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Study on the microstructure Evolution of Spray Deposited Al – Fe – V – Si Alloy

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Abstract: Microstructure characterization of the as – received and hot – extruded spray deposited Al – 8Fe – 1.3V – 1.7Si alloys were comprehensively done by optical and transmission electron microscopy. The results show that densely fine silicide particles uniformly distribute in the α – Al matrix and remain stable after hot extrusion at high temperature. Some h – Al_8Fe_2Si clump particles appear after hot extrusion at 300 ℃ and above, which was transformed from b.c.c α – $Al_{13}Fe_3Si$ phase. The mechanical properties of as – extruded alloys decrease as the extrusion temperature increases.

Key words: spray deposition; microstructure; Al – Fe – V – Si alloy; mechanical property

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Introduction

In recent years, there has been a large amount of researches on the rapidly solidified (RS) Al – Fe based alloys for possible application in aerospace structure. Among these alloys, Al – Fe – V – Si alloys have been shown to have the best combinations of room temperature and elevated – temperature mechanical properties, which make these alloys quite suitable for applications at elevated – temperature up to 350 ℃ and comparable with those of Ti alloys on a specific basis[1~3]. Good elevated – temperature mechanical properties of rapidly solidified Al – Fe – V – Si alloys are attributed to the high volume fraction (24% ~ 37%) of nanoscale α – $Al_{13}(Fe,V)_3Si$ dispersoid, which has very low coarsening rates (10^{-27} m^3h^{-1}) compared to other metastable or equilibrium phases in Al – Fe – X alloys. The Al – Fe – V – Si alloy processed by the planar flow casting or inert gas atomization process requires further consolidation by hot rolling or hot extrusion methods. Alternatively, the spray deposition process can produce near – net shape components employing a minimum number of processing steps. In addition, rapid solidification inherent in spray deposition process can provides alloys with fine – grained microstructure, increased solid solubility, nonequilibrium phases, and ultrafine dispersion of second – phase particles[4,5].

This work aims at studying the structure characteristics and mechanical properties of as – deposited and as – extruded Al – Fe – V – Si alloys, providing theoretical basis for innovative alloy design.

1 Experimental Details

Alloys of nominal composition Al – 8Fe – 1.3V – 1.7Si (wt%) were produced by the spray deposition process in the general research institute of non – ferrous metals. The processing apparatus consists of an induction – heated graphite crucible, a converging gas atomization nozzle and a copper substrate. The alloy charge was melted in the graphite crucible and superheated to the desired temperature using induction heating. After holding for 30 mins, the liquid melt was allowed to flow as a fine stream through the exit hole of the crucible. Using N_2 as atomizing gas, the melt stream was

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atomized into fine droplets and the droplets were deposited directly onto a copper substrate to produce 14 – mm – thick deposit. The deposit was hot – extruded at 16 :1 ratio at 150 ℃, 200 ℃, 250 ℃, 300 ℃, 350 ℃ respectively. Samples machined from the deposit were polished following standard metallographic procedures for optical microscopy (OM) observation. A dilute Keller's reagent was used to etch the sample. The TEM samples of spray deposit foil were prepared by electro – polishing using 80 pct ethanol and 20 pct perchloric acid solution at – 30 ℃ and ion beam milling. TEM samples were examined in a JEOL – 2000FX transmission electron microscope operated at 160 kV, which was equipped with a OXFORD Link – ISIS EDXS system.

2 Results and Discussion

2.1 OM observation

Fig.1 shows the striking microstructures of the deposit, which exhibit a uniform dispersion of second – phase particles in the matrix. The ultrafine particles were located both in the α – Al grain and along the boundaries. The above microstructure in spray deposited materials, generally referred throughout the scientific literature as 'equiaxed', is typified by the presence of non – dendritic, spheroidal grains. The formation of such a morphology during spray deposition is attributed to various factors. The continuous impact of partially solidified droplets increases heterogeneous nucleation, mechanical fracture, remelting of dendrites and grain multiplication, which cause the solids grow into the liquid droplets to produce a fine 'equiaxed' grain structure[5,6].

