THE MICROSTRUCTURE OF A RAPIDLY SOLIDIFIED Al-Fe-V-Si ALLOY PRODUCED BY SPRAY FORMING

Y.Wang[1], N.Ridley[2] and G.W.Lorimer[2*]

1.On leave from Powder Metallurgy Research Institute, Central South University of Technology, Changsha, Hunan, 410083, P.R.China.
2.Manchester Materials Science Centre, University of Manchester/UMIST, Grosvenor Street, Manchester M1 7HS, U.K.
* Author to whom correspondence should be addressed.

Abstract

A rapidly solidified Al-8.5wt%Fe-1.5wt%V-1.7wt%Si alloy was produced by spray forming. Alloy sheets were obtained by hot rolling the spray-formed billets at 400 to 450°C. The effects of process parameters, especially the substrate temperature, on the microstructures of spray-formed billets and resulting alloy sheets were investigated using optical metallography and transmission electron microscopy. The results revealed that the microstructure of the deposit and rolled materials depends on the substrate temperature, which affects the cooling rate of the deposit. When the substrate was cooled to below 40°C the second phase precipitates in the deposits were fine and showed little coarsening even after repeated hot rolling. Coarse second phase particles were frequently observed in those specimens deposited on a substrate which was not cooled. TEM revealed regions of different-sized precipitates within individual grains. It is proposed that the discontinuity in precipitate size originated from differences in cooling rate.

Introduction

Aluminium alloys produced by rapid solidification processing(RSP) exhibit outstanding mechanical properties at temperatures where conventional aluminium alloys lose their strength. In the last two decades, considerable work has been done in the development of advanced aluminium alloys by using RSP[1-4]. Among these the Al-Fe-V-Si family of alloys appear one of the most promising because of their high elevated temperature strength, stiffness and thermal stability[4-7]. Usually, this class of alloys are produced by melt spinning (both jet casting and planar flow casting) and gas atomization.

Spray forming has been developed as an alternative RSP in recent years. In this process, the molten alloy is disintegrated into fine droplets by gas atomization and the droplet spray is deposited on a collecting substrate. Either the atomising nozzle or the collecting substrate may be moved during the processing in order to produce near net-shape products. The process eliminates secondary operations, such as sieving, canning and pre-compacting of powders. Oxide contamination is significantly reduced compared with alloys produced by conventional powder metallurgy processes[8-11].

Experimental

An alloy of nominal composition Al-8.5Fe-1.5V-1.7Si (in wt%) was produced by spray forming. The process has been described in detail elsewhere[12]. In brief, the production procedure involved induction heating a 1.5-2 kg alloy charge in a graphite crucible under an argon atmosphere to a temperature about 150-200°C above the liquidus. Molten alloy was extracted from the bottom of the crucible at a flow rate of 18-25 g/s into a gas-atomization nozzle, which was scanned at a rate of 80-120mm/s. The pressure of the atomising gas, N_2, was 8 to 12MPa. The atomised droplet-spray was collected on a roughened, degreased aluminium substrate plate which was scanned at right angles to the nozzle and at a distance of 300-400mm from it. The substrate plate was water-cooled, to produce high substrate cooling rates, or not. When water-cooled, the substrate temperature was kept below 40°C[12]. The deposit was built-up to a maximum thickness of 20mm over a time of about 60 seconds. The as-deposited billets were then repeatedly rolled at 350-400°C into sheets 2-3mm thick.

The alloy deposits and sheets were sectioned and prepared for optical metallographic examination using standard techniques of grinding, polishing and etching with Keller's reagent. Thin foils for transmission electron microscopy (TEM) examination were made by using a jet electropolishing technique with a solution of methanol containing 30 (vol)% nitric acid at -30°C and a voltage of 12V. TEM was carried out with either Philips EM301 or EM400T microscopes; the latter was equipped with an energy dispersive X-ray (EDX) analysis system. An in-situ X-ray microanalysis method developed by Lorimer et al[13] was used to determine the composition of the precipitates in the thin foils.

