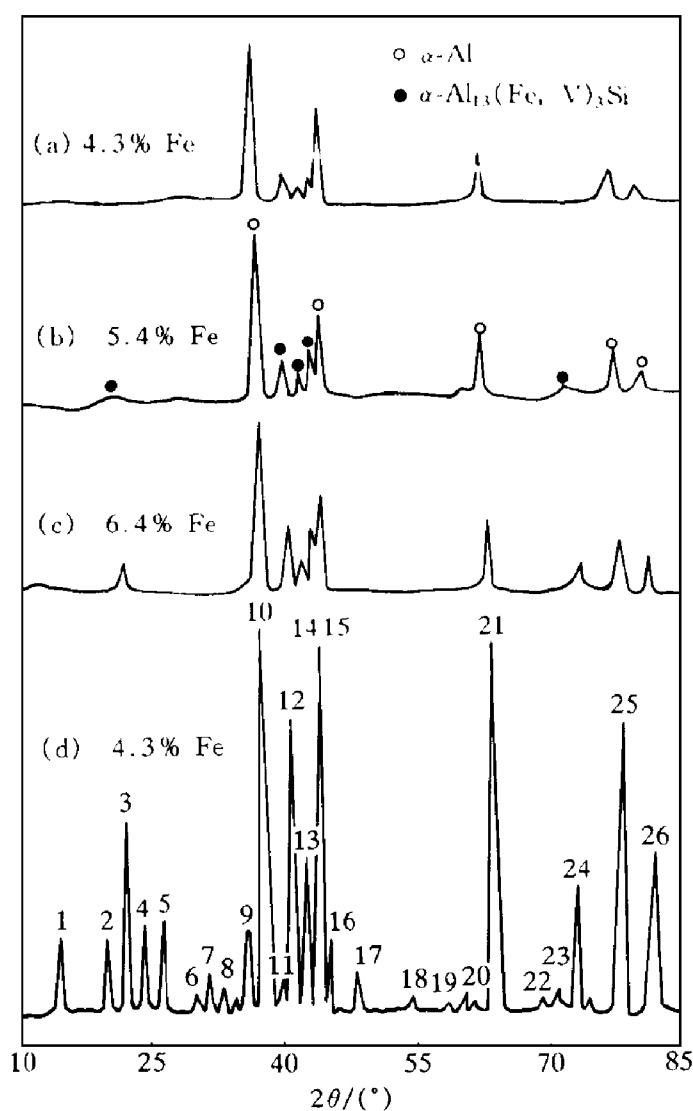


Fig. 2 X-ray diffraction patterns of Al-Fe-V-Si alloys

(a), (b), (c) —as-quenched states;
(d) —aged at 400 °C for 1 h

Table 1 XRD data for $\text{Al}_{93.3}\text{Fe}_{4.3}\text{V}_{0.7}\text{Si}_{1.7}$ alloy ribbon aged at 400 °C for 1 h

No.	$2\theta/(\circ)$	Int/ cps	d /nm	hkl	a_0 /nm
1	14.00	236	0.632	200	1.264
2	20.04	77	0.445	220	1.259
3	22.40	283	0.397	310	1.255
4	24.42	73	0.364	222	1.261
5	26.54	116	0.336	321	1.258
6	30.14	48	0.298	330/ 441	1.264
7	31.86	52	0.281	420	1.257
8	33.48	38	0.269	332	1.262
9	36.50	140	0.247	510/ 431	1.259
10	38.58	10113	0.234	111 -Al	0.405
11	40.68	70	0.223	440	1.261
12	41.94	749	0.216	530/ 433	1.260
13	43.22	308	0.210	600	1.260
14	44.44	1114	0.204	532/ 611	1.258
15	44.84	1796	0.202	200 -Al	0.404
16	45.62	128	0.199	620	1.259
17	48.56	52	0.186	631	1.262
18	55.62	35	0.165	730	1.257
19	61.68	61	0.151	653	1.261
20	62.72	64	0.149	660/ 822	1.261
21	65.20	988	0.143	220 -Al	0.405
22	71.00	44	0.133	754	1.261
23	73.02	41	0.130	763	1.262
24	74.70	240	0.127	770/ 941	1.261
25	78.34	812	0.122	311 -Al	0.405
26	82.52	390	0.117	222 -Al	0.405

* average $a_0 = 1.260 \pm 0.005$ nm

could be effectively utilized in phase analysis and the determination of phase proportion and phase transformation temperature etc. The fitted spectra and hyperfine parameters in Fig. 3 and Table 2 depicted the existence of three subspectra (designated A, B, C) for as-quenched Al-Fe-V-Si alloys, which corresponded to three kinds of Fe

atomic configurations. It has been established that Fe atoms could be located in aluminum solid solution in different structural configurations: substitutional single Fe atoms, or dimer Fe-Fe, interstitial Fe, cluster and associated with vacancies in the grain boundaries due to the existence of supersaturation degree and high defect density in RS alloys. Based on the results of Badan *et al*^[12,13], the singlet (A) and the doublet (B) corresponded to the single Fe and dimer Fe-Fe solid solution, respectively, the doublet (C) was caused by $\alpha\text{-Al}_{13}(\text{Fe}, \text{V})_3\text{Si}$ particles having the similar spectra parameters to Al_6Fe phase.

