ScienceAsia 30 (2004): 25-31

Microstructure-Processing-Property-Relationship of Rapidly Solidified Al-Fe-(V, Si) Alloys

Ruangdaj Tongsri,[a,*] Jane Minay,[b] Richard Thackray,[b] Richard Dashwood[b] and Henry Mcshane[b]

[a] National Metal and Materials Technology Center (MTEC), Thailand Science Park, 114 Paholyothin Rd., Klong 1, Klong Luang, Patumthani 12120 Thailand.

[b] Department of Materials, Imperial College of Science, Technology and Medicine, Prince Consort Rd., London SW7 2 BP, UK.

* Corresponding author, E-mail: ruangdt@mtec.or.th

Received 23 May 2003
Accepted 24 Dec 2003

ABSTRACT: Gas-atomised powders of Al-Fe-(V, Si) alloys exhibited microstructures consisting of several forms of icosahedral phase distributed in either supersaturated solid solution of α-Al matrix, or in intercellular/dendritic regions depending on powder particle size. During processing of the powders by hot extrusion, the icosahedral phase particles transformed to more stable phase particles. Microstructures of extrudates produced from fine Al-Fe-V powder particles showed homogeneous precipitation of ultrafine needle-like particles in grains and at grain boundaries. This type of microstructure yielded high compressive strengths at room and elevated temperatures. In microstructures of extrudates produced from coarse Al-Fe-V powder particles, ultrafine needle-like particles were observed to coexist with globular particles. Because of large-size globular particles, low compressive strengths at room and elevated temperatures of the extrudates produced from coarse Al-Fe-V powder particles were obtained. The extrudates produced from fine Al-Fe-V-Si powder particles showed microstructures with homogeneous precipitation of spherical-like particles. In contrast, the extrudates produced from coarse Al-Fe-V-Si powder particles resulted in inhomogeneous microstructures, which were attributed to banded structures or donut-shaped aggregates of spherical-like particles. Because of inhomogeneous microstructures, low compressive strengths of the extrudates at room and elevated temperatures were obtained.

Keywords: microstructure, processing, property, rapidly solidified Al-Fe-(V, Si) alloys.

INTRODUCTION

There has been growing interest in the development of elevated temperature Al-Fe alloy systems[1-6] since the pioneer discovery of rapidly solidified Al-Fe-V alloys using the melt-spinning technique.[7,8] These aluminium-iron alloy systems are potential candidates for production of lightweight alloys with useful mechanical properties at elevated temperatures. Although the melt-spun Al-Fe-(V, Si) alloys exhibited some promising mechanical properties, production of engineering parts from these materials could face many processing steps.Useful mechanical properties of the as-solidified materials might be degraded during multi-step processing. Recently, there has been an idea to reduce production steps of the elevated temperature Al-Fe-(V, Si) alloys employing a simple production route including powder production using inert-gas atomisation and powder consolidation using hot extrusion.[9] The short processing route will not only provide opportunity to maintain as much as possible the mechanical properties of the as-solidified materials during processing, but also keep the total production cost down. Relationship between microstructure, processing and properties needs to be investigated prior to production of commercial engineering parts from gas-atomised Al-6.5Fe-1.5V (Al-Fe-V) and Al-6.5Fe-1.5V-1.7Si (Al-Fe-V-Si) alloy powders. Microstructures of the gas-atomised Al-Fe-(V, Si) powders and their extrudates were carefully observed. Compressive strengths of the extrudates produced from these alloy powders at room and elevated temperatures were also determined.

MATERIALS AND METHODS

Size fractions of gas-atomised Al-Fe-(V, Si) alloy powders, were supplied by ALPOCO, UK. Microstructural observation was carried out using transmission electron microscopy (TEM) equipped with energy dispersive X-ray spectroscopy (EDS)(JEOL-FX 2000 EM, JEOL, Tokyo, Japan). Hot extrusion was performed for each size fractions using an ENEFCO 5 MN hydraulic vertical press acting in the direct mode. A compact made from each size fractions and extrusion tooling were preheated to 500°C and 450°C, respectively. Extrusion of the powder compact was then performed with an extrusion ratio of 18:1 and a ram speed of 3 mm/s.