Results and Discussion

As-deposited Billets

Solidification Mechanism. Figure 1 shows the typical optical metallographic examples of the as-spray-deposited microstructure of the Al-Fe-V-Si alloy under two substrate cooling conditions. It can be seen that, when the collecting substrate plate is water cooled, a lamellar structure is developed in which some pre-solidified droplets are embedded between and within the layers, and pores are frequently observed. In contrast, the billet deposited on a non-cooled substrate shows a completely different optical microstructure in which the level of the porosity is greatly reduced and the lamellar structure is almost completely eliminated. The difference in deposition structure between the billets produced at different substrate temperatures is associated with the solidification mechanism of the spray droplets before and after impingement on the collecting substrate plate[9-11].

The droplets incident upon the substrate are of different sizes and in different states of solidification. Fine droplets may be solidified while large ones are molten. In general, the majority of the droplets are in a semi-solid/semi-liquid or undercooled condition. On

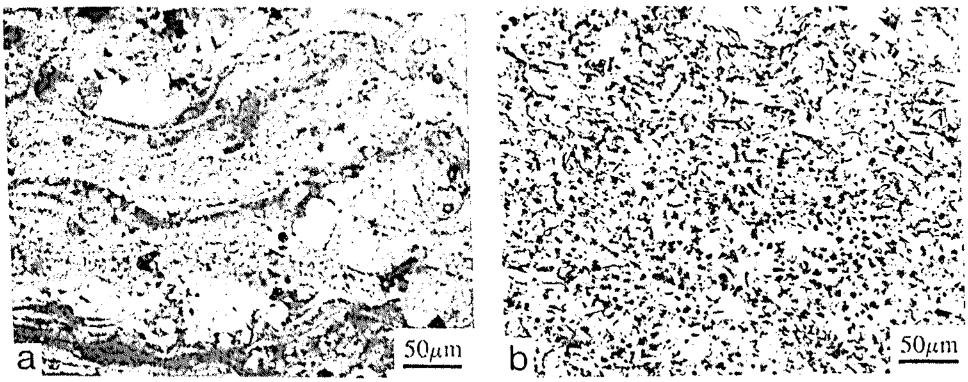


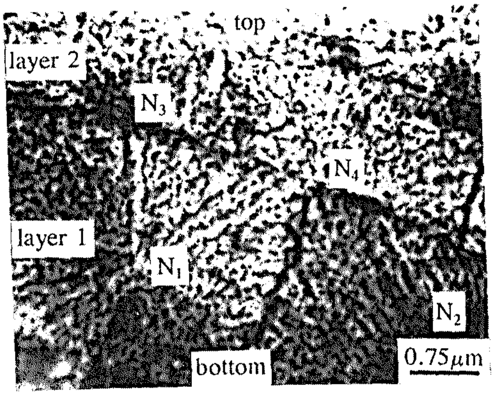
Fig.1 Optical micrographs of the as-deposited billets produced at (a) low and (b) high substrate temperatures, respectively.

impingement on the substrate, these partially solidified droplets are flattened and, after a short time when the heat extraction rate at the substrate is lower than the heat input rate at the upper surface, a molten (semi-solid/semi-liquid) layer forms on the top surface of the deposit. The thickness of the molten layer is dependent on the deposition rate and effectiveness of thermal transfer from the deposit to the environment. The thickness of the molten layer on the upper surface of the pre-deposit determines the resulting structure[9,10].

When the substrate temperature is low, heat transfer from the pre-deposit to the substrate is enhanced. This is particularly effective when the deposit is thin. The temperature of the molten layer formed on the top of the pre-deposit decreases rapidly so that there is insufficient time for the molten layer to fill all the inter-particle pores and there is insufficient heat for the pre-solidified droplets embedded in the layer to be completely remelted. The pre-solidified droplets and voids are found in the resulting deposit, as shown in Figure 1a.

If the substrate is not cooled, its temperature increases during the spray forming proceeds. This produces a relatively thick molten layer. The pre-solidified droplets may be remelted. There is sufficient time for the molten layer to fill the inter-particle voids and the level of the porosity is low, as shown in Figure 1b.

