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# Thermal stability of multi-spray deposition heat resistant Al-Fe-V-Si alloy①

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[Abstract] Rapidly solidified Al-8.4Fe-1.3V-1.7Si heat resistant alloy was made by using multi-layer spray deposition technique. Its thermal stability of mechanical properties was investigated by the means of tensile and hardness tests, thermal stability of microstructure by transmission electron microscopy (TEM), X-ray diffraction (XRD). The results show that after heat exposure (HE) at 753 K for 500 h the tensile strength and hardness of Al-Fe-V-Si alloy at 623 K maintains the same values as those at room-temperature. HE dose not obviously affect the thermal stabilities of $Al_{12}(Fe, V)_3Si$ phase but the lattice constant of $Al_{12}(Fe, V)_3Si$ phase decrease.

[Key words] heat resistant Al alloys; heat exposure; microstructure; metastable phase; mechanical property
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## 1 INTRODUCTION

Rapidly solidified Al-Fe-V-Si alloy is one of the most outstanding heat resistant alloys, because of the low coarsening rate and high thermal stability of the dispersed metastable phase $Al_{12}(Fe, V)_3Si$ formed in rapidly solidification process. Some researches reported that this metastable phase has incoherent interface with the matrix. Its good thermal stability of mechanical property is mainly related to its microstructure[1,2]. There is a lot of research about the thermal stability and phase transformation of this kind of alloy below 698 K[3~5] and above 773 K[6~8], showing that $Al_{12}(Fe, V)_3Si$ metastable phase does not decompose until 873 K. However it decomposes to stripped hexagonal $(Al, Si)_{13}Fe_4$ phase at 923 K after a thermal retardation for 25 h[8]. The mechanical property of this alloy is still very stable after HE for 1 000 h[3]. Mitra[9] and Lee[10] showed that this alloy became brittle at 755 K. Since the extrusion temperature in this study is high (753 K), it is valuable to investigate the thermal stability of mechanical properties and microstructure of this alloy after long time HE at 698 ~ 773 K. In this study, Al-8.4Fe-1.3V-1.7Si alloy pipe blanks were made by spray deposition[11] and hot extrusion and the effect of HE on the mechanical properties and microstructure of this alloy at 689~873 K was investigated.

## 2 EXPERIMENTAL

The master alloy with a nominal composition of Al-8.4Fe-1.3V-1.7Si was produced by melting iron (99.5%), aluminum (99.5%), silicon (99.5%) and Fe-50V intermediate alloy in an electric resistant furnace. Pipe blanks were prepared by means of multi-layer spray deposition set up is seen in Fig. 1. The pipe blanks were hot-forward-extruded with the extrusion ratio of 4.98. Samples (200 mm × 20 mm × 20 mm) taken from the extruded pipe were put into resistive heaters for HE tests at 698~ 873 K for 0~ 500 h. Brinell hardness was measured under the condition of 2 500 N for 30 s. Tensile strength tests were carried out on Instron8032 screw driven tensile machine with a strain rate of $1.5 \times 10^{-3} s^{-1}$ at room temperature and 623 K, respectively. Phase analysis was made on SIMENS D500 diffractmeter with IDR peak search and APP cell parameter calculation software using Cu $K_\alpha$ radiation. Specimens of TEM were prepared by twin jet polishing method of using the 33% $HNO_3$ + 67% $CH_3OH$ electrolyte under the

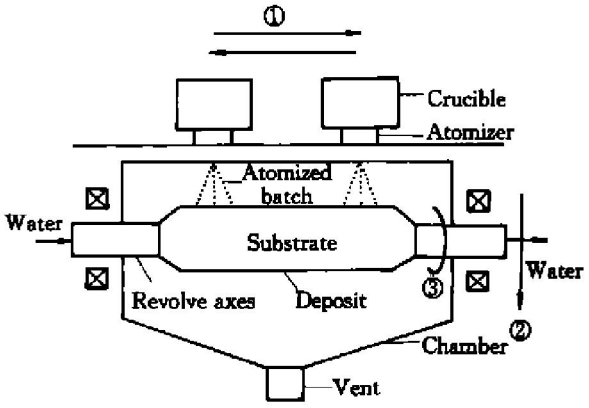


**Fig. 1** Schematic of multi-layer spray deposition device

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condition of 22 V and 257 K. The microstructure details were examined by an H-800 and a JEM-2000FX transmission electronic microscopes with an accelerating voltage of 160 kV.

## 3 RESULTS AND DISCUSSION

Fig. 2 shows the effect of HE time on room-temperature hardness of the samples. Brinell hardness (HB) of as-extruded alloy is 1 180 MPa. HE under the condition of 753 K and 500 h led no significant effect on the hardness of the alloy. For samples heat exposed at 773 K and 873 K, their hardness value first dropped rapidly with increasing HE time, however, the variation rate of the hardness was getting smaller and smaller, and tended to be constant after 100 h.