Procedures of TEM specimen preparation for the powder particles and the extrudates were given as follows.[9] For the powders, preparation steps were followed. In the first step, the powders were embedded in a nickel foil. This could be done by placing the powders on a steel cathode and immersing in a nickel ion-containing electrolyte of an electrolysis unit. With appropriate electric potential and current, nickel ion would be reduced and deposited on the cathode surface and hence the powders were embedded in the deposited nickel. Then, the embedded nickel foil was ground until its thickness was less than 100 mm. In the next step, a disc with diameter of 3 mm was made from the thin foil. Finally, the disc was thinned electrolytically using a Struers Tenupol jet polishing machine, with a solution (by volume) of 10% percholic acid, 10% 2-butoxyethanol, and 80% ethanol, operating at –35°C and 65 V. For the extrudate, a disc with diameter of 3 mm was made from the extrudate. It was finally thinned using a Struers Tenupol jet polishing machine, with a solution (by volume) of 30% nitric acid and 70% methanol, operating at –30°C and 20 V.

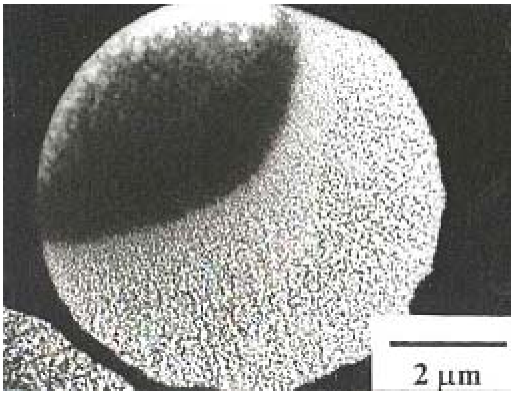
Compressive tests were performed on short rod specimens, with diameters of 8 mm and heights of 10 mm, machined from the extrudates. The tests were performed with 30% total strain, at a constant strain rate of 0.001 s^{-1} and at temperatures of 25°C and 250 °C, using a MAYES machine equipped with a Severn furnace (SFL model RHS 1259).

Results and Discussion

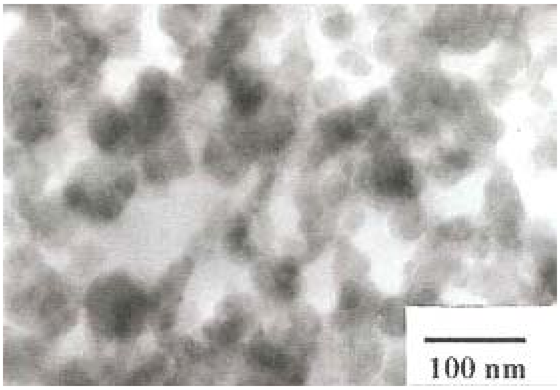
Microstructure of Powder Particles

Fine powder particles, with diameters <15 μm, of Al-Fe-V and Al-Fe-V-Si alloys exhibited two types of microstructures (dark area on the left-hand side of the powder particle in Fig 1a). The first consisted of irregular-shaped aggregates of ultrafine-spherical intermetallic particles distributed homogeneously in supersaturated solid solution α-Al phase matrix (light area on the right-hand side of the powder particle in Fig 1b). The second consisted of ultrafine continuous cellular networks (Figs 3a and 4a). Identification of the ultrafine-spherical intermetallic particles in the aggregates and the intercellular particles revealed that they could be microquasi-crystalline icosahedral (MI) phases.[9] The MI phase particles have been previously observed in a melt-spun Al-Fe-Mo-V ribbon.[10] The MI phase was also called a 'special amorphous phase' by several authors.[11-13] Although the special amorphous phase exhibited a selected area diffraction pattern (SADP) with diffuse rings, it was not a normal metallic glass (dense random packing model).

Coarse powder particles with diameters >15 mm exhibited two types of microstructures. The first consisted of ultrafine continuous cellular networks near nucleation sites (dark areas in the powder particle in Fig 2). The ultrafine cellular networks with high



a Al-Fe-V



b Irregular-shaped aggregates

Fig 1. Microstructures of a fine powder particle of Al-Fe-V alloy.