Microstructure. Figure 2 is the typical microstructure of the as-deposited billet produced with low substrate temperatures. It shows thin molten layers formed in the deposit. As revealed in the TEM micrograph, the nuclei N_1 and N_2 in the centre of layer 1 have grown into the melt until they met. The nucleus may grow through the thickness of the layer. Solidification may also occur heterogeneously on the surface of the pre-solidified material. These nuclei include the pre-solidified droplets and the solid fraction of partially pre-solidified droplets. As shown in Figure 2, two nuclei, N_3 and N_4, in layer 2 are on the upper surface of layer 1.



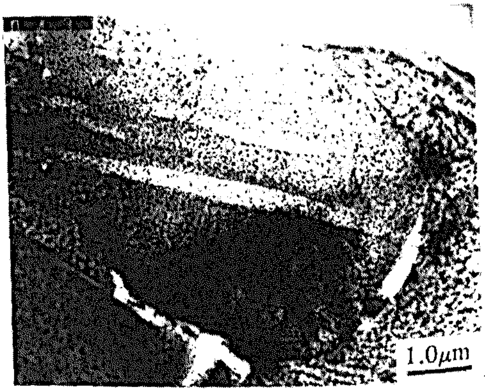


Fig. 2 Typical lamellar structure of the deposit. Note nuclei N_1 and N_2 in the centre of layer 1, and N_3 and N_4 on the surface of layer 1.

Fig.3 TEM micrograph showing a pre-solidified droplet in the deposit produced at low substrate temperature.

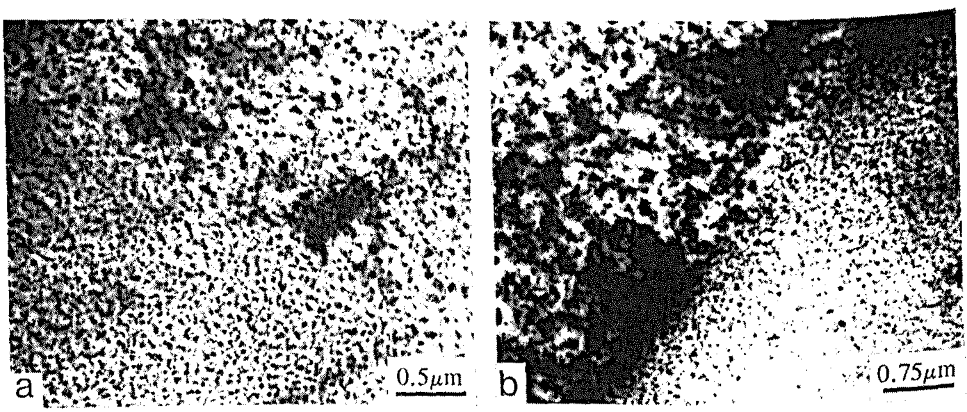


Fig.4 TEM micrographs showing regions with different sized precipitates and a discontinuity in precipitate size in the deposit produced at low substrate temperature.

Pre-solidified droplets were occasionally observed in the samples of the deposit made on the cooled substrate. Figure 3 reveals a pre-solidified droplet embedded in the molten layer. The prior particle boundary (PPB) is thinner and cleaner than that in similar alloys produced by RS/PM route. This reflects the low oxide contamination of the deposit[9-11].

Figure 4 shows two regions with different sized precipitates. This kind of microstructure was frequently observed and is different from those in the other aluminium alloys produced by the consolidation of powders. It is associated with the solidification mechanism of the spray forming process. The difference in precipitate sizes demonstrates a transition in cooling rate. Fine droplets may solidify before impingement on the substrate. As the spray forming proceeds, some of the pre-solidified droplets embedded in the molten layer are partially remelted. The remaining liquid will solidify on the surface of these solid droplets. It is proposed that the areas with fine precipitate particles originated from these partially remelted pre-solidified droplets. Another possibility is that the majority of the droplets are in a partially solidified state on deposition. When incident on the surface of the deposit, these droplets are flattened and solidification of the remaining liquid proceeds outward from the surface of the solid until all the liquid material is solidified. The heat extraction is predominantly controlled by conductive heat transfer from the molten layer to upper surface and to the pre-deposit and the substrate. The remaining liquid in the molten layer experiences a lower cooling rate than the solid fraction of the semi-liquid/semi-solid droplets. The lower cooling rate results in a larger precipitate size in those regions outside the pre-solidified fraction and the microstructure exhibits a discontinuity.

