Journal of Manufacturing Processes 35 (2018) 715–720

Contents lists available at ScienceDirect

Journal of Manufacturing Processes

journal homepage: www.elsevier.com/locate/manpro

Grain refinement of laser remelted Al-7Si and 6061 aluminium alloys with Tibor® and scandium additions

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A R T I C L E I N F O

Keywords:
Grain refinement
Laser melting
Solidification
Aluminium alloys
Microstructure

A B S T R A C T

One of the challenges facing additive manufacturing is the current lack of control over the microstructure that forms during the process. Typically, the rapid cooling rates and steep thermal gradients in the melt pool encourages epitaxial columnar grain growth. In this paper the effectiveness of scandium and TiBor® grain refiners in controlling the microstructure during laser melting of aluminium alloys (Al-7Si and 6061) is investigated. Electron Backscatter Diffraction maps show that the addition of both scandium and TiBor® grain refiners were successful in controlling the microstructure of the melt pools for both alloys. The scandium additions resulted in a mixture of fine equiaxed grains and fine-to-moderately sized columnar grains, whereas TiBor® additions resulted in fine homogenous and evenly distributed equiaxed grains. Despite the very high cooling rates the grain refinement principles for laser melting are the same as classical solidification. The results from this study can be used as a basis for further studies on developing customised alloys for Selective Laser Melting (SLM).

1. Introduction

Selective laser melting (SLM) is an additive manufacturing (AM) technique that uses a laser to systematically build a component layer by layer from 3D CAD data. The laser traces the outline of the component on a powder bed that melts and fuses the particles together to form a solid component. The technology is maturing rapidly and a number of businesses worldwide now offer commercial additive manufacturing services. Furthermore, the ASTM and ISO have recently developed additive manufacturing standards in titanium (Ti-6Al-4 V), nickel and stainless steel alloys. Although this list of alloys is certain to grow, at present, no standards are available relating to the additive manufacture of aluminium alloys. This in part could be related to the lack of research focusing on the development of specific aluminium alloys suited for additive manufacturing.

One of the challenges facing additive manufacturing is the current limited control over the microstructure that forms during the process. SLM is characterised by small melt pools undergoing rapid cooling, typically across steep thermal gradients [1]. This inherently encourages epitaxial growth [1,2] resulting in columnar grain formation [2–8]. Furthermore, the heat flow between bordering laser tracks results in complex cooling rates encouraging the growth of larger columnar grains [3,4,9]. Attempts have been made to optimise the AM processing conditions [1–5,7–14] in order to overcome these challenges. For example, modifying the scan speed, laser power and laser scan strategy have shown promise in modifying the as-built grain size [3,9,14]. While controlling processing parameters is one approach, there is an alternative opportunity to develop alloys containing chemical grain refiners that promise to produce homogenous fine grain structures and promote the columnar to equiaxed transition under the rapid cooling conditions of SLM.

Aluminium alloys have been the most widely studied alloy system for grain refinement on account of the significant commercial benefits offered by the refinement of cast aluminium products. Over decades the science of grain refinement has evolved and now a much more comprehensive understanding of the grain refinement mechanisms exists [15]. The works of Eborall and Cibula first identified the addition of titanium and boron to be extremely effective grain refiners for Al [16–18], and nowadays the Al-Ti-B grain refining system is still one of the most effective commercial grain refining systems available for Al alloys [15]. This system is particularly powerful because it combines potent nucleant particles (that encourage heterogeneous nucleation at

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https://doi.org/10.1016/j.jmapro.2018.08.030
Received 9 February 2018; Received in revised form 9 August 2018; Accepted 27 August 2018
Available online 25 September 2018

small undercooling) with solute that provides constitutional supercooling and high growth restriction [19,20]. The duplex theory has emerged as a popular theory for Al-Ti-B refinement [21] and proposes that the peritectic reaction envelops TiB_2 particles with a thin Al_3Ti substrate that has excellent crystallographic matching with the Al matrix [19]. Recent work has further suggested that titanium rich atomic interfaces on TiB_2 may also play important role in nucleating Al [22,23]. Other effective aluminium grain refiners include scandium [24–26] which promotes heterogeneous nucleation through the formation of peritectic Al_3Sc, which has a similar crystal structure to that of Al_3Ti particles and thus has a small lattice parameter mismatch (approximately 1.6% [26–28]) with Al [24–27].

Despite the substantial advancements in grain refinement technology of aluminium foundry alloys, little translation has occurred to rapid solidification processes such as AM. Welding typically encounters faster cooling rates than castings and promising results have been achieved through the use of titanium-boride and scandium grain refiners during this process [28–35]. Notably, the promotion of the columnar-to-equiaxed transition in the weld pool has been achieved with the addition of grain refiners. Furthermore, the grain refinement process also results in reduced defect susceptibility, including hot tears and large grain size distribution [31]. In casting, grain refinement of aluminium alloys has long been known to improve mechanical properties [36]. Very limited literature exists on the use of grain refiners in aluminium alloys during additive manufacturing (where cooling rates are estimated to be in the range of 10^5–10^8 k/sec [37]). Recent attention has been given to adapting a scandium containing aluminium alloy (Scalmalloy®) developed for aerospace applications for additive manufacturing. Spierings et al. [38] reported that Scalmalloy® components produced by SLM contained a mixture of very fine equiaxed grains as well as coarser < 100 > columnar grains growing parallel to the build axis. In this instance, the addition of scandium was unable to fully promote the columnar-to-equiaxed transition.