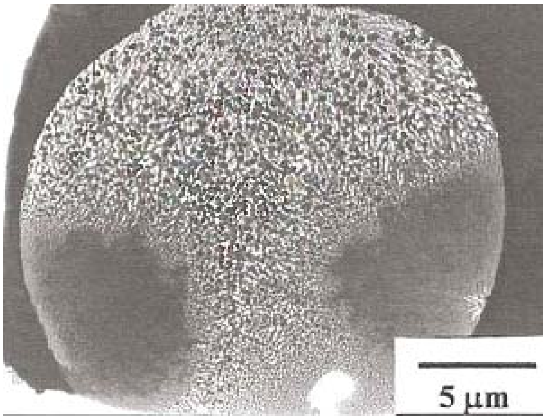
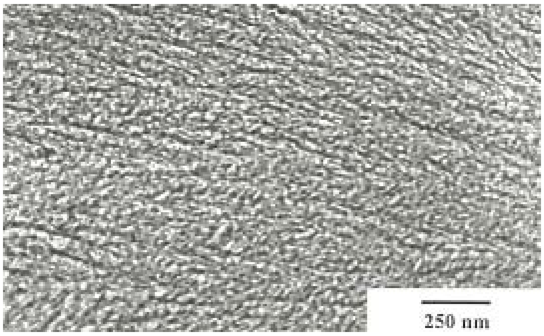
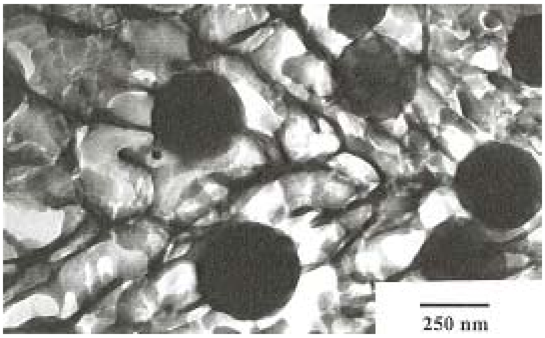


Fig 2. Microstructure of a coarse Al-Fe-V powder particle.

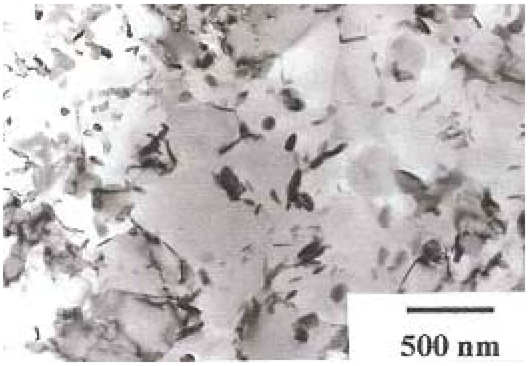
magnification are shown in Figs 3a and 4a. The second consisted of coarse continuous cellular networks and globular particles (light area with cells and spots in the powder particle in Fig 2). These microstructures, consisting of coarse cellular networks and globular particles with high magnification, are shown in Figs 3b and 4b. The globular particles exhibited different SADPs, depending on alloy composition.[9] The globular particles in coarse Al-Fe-V powder particles showed SADPs with diffuse rings so they were speculated to be globular



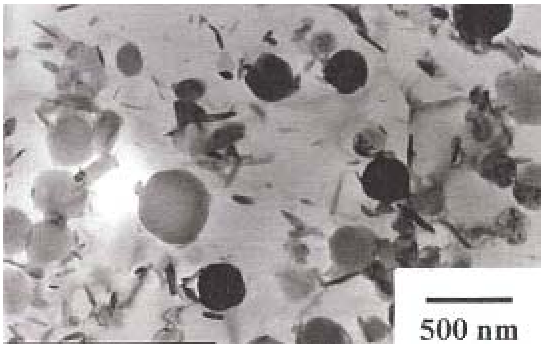
a Ultrafine cellular networks.



