Selective laser melting of Al–8.5Fe–1.3V–1.7Si alloy: Investigation on the resultant microstructure and hardness

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Abstract This article presents the microstructure and hardness variation of an Al–8.5Fe–1.3V–1.7Si (wt%, FVS0812) alloy after selective laser melting (SLM) modification. Three zones were distinguished across the melting pool of the SLM-processed FVS0812 alloy: the laser melted zone (LMZ), the melting pool border, and the heat affected zone (HAZ) in the previously deposited area around the melting pool. Inside the LMZ, either an extremely fine cellular-dendritic structure or a mixture zone of the α-Al matrix and nanoscale Al12(Fe,V)3Si particles appeared. With a decreased laser beam scanning speed, the cellular-dendritic structure zone within the LMZ shrank significantly while the mixture zone expanded. The α-Al and Al12(Fe,V)3Si mixture zone was also observed in the HAZ, but another phase, submicron θ-Al13Fe4 particles with rectangular or hexagonal shapes, formed along the melting pool border. Microhardness tests indicated that the hardness of the SLM-processed FVS0812 samples far exceeded that of the as-cast FVS0812 alloy.

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1. Introduction

With high strength, ductility, and toughness at temperatures up to 650 K, rapid solidification Al–Fe–V–Si alloys show great promise as a competitive alternative to titanium alloys for aerospace industry applications.1–3 The excellent mechanical properties of the Al–Fe–V–Si alloys are attributed to the presence of an ultrafine Al12(Fe,V)3Si phase and its low coarsening rate at high temperatures, which is extremely sensitive to the cooling rate and becomes uniformly distributed in the α-Al matrix only at an ideal high cooling rate.4,5 Many processes, e.g., rapid solidification-powder metallurgy (like planar flow casting, gas atomization), and spray deposition have been used to fabricate Al–Fe–V–Si alloys over the past few decades. Rapid solidification-powder metallurgy offers several inherent advantages over conventional ingot metallurgy processing, including alloy flexibility, chemical homogeneity and fine microstructures. However, the complicated procedures and the high-cost of this process have hindered its development. By comparison, spray deposition is a near net-shape forming technique and has many advantages such as simple equipments and process, little pollution and oxidation. However, during
the process of preparing Al–Fe–V–Si alloys, the cooling rate of spray deposition is too low to avoid producing harmful phase in matrix and this process has difficulty in manufacturing free-form components.\(^1\)–\(^5\) In conclusion, these traditional rapid solidification techniques are limited by complicated procedures and the difficulty in producing intricate parts, inevitably impeding further Al–Fe–V–Si alloys development.

Selective laser melting (SLM) has recently emerged as a promising additive manufacturing technology, capable of producing geometrically complex prototypes directly from metal powders.\(^6\)–\(^9\) SLM is a solid freeform fabrication process whereby a three-dimensional metallic component having any potential geometry is efficiently built layer by layer. Based on a mathematically sliced CAD model, fine powder layers are spread on the building platform, followed by laser beam scanning of each layer to selectively melt the particles. The highly localized heat input leads to fast melting and solidification, resulting in a unique microstructure.\(^10\)–\(^12\) This technology provides an opportunity to produce ready-to-use parts with minimal post processing requirements. Moreover, the cooling rate of the SLM process can reach \(10^5\)–\(10^8\) K/s, crucial for rapid solidification technologies.\(^13\)

There have been several metallic alloy systems developed from the above guidelines, e.g., Ti–6Al–4V,\(^14\) 304 stainless steel,\(^15\) and AlSi10Mg.\(^16\) However, the SLM of Al–Fe–V–Si alloys has not been reported. Here, the modification of Al–Fe–V–Si alloys using SLM was attempted to provide a new approach for manufacturing rapid solidification Al–Fe based alloys. Understanding the microstructural evolution during the SLM process is of the utmost importance for developing high performance Al–Fe–V–Si alloys. Hence, the microstructures of the Al–Fe–V–Si alloys prepared via SLM were investigated in detail, and their microhardness was examined to evaluate the mechanical properties.

2. Experimental procedures

With a nominal composition of Al–8.5Fe–1.3V–1.7Si (wt\%, FVS0812), the master alloy ingot, was prepared by vacuum induction melting in an argon atmosphere. Removing the surface oxide, the as-cast alloy ingot was atomized into powder using a ZGW-10 vacuum gas atomization instrument. The FVS0812 powder had a spherical morphology (Fig. 1(a)) and the particle size distribution determined by laser light scattering was homogeneous. \(D(0.5) = 22.1\ \mu m\) shows that the powder whose diameter is smaller than 22.1 \(\mu m\) accounts for 50\%, displaying a mean diameter close to 22.1 \(\mu m\) (Fig. 1(b)).

A 50 mm × 50 mm × 10 mm as-cast FVS0812 alloy plate was used as the building platform to guarantee good metallurgical bonding during processing. Prior to installation, the platform was sanded with abrasive paper and cleaned by alcohol.