Precipitates. Figure 5 shows the size and morphology of the precipitates in the alloys produced with two substrate temperatures. The coarse, angular precipitates were frequently found in the deposits produced at high substrate temperature while spherical and fine precipitates were observed in those samples produced at lower substrate temperature. The EDX analytical data from these fine precipitates are shown in Figure 6. The data lies on straight lines, which indicates that the precipitates have a uniform composition. The atomic ratio of (Fe+V) to Si is 3:1. This is consistent with precipitate composition of $Al_{12\text{-}13}(Fe,V)_3Si$[5,6,14,15]. This conclusion is confirmed by selected area electron diffraction evidence which indicates the particles have a b.c.c. crystal structure.

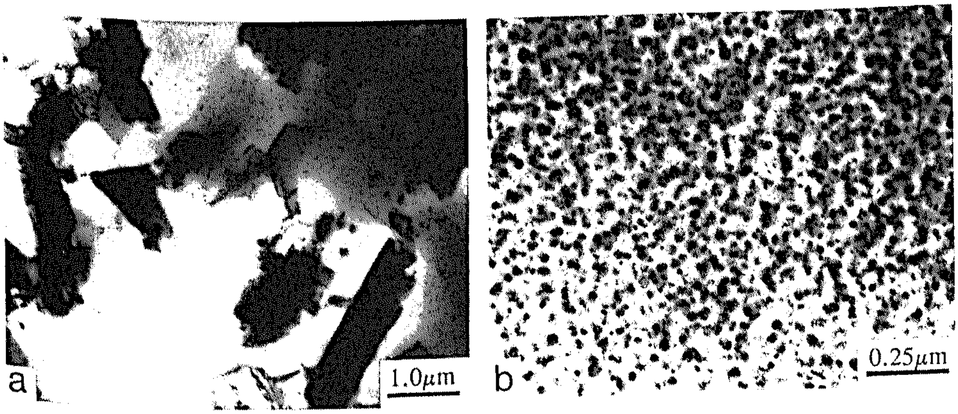


Fig.5 TEM micrographs showing the size and morphology of the precipitates in the deposits produced at (a) high and (b) low substrate temperatures.

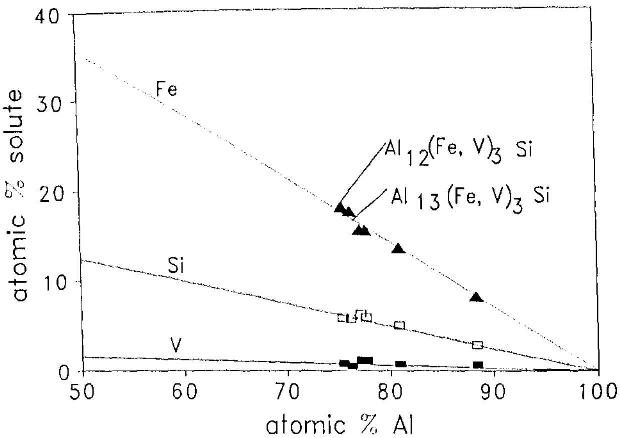


Fig.6 EDX analytical data from the fine precipitates present in Figure 5b.

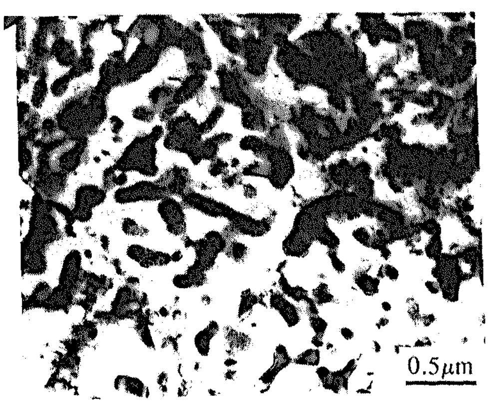


Fig.7 Variation in size and morphology of the precipitates near the upper surface of the deposit.