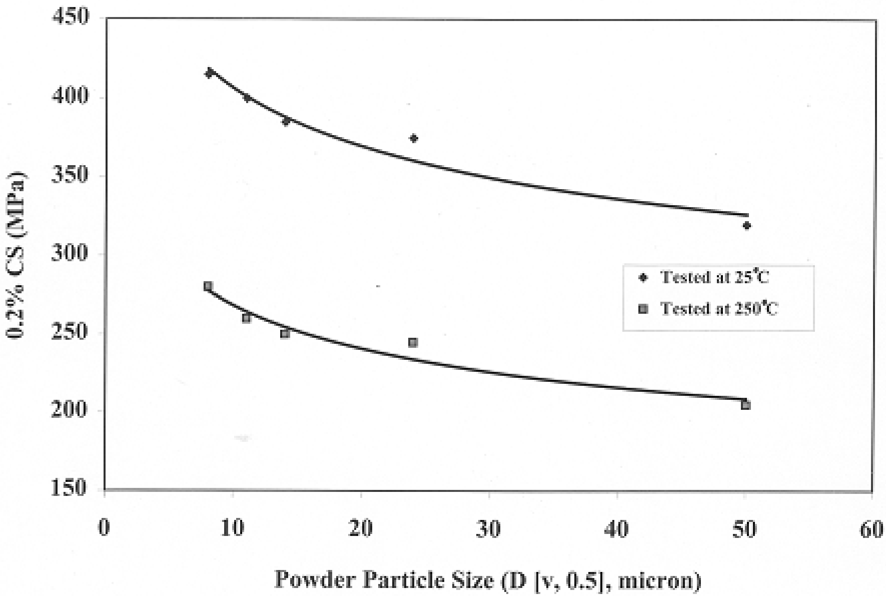
b Coarse cells and globular particles.



c Microstructure of the extrudate produced from fine powder particles.



d Microstructure of the extrudate produced from coarse powder particles.



e Compressive strengths of the extrudates

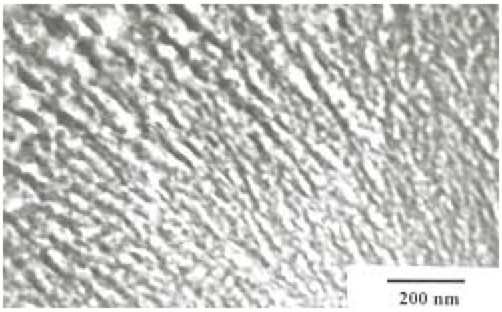
Fig 3. Microstructure-processing-mechanical property-relationship of Al-Fe-V alloy.

clusters of MI phase. In coarse powder particles of Al-Fe-V-Si alloy, the globular particles exhibited SADPs with arrays of diffraction spots, which did not possess a long-range periodic translation order, but showed a crystallographically forbidden rotational fivefold symmetry. This indicates that the globular particles in coarse Al-Fe-V-Si alloy powders are particles of single icosahedral phase.

Microstructure of the Extrudate

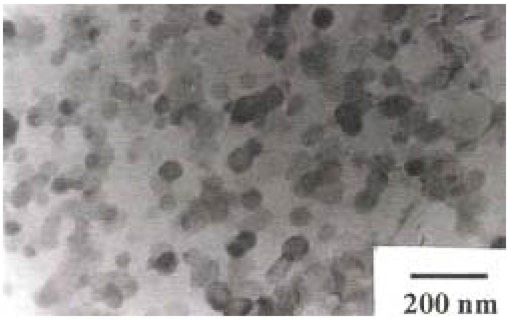
Al-Fe-V alloy

The extrudates produced from fine Al-Fe-V powders exhibited microstructures consisting of fine needle-like particles with high aspect ratios (the ratio of length to width) distributed homogeneously in grains and at grain boundaries (Fig 3(c)). The needle-like particles in the extrudate were found to be either $Al_{13}Fe_4$,

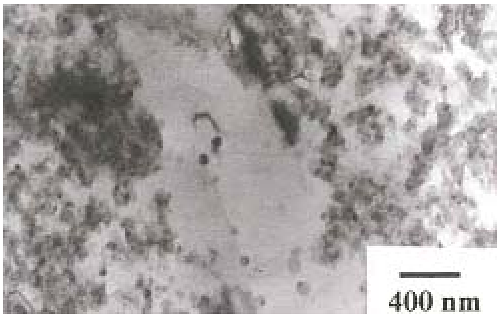


a Ultrafine cellular networks.