![Fig. 1](image1.png)  
**Fig. 1** Characteristics of FVS0812 powder.

![Fig. 2](image2.png)  
**Fig. 2** Pictures showing visual appearance of SLM-modified cylinders.
The SLM experiments were conducted at room temperature using a self-developed DEYU LM 200 SLM instrument. This instrument is equipped with an IPG YLR-500 SM Yb: YAG fiber laser, which produces a laser beam with a wavelength of 1060 nm and a spot size of 80 μm in continuous mode. The working chamber was initially evacuated, followed by filling it with an inert argon atmosphere in order to prevent aluminum alloy oxidation during the fabrication process. Two cylindrical components (20 mm diameter × 10 mm length) were prepared using SLM (Fig. 2). Table 1 provides a production overview for the two different SLM-processed samples and their relative density values calculated using Archimedes’ principle. Fig. 3 shows the schematic of the scanning strategy applied in this work, with each layer scanned twice, using identical process parameters but rotating over 90° between the two scans.

Microstructural analysis was performed using electron probe microanalyzer (EPMA; JXA8100) and transmission electron microscopy (TEM; Tecnai G2 F30) equipped with an energy dispersive X-ray spectroscopy (EDS, INCA PentaFETx3). The phases were identified using X-ray diffraction (XRD, D/max2200PC) with CuKα radiation at 40 kV and 40 mA. The Vickers hardness measurements were performed on a standard Vickers microhardness tester (FM-800) with a 100 g load.

3. Results and discussion

3.1. Microstructure

The typical XRD patterns of the as-cast alloy, powder and SLM-modified samples are illustrated in Fig. 4. The as-cast alloy consisted of α-Al, θ-Al13Fe4 and Al2Fe2Si. In comparison, the XRD spectra of the powder and SLM-modified samples changed considerably. The FVS0812 powder and the SLM-modified specimens had similar constituent phases and strong diffraction peaks corresponding to α-Al, Al12(Fe,V)3Si, and θ-Al13Fe4 were detected, matching previous investigations on the phase compositions of rapid solidification Al–Fe–V–Si alloys.

Fig. 5 depicts the EPMA images of the macrostructure viewed along cross sections of the SLM-processed samples. The macrostructure of the SLM-processed samples displayed the typical laser melting tracks, which is similar to the Al–12Si alloy macrostructure. Melting pools formed side by side with clear borders between them. The majority of the melting pools were fan-shaped. It should be noted that owing to variations in depth and shape, the melting pools do not always appear continuous in the cross sections. Additionally, there were some small pores in the SLM-processed samples, which were difficult to avoid due to the oxidation of the aluminum alloy powder. From the magnified image of the macrostructure around the melting pool border (Fig. 5(a)), three zones could be distinguished: the laser melted zone (LMZ), the melting pool border, and the heat affected zone (HAZ) in the previously deposited area next to the melting pool. To clearly understand the SLM-processed alloy macrostructure, we conceptualized the melting pool, shown in Fig. 6.

The distinctive process of fabricating the FVS0812 parts track after track, and layer after layer combined with the fast
and directional cooling rates occurring during the SLM process created a unique microstructure. A closer look at the samples’ microstructures using TEM revealed the presence of extremely fine structures. Fig. 7 shows the microstructures across the melting pool in the SLM-processed samples and the three different zones mentioned above could be clearly distinguished. For sample A, a fine cellular-dendritic solidification structure appeared in the LMZ (Fig. 7(a) and (b)), which has been previously observed upon laser melting of aluminum alloys.\textsuperscript{21} During the SLM, the heat is extracted from the liquid melting pool to the previously consolidated layer and the substrate. For alloys, due to the solute redistribution, the liquid may become undercooled, destabilizing the solidification front and generating a transition from a planar solidification mode to a cellular or dendritic mode.\textsuperscript{22} Furthermore, the high-energy density of the laser gives rise to a directional heat transfer, resulting in a directional solidification. Consequently, the cellular-dendritic structure grew towards the center of the melting pool. This cellular-dendritic structure is similar to the “zone A” in melt spun Al–Fe–V–Si ribbons, reported by Park et al.\textsuperscript{23} They also reported that the $\alpha$-Al solid solution was present in the intracellular zone; whereas the intercellular region primarily consisted of a micro-quasicrystalline phase. In the HAZ, the previously deposited zone suffered heat treatment from the moving melting pool. The subsequent cooling rate was very fast but lower than that in the LMZ. The previously formed cellular-dendritic structure was affected by this reheating process, leading to its decomposition. Substantial nanoscale (20–70 nm) spherical particles precipitated and distributed themselves uniformly throughout
the \( \alpha \)-Al matrix in the HAZ (Fig. 7(c)). The electron diffraction pattern of the spherical phase could be indexed to the body-centered cubic \( \text{Al}_{12}\text{(Fe,V)}_3\text{Si} \) phase. This desirable mixture zone of \( \alpha \)-Al and \( \text{Al}_{12}\text{(Fe,V)}_3\text{Si} \) was extremely stable, significantly contributing to the FVS0812 alloy’s excellent mechanical properties at high temperatures. Moreover, there was a distinct boundary (the melting pool border) between the LMZ and the HAZ (Fig. 7(a)). Fig. 7(d) reveals the presence of a new rectangular or hexagonal phase approximately 0.2–0.8 \( \mu \)m in size along the melting pool border. A quantitative EDS microanalysis revealed that this new phase had a composition of 75.33 at% Al, 22.74 at% Fe, 0.91 at% V, and 1.00 at% Si. Based on the XRD and EDS results, the presence of \( \theta\text{-Al}_{13}\text{Fe}_4 \) was confirmed. The melting pool borders corresponded to overlapped regions of two different laser tracks, thus having melted twice and attaining relatively lower cooling rate than the cooling rate in the LMZ and HAZ. It is furthermore found that the morphology and dimensions of this submicron \( \theta\text{-Al}_{13}\text{Fe}_4 \) phase changed considerably from the harmful needle-like \( \text{Al}_{13}\text{Fe}_4 \) phase in the conventional as-cast alloy, measuring hundreds of microns in length, shown in Fig. 8. However, the effect of this submicron \( \theta\text{-Al}_{13}\text{Fe}_4 \) phase on the FVS0812 alloy’s performance requires further investigation.