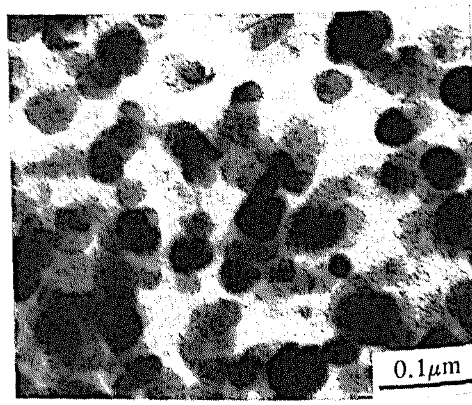


Fig.8 TEM micrograph showing the fine precipitates in the alloy produced at low substrate temperature after rolling.

There is a variation in size and morphology of precipitates through the thickness of the deposit. Figure 7 shows the large and irregular second phase particles present at the position about 18mm from the bottom surface of the deposit. EDX analysis indicated that these precipitates contain only aluminium and iron. As the thickness of the deposit increases, the cooling rate of the deposit decreases and the type of second phase formed changes.

As-rolled materials

The level of the porosity of the deposit was greatly reduced and only a few . . pores were observed in the rolled materials. The microstructure of the rolled materials produced at high substrate temperature exhibited coarse and angular-shaped second phase particles. The size and morphology of the precipitates showed little variation after rolling. Figure 8 shows that the precipitates in some regions of deposit produced at low substrate temperature remain fine after repeatedly hot rolling. However, microstructural inhomogeneities were occasionally observed in other regions of the alloy sheets. TEM revealed a cellular structure in which the walls of the cells are composed of precipitate particles. In other regions preferential coarsening of the second phase occurred along subgrain boundaries, as shown in Figure 9a and 9b.

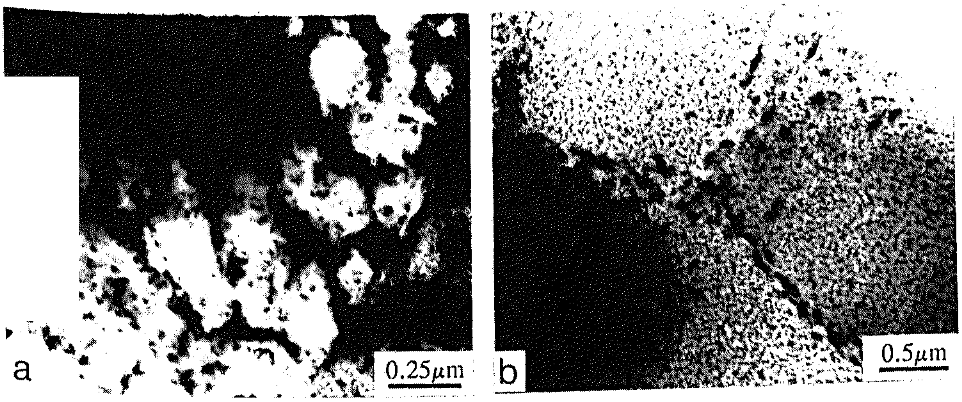


Fig.9 TEM micrographs showing the cellular structure (a) and heterogeneous precipitation of the second phase along the subgrain boundaries (b) in the rolled alloy.

Conclusions

The microstructure of an Al-8.5Fe-1,5V-1.7Si alloy produced by spray forming is dependent on the process parameters including substrate temperature. In the present investigation, substrate temperature influences the density, structure and type of second phases in the deposit. The deposits built up on a cooled substrate exhibited a lamellar structure in which pre-solidified droplets and pores were embedded. The deposits made on a non-cooled substrate showed a low level of porosity.

In the deposit produced at a low substrate temperature, fine spherical precipitates of $Al_{12-13}(Fe,V)_3Si$ with a b.c.c crystal structure were present. In those samples produced at high substrate temperature, coarse angular second phase were frequently observed.

A discontinuity in microstructures was observed in individual grains in the spray-deposited billets. It is proposed that this microstructure originated from experienced during spray forming.

The type, size and morphology of precipitates show variations at different positions in the deposit. The level of porosity in the deposits is greatly reduced after rolling, but microstructural inhomogeneities were frequently found in the alloy sheets. In some cases, the second phase particles formed a cellular structure.

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