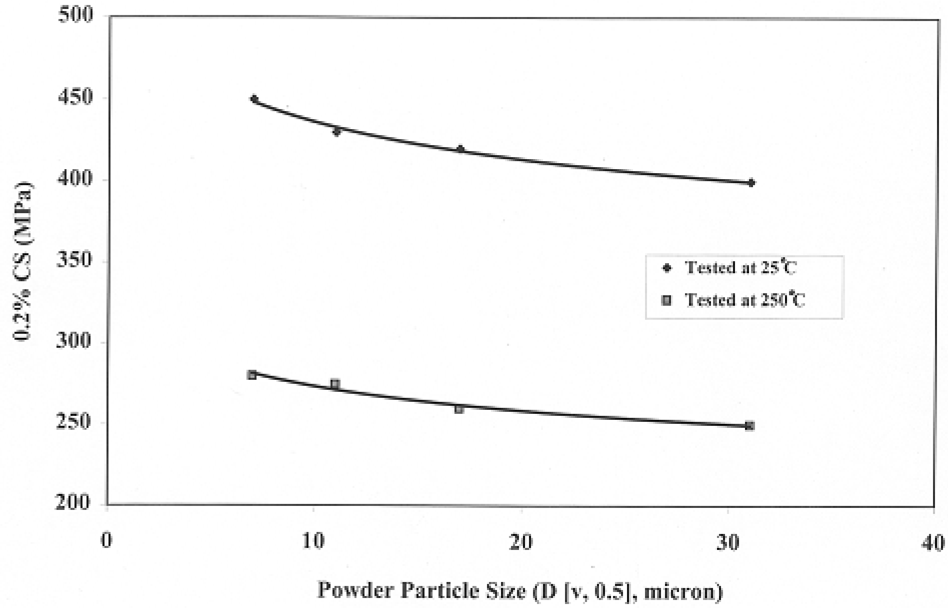
b Coarse cells and globular particles.



c Microstructure of the extrudate produced from fine powder particles



d Microstructure of the extrudate produced from coarse powder particles



e Compressive strengths of the extrudates.

Fig 4. Microstructure-processing-mechanical property-relationship of Al-Fe-V-Si alloy.

$Al_{45}(Fe, V)_7$, or unknown phases.[9] They were hardly observed in the original powder microstructures in the extrudates. It may be implied that the irregular-shaped aggregates and the continuous cellular networks are decomposed to form needle-like particles during hot extrusion. This may also indicate that the MI phase in powder microstructures is metastable. Because of ultrafine microstructures in alloy powders, homogeneous distribution of ultrafine precipitates in the extrudates produced from the powders is obtained.

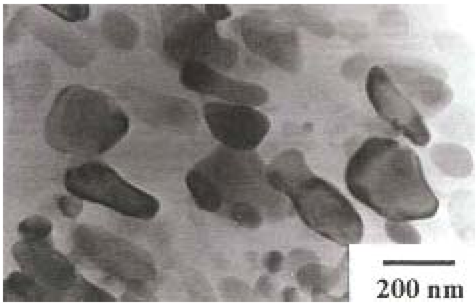
The extrudates produced from coarse Al-Fe-V powder particles exhibited different microstructures from those of the extrudates produced from fine powder particles. Fig 3d illustrates that globular particles are coexisting with ultrafine needle-like particles. This indicates that during hot extrusion the continuous cellular networks are broken up but the shape and size of globular particles are not affected. However, the globular particles in the extrudates, produced from coarse Al-Fe-V powder particles, exhibited SADPs with arrays of diffraction spots, which did not possess a long-range periodic translation order, but showed a crystallographically forbidden rotational fivefold symmetry.[9] This indicates that the globular particles are definitely single icosahedral phase. It may be implied that a change from globular clusters of MI phases to single icosahedral phase particles, occurring during hot extrusion, is a polymorphous transformation.