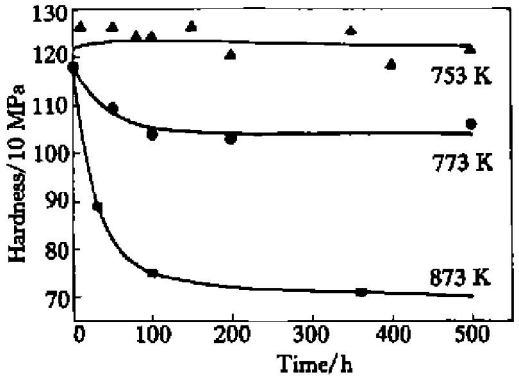


**Fig. 2** Effect of HE on room-temperature hardness of alloy

Fig. 3(a) shows the plots of room-temperature strength and ductility Verses HE temperatures (all samples were heat exposed for 500 h). Obviously the room-temperature tensile strength and yield strength decrease as HE temperature increases. But the decreased extents of the tensile and yield strengths were lower than 7%. Compared with extruded examples, the ductility of all heat exposed samples increased, except those heat-exposed at 698 K. The ductility of samples heat-exposed at 753 K increased by 7.6%.

Fig. 3(b) shows the plots of high-temperature strength and ductility vs HE temperatures (all samples were heat exposed for 500 h). Tensile strength and yield strength of the samples were higher than that of extruded ones. When HE temperature was above 773 K, compared with extruded samples, the tensile strength and yield strength of HE samples decreased by 8% and 17.5% respectively. Ductility increased gradually with increasing HE temperature. However, if the HE temperature was over 773 K, it was decreased by 39.7%.

Typically the microstructure of extruded samples is characterized by that spheroid particles of α-$Al_{12}(Fe, V)_3Si$ phase (between 40 and 200 nm) distributed in the grain boundaries of the α(Al) matrix phase

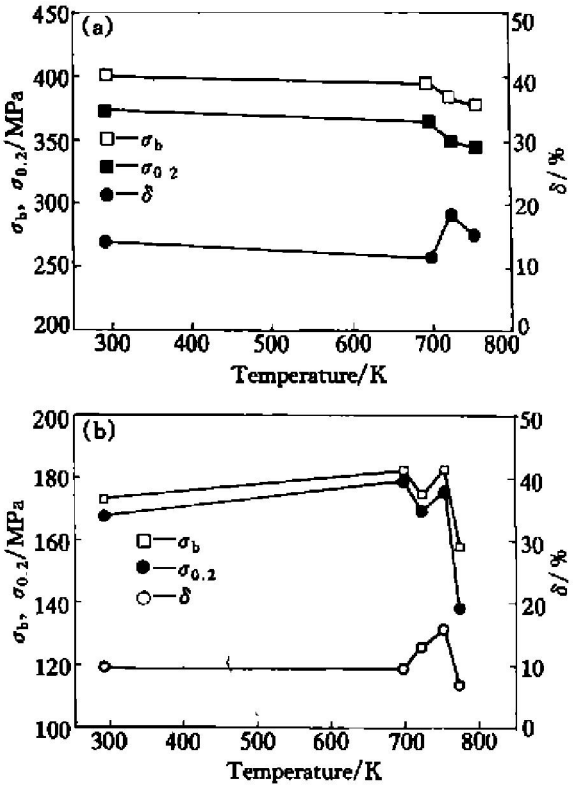


**Fig. 3** Mechanical properties of HE alloy at different temperatures for 500 h
(a) —At room temperature; (b) —At high temperature

(between 0.4 and 0.9 μm). Besides, a little $Al_8Fe_2Si$ and $Al_{13}Fe_4$ (verified by SAED) appeared in the α(Al) matrix phase. Another characteristic is its stratification phenomenon with layer width of 3 ~ 5 μm, this may be caused by the colliding between the solidified deposition layer and pulverized particles. SAED verified that the coarsened and rectangular second phase (200 nm) appeared in some part of the matrix still was α-$Al_{12}(Fe, V)_3Si$ particles with the theoretical lattice constant being around 1.256 mm (see in Fig. 4, A). Moreover, fine $Al_{12}(Fe, V)_3Si$ phase precipitated in the process of extrusion was observed in the same part (see in Fig. 4, B). The fact that no dislocations were found in the α(Al) matrix suggest that most of the matrix of the extruded samples has undergone recovery. In the samples heat exposed at 753 K, some grains of the matrix have grown up to the size of 1.2 μm in diameter, but no significant coarsening was observed in $Al_{12}(Fe, V)_3Si$ phase. However, after HE at 873 K the growth rate of α(Al) matrix and the coarsening rate of $Al_{12}(Fe, V)_3Si$ metastable phase accelerated obviously. A lot of dislocations appeared in α(Al) matrix grains, tangled each other and turned into small-angle sub-boundary. The $Al_{12}(Fe, V)_3Si$ particles at grain boundaries coarsen obviously and the number of the particles decrease. A TEM image of HE sample (873 K, 500 h) is shown in Fig. 5. α(Al) matrix grain has a size up to 3 μm in diameter.