The purpose of this paper is to investigate the effectiveness of grain refiners in controlling the primary grain size and morphology during laser melting of aluminium alloys. Custom alloys are first produced via casting and then remelted in a commercial SLM machine across a range of laser scan speeds typically adopted during AM of aluminium components. Thus, although the process studied in this work is not SLM, the cooling rates experienced during single track laser melting is comparable to SLM and will provide new insights into alloy design principles for AM.

2. Materials and method

2.1. Experimental design

The purpose of this work is to evaluate titanium-boron and scandium based grain refiners during solidification under cooling conditions typically experienced during SLM. As commercial powders of the desired compositions were unavailable, the alloys in this study were firstly produced by casting and then single tracks were remelted using various laser parameters in an EOSINT M280 SLM (details below). This effectively simulates single layer laser melting at cooling rates similar to those experienced during SLM. Thus, this experimental method is a first step approach which enables non-standard compositions to be easily investigated under cooling rates similar to that during AM. This lays the foundation for future work to investigate promising compositions in AM trials.

Two base aluminium alloys were selected. The first alloy, Al-7Si is a popular aluminium foundry alloy known for its very good castability. The response of this alloy (and other silicon containing alloys) to grain refinement during casting at slow cooling rates is widely understood [16–18,21,26,39–43]. One of the peculiarities with Al-Si casting alloys refined with titanium-boron is the apparent poisoning phenomenon that occurs at around 7 wt% silicon, at which, the effectiveness of the grain refiner addition is limited compared with lower and higher silicon contents [44,45]. For this reason, a second low silicon containing alloy was also studied. Aluminium 6061 is a commonly used wrought alloy (containing 0.6 wt% silicon as well as other elements including Mg, Fe, Cu, Cr, Ti, Zn and Mn, see Table 1 for full composition) and there have been several recent attempts to adapt it to SLM [46]. A known problem with adapting this wrought alloy to solidification processes is its susceptibility to cracking defects, including during SLM [47].

2.2. Materials

The base aluminium alloys used for this study were Al-7Si and 6061. A commercial titanium-boron grain refiner was used (Tibor®; 5 wt%Ti, 1 wt%B with Al balance) to result in a targeted alloy concentration of 0.33 wt%,[1] which is expected to provide a sufficient level of grain refinement [19,39,44]. Scandium additions were made to the appropriate alloys using an Al-2 wt%Sc master alloy which was added to give a targeted overall composition of 0.4 wt%Sc. This is a known composition for effective grain refinement in casting and results in the formation of primary Al_3Sc phase during solidification [27]. The resulting alloys with added Al-2 wt%Sc master alloy are based on Al-7Si and 6061 and for simplicity for the rest of the paper will be called Al-7Si with Sc and 6061 with Sc.

The alloying components were placed in boron-nitride coated crucibles and preheated in an air furnace above 200 °C to remove all moisture. The crucibles were then transferred and melted in an induction furnace (Inductotherm Powertrak 20-96R). The melt temperature was monitored using a K-type thermocouple and casting occurred once the melt stabilised at 720 °C. A permanent steel mould was used for casting and the cooling rate was approximately 6 °C/sec during solidification. Samples for laser remelting were sectioned from the castings and ground flat using silicon carbide paper. Table 1 presents the chemical composition of each of the alloys produced.

An EOSINT M280 machine was used for remelting experiments. The laser spot size was 100 μm and the laser power was held constant for all scan speeds at 370 W, a power commonly used for SLM of Al alloys [1,48–50]. Three scan speeds were used for the laser melting: 500 mm/s, 1500 mm/s and 2500 mm/s. It is important to note that the initial alloys were all effectively melted by the laser at these laser scanning speeds. Studies have shown that scan speeds between 500–2500 mm/s result in the highest relative densities of aluminium alloys during SLM [3,12,49]. In general, slower scan speeds result in better surface finish whereas faster scan speeds have the advantage of increased productivity. After laser re-melting, the alloys were sectioned normal to the laser scan direction (revealing the cross section of the laser melt pool) and prepared for microstructural examination using conventional techniques. SEM and EBSD (step size of 0.2335 μm and accelerating voltage of 25 kV) were used for examination and to reveal the grain morphology. The grain size was measured using a linear intercept method based on ASTM E112-13.

3. Results and discussion

Fig. 1 shows an example of a SEM and EBSD map of a sample revealing the laser melted area. From SEM examination, a clear distinction exists between the parent cast material and the small volume of metal that has undergone remelting by laser scanning. This distinction is more difficult to observe after EBSD and for this reason the outline of the laser melt pool has been included as indicated by a dashed line. Figs. 2–4 show the EBSD maps with the melt pool outlined on each of the alloys at scan speeds of 500 mm/s, 1500 mm/s and 2500 mm/s respectively.

[1] While this is considered a high level of addition (typically 0.1% is effective), it was selected to ensure that any effect of the grain refiner was observable.