Al-Fe-V-Si alloy

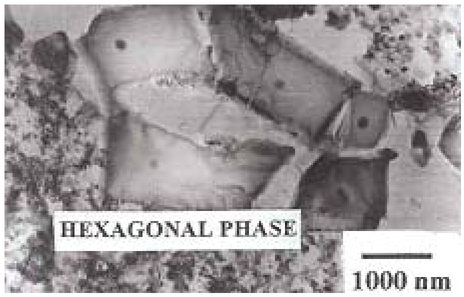
The extrudates produced from fine Al-Fe-V-Si powder particles exhibited microstructures consisting of ultrafine spherical-like particles distributed homogeneously throughout the α-Al matrix (Fig 4 c). The spherical-like particles have been previously observed in other rapidly solidified Al-Fe-V-Si alloys.[1-6] The particles in the extrudates could be one of the body-centered cubic (bcc) silicide phases, namely, α_{13}-AlFeVSi (with composition of $Al_{13.37}(Fe, V)_{3.0}Si_{1.11}$) and α_{12}-AlFeVSi (with composition of $Al_{12.86}(Fe, V)_{3.0}Si_{2.1}$).[3] The α_{13}-AlFeVSi and α_{12}-AlFeVSi have structures designated as bcc (Im3) and bcc (Pm3), respectively. Chemical analysis of the spherical-like (silicide) particles in the extrudates, by using EDS, revealed that their composition was $Al_{13}(Fe, V)_3Si$.[9] Precipitation of the silicide phase may have resulted from transformations of MI particles in the irregular-shaped aggregates and in the continuous cellular networks during hot extrusion. Homogeneous distribution of stable silicide particles, resulted from ultrafine powder microstructures, is expected to provide a dispersion strengthening effect at high temperatures, because this phase exhibits high resistance to particle coarsening.[5]

It was observed that microstructures of the extrudates produced from coarse Al-Fe-V-Si powder particles exhibited banded structures. The banded structures consisted of a band with a high volume fraction of ultrafine silicide particles parallel to a band with a low volume fraction of coarse silicide particles (Fig 4d). In some banded structures, coarse elongated silicide particles (Fig 5a), large plate-like particles (Fig 5b), and doughnut-shaped precipitates of ultrafine silicide particles (Fig 5c), were also observed. The plate-like particles were identified as hexagonal phase.[9]

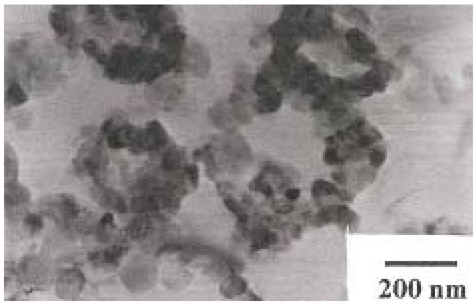
Formation of elongated silicide particles is probably attributed to either disintegration of coarse continuous cellular networks or decomposition of globular single icosahedral particles. However, decomposition of the globular particles were observed to yield doughnut-shaped precipitates of fine silicide particles, with the core being aluminium.[6] Therefore the possible reason for formation of elongated silicide particles is disintegration of coarse continuous cellular networks.



a Coarse elongated silicide particles

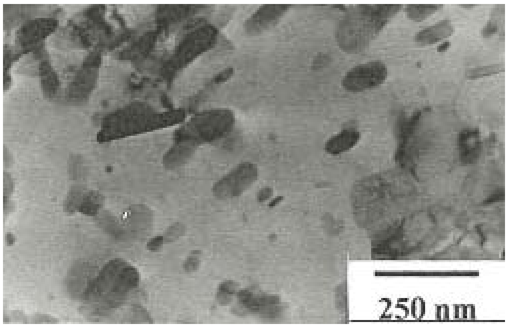


b Plate-like particles

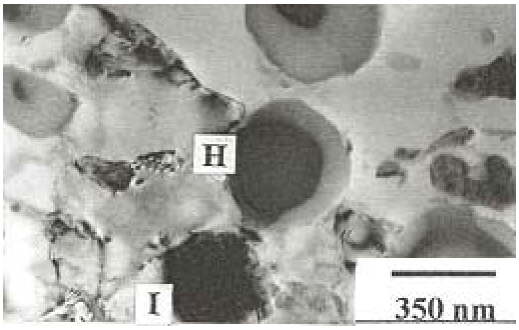


c Doughnut-shaped aggregates of ultrafine silicide particles

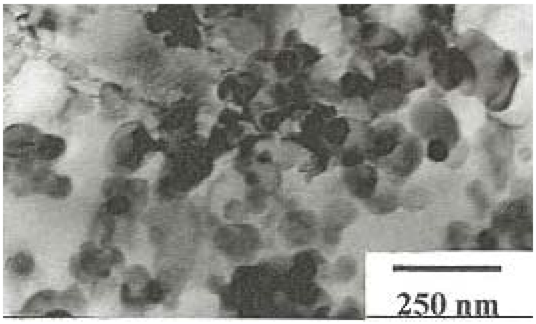
Fig 5. Microstructures of extrudates produced from coarse Al-Fe-V-Si powder particles.