From the observation above, we concluded that

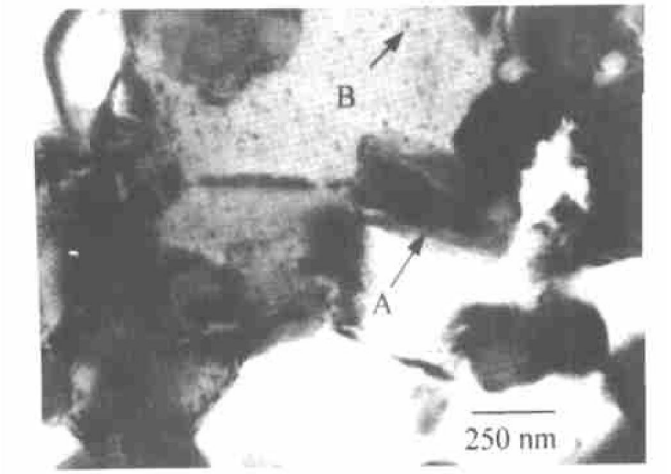


**Fig. 4** Typical microstructure of warm extruded sample

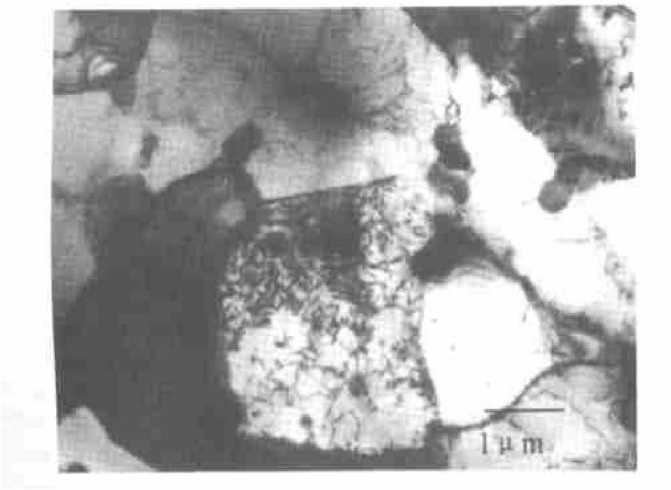


**Fig. 5** Microstructure of HE sample at 873 K for 350 h

the variations of hardness, tensile and yield strength after HE were attributed to the recovery and recrystallization of α(Al) matrix, but the variations of ductility mainly related to the size, the distribution and the shape of the $Al_{12}(Fe, V)_3Si$ particles. Since dislocations were hardly seen in the as-extruded sample, Lee[10] thought the matrix mainly underwent recrystallization in the process of extrusion, however, ZENG[7] considered the matrix mainly underwent recovery. We deemed the matrix mainly underwent dynamic recovery. It is known that the stack fault energy of Al alloy is very high, so that distance between two particle dislocations is narrow and interslipping tend to occur. The dislocations created in the process of deforming tend to converged and turned into dislocation tangle. However, for the rapidly solidified alloy, grains of the matrix are very fine, the dislocations slip distance is short and not easy to have interaction. In addition, the dislocations could disengage the second phase particles by climbing at the high extrusion temperature (e. g. 753 K). As a result, the dislocation density was very low and the recrystallizational driving force was small. The matrix did not undergo recrystallization but recovery. The sample heat exposed at 753 K for 500 h mainly underwent the decrease of interface energy of α(Al) matrix, that is, the process of grain growth. However, since a lot of fine $Al_{12}(Fe, V)_3Si$ phase particles dispersed in the matrix, especially at grain boundaries, which hindered the migrating of grain boundaries, the fine-grain strengthening effect was that remained. Therefore, the alloy displayed a good mechanical property at both room-temperature and 623 K.

Obviously, high HE temperature does good for the grain growth. Therefore, the decrease of hardness, tensile and yield strength of the samples, which were heat exposed at both room-temperature and 623 K, may be related to the diminishing of grain growth and fine-grain strengthening effect. However, the recrystallization of the matrix is the reason for the decrease of tensile and yield strength at both room-temperature and 623 K in the sample heat-exposed at 873 K for 350 h. ZENG[7] calculated the strain field of α(Al) matrix around $Al_{12}(Fe, V)_3Si$ phase, which was caused by the difference of the expansion coefficients of $Al_{12}(Fe, V)_3Si$ phase and α(Al) matrix, and verified the maximum shearing stress $\tau_{max}(\approx G/33)$ was commensurate to the critical stress of dislocations slipping. So, under certain HE condition, the heat stress would be big enough for the multiplication of dislocation (like the Fank-Read mechanism). Dislocations climbing may cause the dislocation tangle. Then the dislocations could turn into sub-boundaries and crystal nucleus. The decrease of ductility maybe related to the canalization effect of HE on the concentration for second phase particles in the grain boundaries and its shape is randomized, which made the interface reluctant to be source for microcracks.