In comparison to sample A, the cellular-dendritic structure zone within sample B’s LMZ shrank significantly, while the mixture zone of \( \alpha \)-Al and 20–100 nm \( \text{Al}_{12}\text{(Fe,V)}_3\text{Si} \) particles within the LMZ expanded (Fig. 7(e) and (f)). This can be explained by the decreased cooling rate of sample B, as the cooling rate has a considerable influence on the microstructure development. As the scanning speed decreased, there was a corresponding cooling rate decrease. Furthermore, sample B’s 30–200 nm \( \text{Al}_{12}\text{(Fe,V)}_3\text{Si} \) particles in the HAZ and its 0.3–1 \( \mu \)m \( \theta\text{-Al}_{13}\text{Fe}_4 \) particles along the melting pool border were larger than those of sample A, reinforcing the above conclusion (Fig. 7(g) and (h)).

Based on the TEM results, the microstructural characteristics of samples A and B across the melting pools are summarized in Fig. 9. For a laser power of 150 W, decreasing the scanning speed from 250 mm/s to 200 mm/s significantly altered the microstructural evolution in the LMZ, suggesting that process control is crucial for this alloy.

### 3.2. Hardness

Vickers hardness values of the as-cast and SLM-processed FVS0812 alloys were measured. For the given process parameters, the corresponding hardness values of sample A were 135–170 HV and 145–175 HV for sample B, which is comparable to the hardness of the FVS0812 alloy prepared by planar flow casting (about 140–160 HV). However, compared with the conventional as-cast FVS0812 alloy with hardness values of 40–45 HV, the hardness values of the SLM-processed alloys improved significantly, as shown in Fig. 10, mainly attributed to their improved microstructures. The nanoscale spherical \( \text{Al}_{12}\text{(Fe,V)}_3\text{Si} \) particles, along with the submicron \( \theta\text{-Al}_{13}\text{Fe}_4 \) particles and the solid solution strengthening resulting from the high cooling rate of the SLM-process have improved the hardness values in both samples.

![Fig. 8](image) EPMA image of as-cast FVS0812 alloy.

![Fig. 9](image) Microstructure schematic across the melting pool for SLM-modified samples.

![Fig. 10](image) Microhardness comparison of SLM-processed samples, FVS0812 alloy produced by planar flow casting and as-cast FVS0812 alloy.
4. Conclusions

(1) The Al–8.5Fe–1.3V–1.7Si alloy powder and the SLM-processed samples had similar constituent phases, consisting of α-Al, Al₃(Fe,V)₃Si, and θ-Al₃Fe₄.

(2) Three zones could be distinguished across the melting pool for the SLM-processed samples: the LMZ exhibiting a region of either a cellular-dendritic structure or a mixture zone of α-Al and nanoscale Al₃(Fe,V)₃Si particles; the melting pool border with distributed submicron θ-Al₃Fe₄ particles; and the HAZ with an identical mixture zone of α-Al and Al₃(Fe,V)₃Si. For a laser power of 150 W, as the scanning speed decreased from 250 mm/s to 200 mm/s, the cellular-dendritic structure zone within the LMZ shrank significantly while the mixture zone expanded.

(3) The hardness values of sample A were 135–170 HV and 145–175 HV for sample B. The hardness of the SLM-processed samples far exceeded that of the as-cast alloy with hardness values of 40–45 HV, ascribed to the SLM-processed alloys’ improved microstructures.

Acknowledgements

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References


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