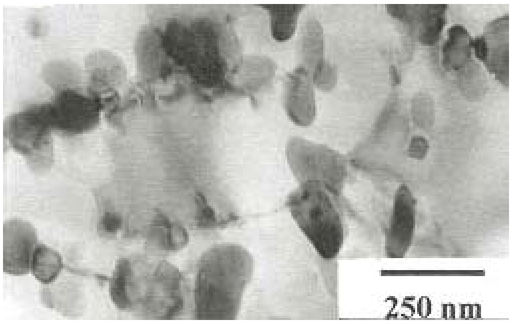
a Al-Fe-V alloy



b Al-Fe-V alloy



c Al-Fe-V-Si alloy



d Al-Fe-V-Si alloy

Fig 6. Microstructures of the extrudates compressed at 250 °C with 30% total strain at a constant strain rate of 0.001 s^{-1}.

After disintegration, the discrete particles may not be able to complete spherodisation due to constraints, such as slow dissolution of the particles and slow solute diffusion in the matrix.

Formation of large plate-like particles (hexagonal phase) may occur during solidification of coarse Al-Fe-V-Si alloy powders due to slow cooling rates, which in turn cause a low degree of undercooling. Transformation of the globular single icosahedral particles to large plate-like particles is irrational because of the following reasons. First, all forms of icosahedral phase in melt-spun Al-Fe-V-Si alloy transformed directly to silicide phase particles,[6] so no intermediate phases were observed. Second, the hexagonal phase was observed to form directly from the melt on the air-side of the Al-Fe-V-Si alloy strips, which experienced a low degree of undercooling.[14]

Compressive strength (CS) of the extrudate

Extrudates produced from fine Al-Fe-V and Al-Fe-V-Si powder particles exhibited high compressive strengths at room temperature (Figs 3e and 4e). This promising mechanical property of the extrudates may be attributed to homogeneous distribution of ultrafine needle-like particles in Al-Fe-V alloy and ultrafine spherical-like particles in Al-Fe-V-Si alloy. In contrast, the extrudates produced from coarse Al-Fe-V and Al-Fe-V-Si powder particles showed lower compressive strengths at room temperature. The inferior mechanical properties of the extrudates produced from coarse Al-Fe-V and Al-Fe-V-Si powder particles may be attributed to precipitation of large globular icosahedral particles for Al-Fe-V alloy and to inhomogeneous banded-structures for Al-Fe-V-Si alloy.

When the extrudates were compressed at elevated temperatures, a decrease in of strengths of the extrudates was clearly observed (Figs 3e and 4e). The decrease of strengths may be attributed to reduction of materials yield strength with increasing temperatures, associated with precipitate coarsening and phase changes at elevated temperatures. Fig 6 shows evidences of particle coarsening and phase transformation during elevated temperature compressive testing. Needle-like particles of $Al_{13}Fe_4$, $Al_{45}(Fe, V)_7$ and unknown phases were transformed to rod-like particles with lower aspect ratios (Fig 6a). The globular single icosahedral (I) phase particles were transformed to globular crystalline hexagonal (H) phase particles (Fig 6b). Compact aggregates of coarse silicide particles were observed in microstructures of the extrudates produced from Al-Fe-V-Si alloy powders compressed at elevated temperatures (Fig 6c). In the banded structure with low volume fraction of coarse elongated silicide particles, evidence of particle coarsening was also noticed (Fig 6d).

It is worth noting the coarsening of the silicide phase particles. Although the silicide phase particles have been reported to be thermally stable due to very slow coarsening rates of between 2.9×10^{-6} and 8.4×10^{-27} m^3/h at 425°C,[1,5] some evidence of particle coarsening was observed (Figs 6c and d). The coarsening of these particles may be assisted by two parameters. The first is pressure, which mechanically compresses or brings the silicide particles to be closer. The second is heat, which thermally activates particle dissolution and diffusion of constituent elements from the dissolved particles or from the matrix to the growing particles. When a distance between particles is shorter and diffusion of solute elements is activated, the particle coarsening may be accelerated.