In order to clarify the effect of HE on the stability of heat-resistant strengthening phase α-$Al_{12}(Fe, V)_3Si$, we measured the lattice constants of α-$Al_{12}(Fe, V)_3Si$ phase in the samples, which were heat exposed at 723, 753, and 773 K for 500 h, 873 K for 350 h. Since the diffraction peak in large diffraction angel of α-$Al_{12}(Fe, V)_3Si$ phase is too weak, lattice constants was calculated by measuring the value of d of (211), (220), (222), (321), (510), (521), (440), (530), (770) diffraction peaks. Results were shown in Fig. 6.

We can see that the lattice constant ($a_0$) of α-$Al_{12}(Fe, V)_3Si$ phase decreases compared with as-extruded samples. Skinner[3] showed the variations of lattice constant ($a_0$) and mechanical property of Al-Fe-V-Si alloy after using V as a substitute for Fe in $Al_{12}(Fe, V)_3Si$ phase. Their results demonstrated that when Fe: V atomic ratio was smaller than 5, variations of Fe: V atomic ratio could not affect $a_0$. However, when Fe: V atomic ratio was larger than 5,

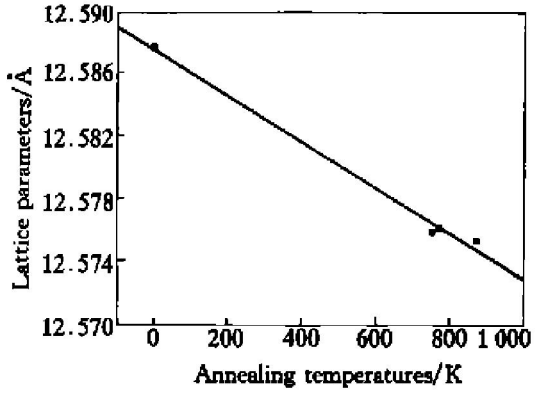


**Fig. 6** Lattice parameters of $Al_{12}(Fe, V)_3Si$ phase after HE for 500 h vs annealing temperature

the $a_0$ decreased with increasing Fe: V atomic ratio. The reason for decrease of tensile and yield strength with increasing Fe: V atomic ratio in the $Al_{12}(Fe, V)_3Si$ phase at both room-temperature and high-temperature may be that variations of Fe: V atomic ratio change the interface energy of $Al_{12}(Fe, V)_3Si$ phase particles and α(Al) matrix. WANG[12, 13] investigated the effect of V substituting for Fe on structure stability. Their results showed that V increased its structure stability. The variation of lattice constant $a_0$ of $Al_{12}(Fe, V)_3Si$ phase was caused by the change in Fe: V atomic ratio. At high temperature the diffusion ability of Fe, V, Si atoms in α(Al) matrix was enhanced so their solubility became larger and Ostwald ripening process occurred. The coarsening of $Al_{12}(Fe, V)_3Si$ phase was attributed to diffusion mechanism which is controlled by the diffusion ability of Fe atom[14, 15]. V atoms were used to stabilize the $Al_{12}(Fe, V)_3Si$ phase particles. Since the diffusion ability of Si, V atoms is better than that of Fe atom, the densities of the former atoms in α(Al) matrix and $Al_{12}(Fe, V)_3Si$ phase can be changed by HE, causing the changes of Fe: V atomic ratio in $Al_{12}(Fe, V)_3Si$.

## 4 CONCLUSIONS

1) After HE at 753 K for 500 h the high-temperature and room-temperature mechanical properties of Al-Fe-V-Si alloy had no change compared with that at room temperature. But they drop remarkably after HE above 753 K.

2) The Al-8.4Fe-1.3V-1.7Si alloy underwent dynamic recovery in the process of extrusion. The grains of α(Al) matrix grow up. For $Al_{12}(Fe, V)_3Si$ metastable phase there is no evident coarsening of its particles after HE at 753 K for 500 h. At 873 K recrystallization in α(Al) matrix occurred, the coarsening process of $Al_{12}(Fe, V)_3Si$ occurred at the grain boundaries.

3) Lattice constant of the metastable phase $Al_{12}(Fe, V)_3Si$ decreased with increasing HE temperature, that may be related to the increasing of Fe: V atomic ratio in this phase.

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