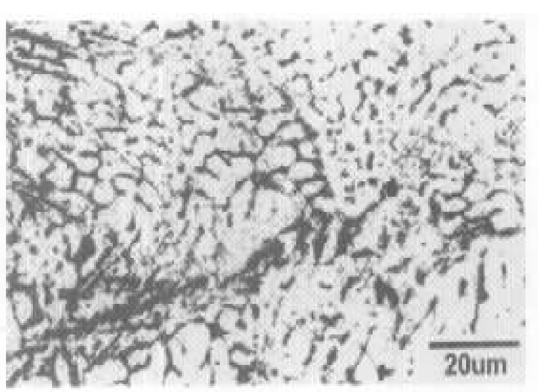


Fig.1 OM micrograph of spray deposit

2.2 TEM observation of as – deposited alloy

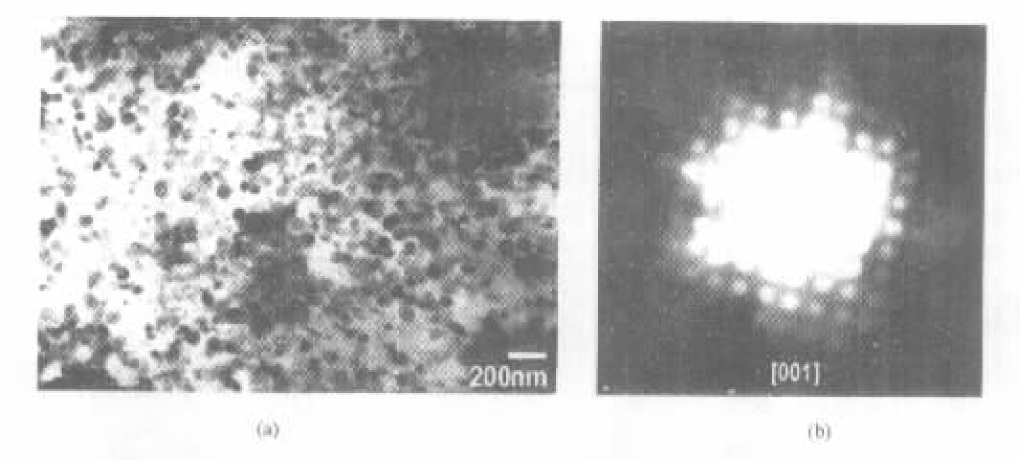
From Fig.2(a), the as – deposited microstructure can be, in general, characterized by a high density of nearly spherical particles in the α – Al matrix. The spherical particles range from 10 ~ 80 nm in an individual single particle. In the present case, a large undercooling is necessary to promote the high nucleation frequency and low growth rate to produce the fine particles. TEM analysis revealed that the microcellular – Al matrix and the extremely fine particles have no orientation relationship. The reason for the formation of these randomly oriented intermetallic compounds could be explained more reasonably by the dispersoid formed prior to the α – Al cell, or as a primary phase.

Closer examination of these fine spherical particles by μ – D (Fig.2(b)) combined with EDXS analysis revealed that it was cubic silicide phase. EDXS result shows that the elements content in the silicide phase is 80.33at% Al, 12.37at% Fe, 2.30at% V and 5.00at% Si respectively. The silicide strengthening phase in the rapidly solidified Al – Fe – V – Si alloy is a complex cubic α – $Al_{13}(Fe,V)_3Si$ phase (138 atoms per unit cell; $a = 1.256$ nm) based on the metastable α – $Al_{13}Fe_3Si$ phase[7]. In Al – Fe – Si ternary alloys the metastable α – $Al_{13}Fe_3Si$ phase would normally revert to the equilibrium Al_8Fe_2Si or $Al_{13}Fe_4$ phase during heat treatment, but is stabilized by the addition of the b.c.c transition metal elements V, Mo, Cr, Mn or W.