Conclusions

Microstructures of Al-Fe-V and Al-Fe-V-Si alloy powders were influenced by powder particle size. Ultrafine microstructures were observed in fine powder particles, whereas coarse and inhomogeneous microstructures were observed in coarse powder particles. Homogeneous distribution of precipitates in the extrudates, which exhibit high compressive strengths at room temperature, can be produced from powder particles with fine microstructures. In contrast, the extrudates of coarse powder particles exhibited inhomogeneous distribution of precipitates with different shapes and sizes. Inferior compressive strengths at room temperature of the extrudates of coarse powder particles may be attributed to this inhomogeneous distribution and size of the precipitates. Precipitation hardening became less effective as the temperatures were elevated due to softening of the materials and precipitate coarsening with increasing temperatures.

Acknowledgements

The authors would like to thank the Department of Materials, Imperial College of Science, Technology and Medicine, UK., the Brite Euram Project, and the Royal Thai Government.

References

1. Gilman PS and Das SK (1988) Rapidly Solidified Aluminum Alloys for High Temperature/High Stiffness Applications: In: Proceedings of the international Conference on PM Aerospace Materials (A metal powder report conference, Luzern, November 2-4, 1987), MPR Publishing Services, Shrewsbury, 27.1-27.12.
2. Gilman P (1990) Rapidly Solidified Aluminium Alloys for Aerospace. *Metals and Materials* **6**, 504-7.
3. Rodriguez MA and Skinner DJ (1990) Compositional Analysis of the Cubic Silicide Intermetallics in Dispersion Strengthened Al-Fe-V-Si Alloys. *J Mater Sci Lett* **9**, 1292-3.
4. Kim NJ (1991) Analytical Electron Microscopy Studies of Silicide Phase in a Rapidly Solidified Al-8.1Fe-1.8V-0.7Si Alloy. *Int J Rapid Solidification* **6**, 175-84.
5. Wilkes DMJ and Jones H (1995) A Perspective on the High Resistance to Coarsening and Durability of Silicide Dispersions in RSPM-Al-Fe-V-Si Alloys. In: Science and Technology of Rapid Solidification Processing. (Otooni, M. A., ed.), Kluwer Academic Publishers, Netherlands, pp. 157-71.
6. Park WJ, Ahn S and Kim NJ (1994) Evolution of Microstructure in a Rapidly Solidified Al-Fe-V-Si Alloy. *Mat Sci Eng* **A189**, 291-9.
7. Skinner DJ and Okazaki K (1984) High Strength Al-Fe-V Alloys at Elevated Temperatures Produced by Rapid Quenching from the Melt. *Scripta Metall* **18**, 905-9.
8. Skinner DJ, Okazaki K and Adam CM (1986) Physical Metallurgy and Mechanical Properties of Aluminium Alloys Containing Eight to Twelve Weight Percent Iron. In: Rapidly Solidified Powder Aluminum Alloys. (Fine ME and Starke Jr EA, Eds.) ASTM-STP890, Philadelphia, PA, 211-36.
9. Tongsri R (2000) Microstructural development during processing of rapidly solidified Al-Fe-(V, Si) alloys, Ph.D. Thesis, University of London.
10. Field RD, Zindel JW and Fraser HL (1986) The Intercellular Phase in Rapidly Solidified Alloys based on the Al-Fe System. *Scripta Metall* **20**, 415-8.
11. Bendersky LA and Ridder S D (1986) Nucleation Behavior of Al-Mn Icosahedral Phase. *J Mater Res* **1(3)**, 405-14.
12. Bendersky LA, Biancaniello FS and Schaefer RJ (1987) Amorphous Phase Formation in $Al_{70}Si_{17}Fe_{13}$ Alloy. *J Mater Res* **2(4)**, 427-30.
13. Bendersky LA, McAlister AJ and Biancaniello FS (1988) Phase Transformation during Annealing of Rapidly Solidified Al-Rich Al-Fe-Si Alloys. *Metall Trans A* **19A**, 2893-900.
14. Koh HJ, Park WJ and Kim NJ (1998) Identification of Metastable Phases in Strip-cast and Spray-cast Al-Fe-V-Si Alloys. *Mater Trans JIM* **39**, 982-8.