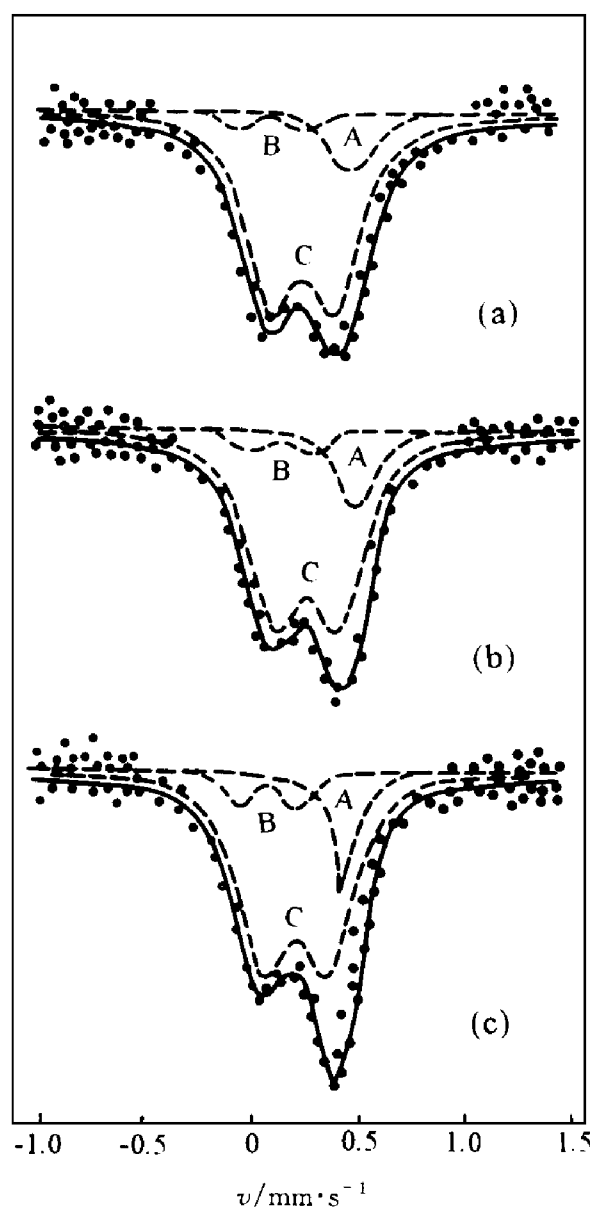


Fig. 3 Mössbauer spectra of as-quenched $\text{Al}_{97.6-x}\text{Fe}_x\text{V}_{0.7}\text{Si}_{1.7}$ alloys

(a) $-x = 4.3$; (b) $-x = 5.4$; (c) $-x = 6.4$

According to the fitted subspectra area the relative proportion of every Fe atomic configuration could be determined, simultaneously, the quantitative number of Fe atom forming solid solution and intermetallic compound could also be obtained. The calculation results of subspectra area were listed in Table 2.

Table 2 Mössbauer spectra parameters of as-quenched Al-Fe-V-Si alloys

Alloys / %	Spectra composition	Isomer shift $\delta / \text{mm} \cdot \text{s}^{-1}$	Quadrupole split $\Delta / \text{mm} \cdot \text{s}^{-1}$	Area fraction / %
Al _{93.3} Fe _{4.3} V _{0.7} Si _{1.7}	singlet(A)	0.4487	-	12.612
	doublet(B)	0.0912	0.2985	5.835
	doublet(C)	0.2177	0.3065	81.553
Al _{92.2} Fe _{5.4} V _{0.7} Si _{1.7}	singlet(A)	0.4495	-	12.934
	doublet(B)	0.0921	0.2987	6.095
	doublet(C)	0.2181	0.3069	80.971
Al _{91.2} Fe _{6.4} V _{0.7} Si _{1.7}	singlet(A)	0.4512	-	13.302
	doublet(B)	0.0928	0.2990	6.314
	doublet(C)	0.2189	0.3072	80.384

shows that the Fe atomic contents are 0.79%, 1.03% and 1.26% in α -Al solid solution (subspectra A + B) and 3.51%, 4.37% and 5.14% in α -Al₁₃(Fe, V)₃Si intermetallic compound (subspectra C) for Al_{97.6-x}Fe_xV_{0.7}Si_{1.7} ($x = 4.3\%$, 5.4% , 6.4%) alloys, respectively. In addition, the ratio of 3.51% to 5.14% is close to that of 24% to 37% (in volume) of dispersive intermetallic particles in Al-Fe-V-Si alloys reported by Skinner *et al.*^[10]. From the results of Mössbauer spectroscopy, it could be obtained that the numbers of α -Al(Fe), α -Al(Fe-Fe) substitutional solid solution and dispersive α -Al₁₃(Fe, V)₃Si particles formation enhanced with the increase of Fe concentration. However, the values of matrix lattice parameter demonstrate the declining tendency, which mainly results from the increase of Fe content in α -Al substitutional solid solution.

4 CONCLUSIONS

(1) The as-quenched microstructures of RS Al-Fe-V-Si alloys are composed of the coexistence of microcellular α -Al and dispersive nanoscale α -Al₁₃(Fe, V)₃Si particles. The particle finning and conglomeration exists with the increase of Fe concentration.

(2) The α -Al₁₃(Fe, V)₃Si particle belongs to bcc structure, the lattice parameter a_0 is about 1.260 nm, it is the extensive phase of α -AlFeSi compound.

(3) The results of Mössbauer spectroscopy show that there are three kinds of Fe atomic configurations corresponding to α -Al(Fe), α -Al(Fe-Fe) substitutional solid solution and α -Al₁₃(Fe, V)₃Si intermetallic compound.

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