2.3 Microstructure of hot – extruded alloys at different temperature

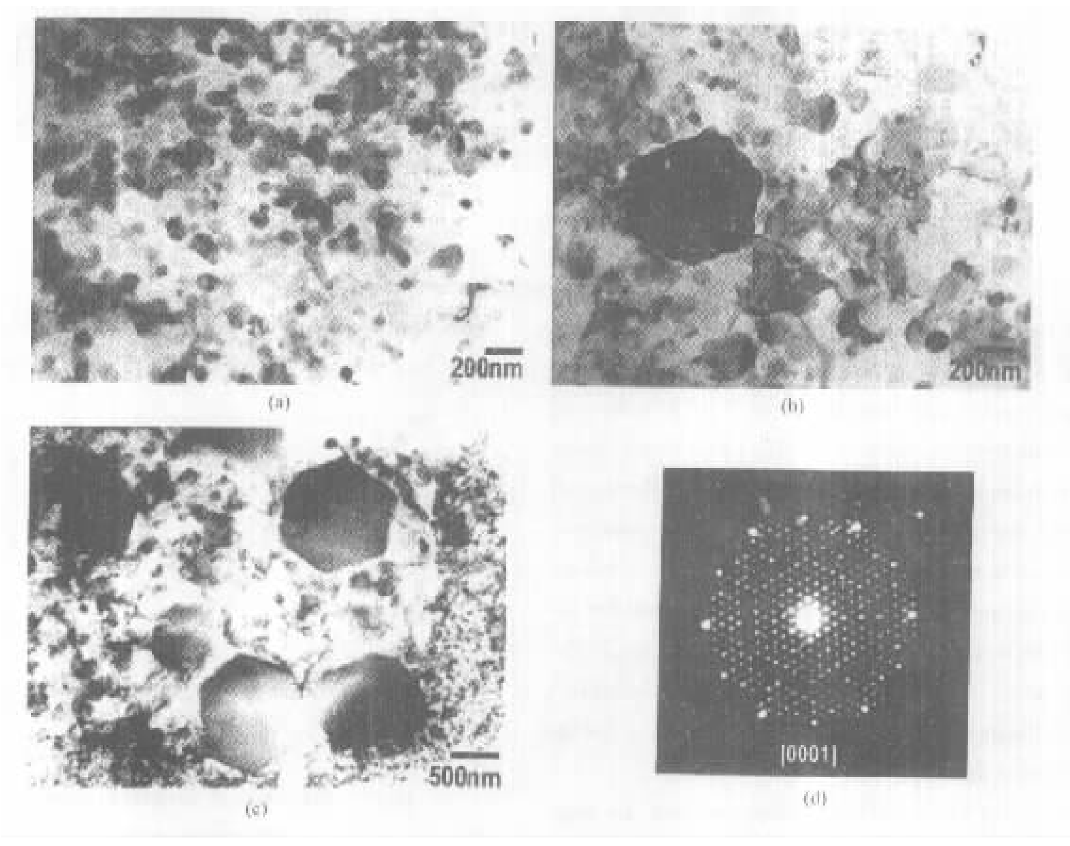
Fig.3(a),(b),(c) is the TEM images of as – extruded spray formed Al – Fe – V – Si alloy after hot – extruded at 250 ℃, 300 ℃ and 350 ℃ respectively. Transmission electron microscopy investigations of the alloys after high – temperature extrusion at 250 ℃ and below show that the overall microstructures of the alloys remain basically unchanged from the as – deposited condition. The strengthening silicide particles remain roughly spherical and show minimal coarsening, indicating the superior stability of the silicide phase at high temperature. The most noticeable microstructural feature observed in the sample hot – extruded at 300 ℃ and 350 ℃ was the presence of hexahedral particles. SAED combined with EDS analysis of the hexahedral particles show that their nature is hexagonal h –

Al_8Fe_2Si phase. Fig.3(d) is the SAED pattern of the hexagonal particle. EDXS result shows that the elements content in it is 70.39at% Al, 16.32at% Fe, 0.34at% V and 6.96at% Si respectively. The metastable hexagonal phase (h phase) was newly found by Koh et al in RS Al – Fe – V – Si alloy, and its formation was related to the undercooling of the melt. Its space group is P6/mmm, lattice parameters are a = 2.514 nm, c = 1.257 nm[8].



(a) bright field image (b) SAED of silicide phase in (a)

Fig.2 TEM image of the as – received spray deposit



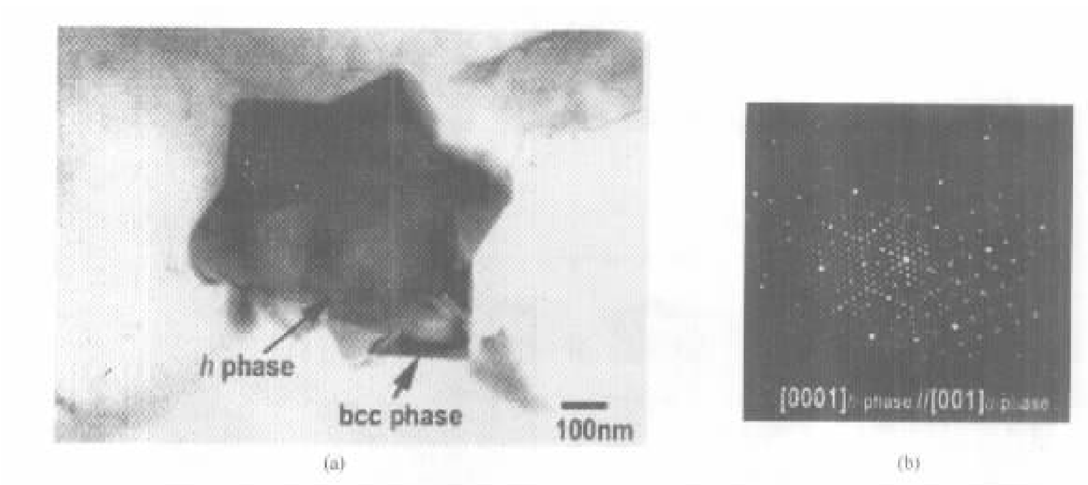
a.at 250 ℃; b.at 300 ℃; c.at 350 ℃; d.SAED pattern of hexagonal phase

Fig.3 TEM images of Alloys hot – extruded at different temperature

3 Mechanism of Phase Transformation

The microstructural features all have a strong influence on the mechanical properties of the materials. So it is important to try to understand the microstructural evolution of the materials. In RS Al – Fe – V – Si alloys formed by various processing routes, the b.c.c α –

AlFeSi phase was found adjacent to the six prismatic face of the h – phase by many authors, which is shown in Fig.4(a). When the aperture was placed over the two crystals, the SAED pattern composed of a [0001] zone axis pattern of the hexagonal phase and a [001] zone pattern of the bcc phase was obtained (Fig. 4 (b)). The pattern obeys the following orientation relationship between the hexagonal and b. c. c phase: $(001)_{bcc}//(0001)_{hex}$, and $[0001]_{hex}//[001]_{b.c.c}$. Many earlier researchers thought there must be a certain relationship between the transform of the b.c.c α – AlFeSi phase and h phase in RS Al – Fe – V – Si alloys. But there was a controversial opinion among them. Some researchers thought the b.c.c α – AlFeSi phase would transform to h phase, whereas some other researchers thought the presence of h phase must be transformed from b. c. c α – AlFeSi phase[7~11]. In this experiments, we found that there is no h phase in as – deposited alloys and the alloys hot – extruded at temperature below 300 ℃. The h phase must be more stable than b. c. c α – AlFeSi phase at high temperature and it can be reasonably claimed that the h phase was transformed from b.c.c α – AlFeSi phase.



(a) adjacent hexagonal and bcc phases (b) SAED pattern

Fig.4 TEM image of adjacent hexagonal and bcc phase

It has been reported that the silicide dispersoid may have a range of compositions, particularly of Fe and V. The quaternary element additions of V to Al – Fe – Si alloys stabilize the cubic α – $Al_{13}(Fe,V)_3Si$ phase with a more symmetrical lattice over the hexagonal Al_8Fe_2Si and monoclinic Al_3Fe phases that would normally form in the absence of the V addition[7]. However, the thermal and solute fluctuation may cause the element contents to be non – uniformly distributed in the molten alloy. A V – free area may form after solidification. Without the addition of V, the b.c.c α – AlFeSi phase would gradually transform to h phase.

The curves of the mechanical properties and the temperature of the as – deposited and as – extruded Al – Fe – V – Si alloys are plotted in Fig. 5. The yield strength (YS) and the ultimate tensile strength (UTS) of the alloy decrease monotonically as the hot extrusion temperature increase, which is due to the presence of clump h phase. The elongation rate of the alloy has a minimum value at 200 ℃, which is a common occurrence in any high temperature Al – based alloys.

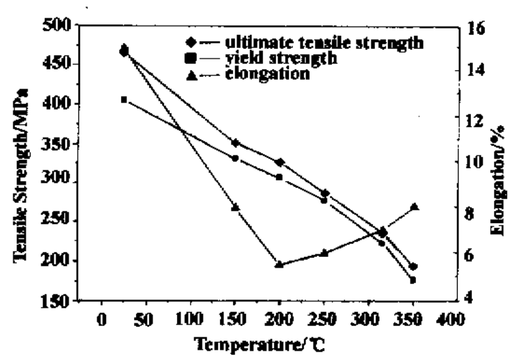


Fig.5 The curves of mechanical properties and temperature

4 Conclusion

(1) Densely fine silicide particles uniformly distributed in the matrix of as – deposited Al – Fe – V – Si alloy and the alloys hot extruded below 300 ℃, which play a

important role in increasing the mechanical properties of the alloy at high temperature.

(2) Some clump *h* phase are present in the Al – Fe – V – Si when hot – extruded at 300 ℃ and above, which is transformed from b.c.c α – AlFeSi phase due to the lack of V around them.

(3) The mechanical properties of the Al – Fe – V – Si alloys decrease which the hot extrusion temperature increases.

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喷射沉积 Al – Fe – V – Si 合金的组织演变研究

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摘　要：通过光学和透射电子显微镜研究了喷射沉积 Al – Fe – V – Si 合金在沉积态和不同温度热挤压后的显微组织，发现在 α – Al 基体上均匀分布着高密度的细小硅化物颗粒，这些细小硅化物颗粒在高温挤压后仍能保持较好的稳定性，在提高合金高温性能中起主要作用．在 300 ℃及其以上温度的挤压态合金中发现了一些六边形块状相，经分析是由于合金局部元素成分波动造成 V 元素的缺乏促使亚稳 α – AlFeSi 相矢稳转变而成，六边形块状相的存在是影响合金高温性能的主要原因．

关键词：喷射沉积；显微组织；Al – Fe – V – Si 合金；机械性能