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A comparison of Selective Laser Melting with bulk rapid solidification of AlSi10Mg alloy

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ABSTRACT

In Selective Laser Melting (SLM) layers of atomized powder are spread sequentially on a building platform and melted locally by a laser beam. The melt pool is quenched by the underlying material. SLM of AlSi10Mg alloys results in the development of microstructures consisting of supersaturated primary Al-rich phase surrounded by varied amounts of Al-Si eutectic. The origin of such microstructure is not fully understood. For insight into this issue, this work compares the results of processing AlS10Mgi alloys by SLM and by single-step rapid solidification techniques: Melt Spinning (MS) and Copper Mould Casting (CMC) achieving a range of cooling rates and microstructures which are analysed by means of microscopy, XRD and DSC.

The results obtained in these experiments together with the literature available on rapidly solidified Al-Si alloys suggest a correlation among microstructures of the products made with the three techniques. Data on lattice parameter and enthalpy of Si precipitation from primary Al concur in indicating that Si supersaturation scales in the order SLM > CMC > MS. The type and size of microstructural features, i.e. cells, columns, fibrous and lamellar eutectic, reveal the role of solidification conditions (undercooling, recalescence) and precipitation in the solid state for all techniques. Dendrite growth modelling validates the solidification results.

1. Introduction

Additive Manufacturing (AM) is the term used to indicate all 3D manufacturing processes employed to build components by adding material step by step, in contrast with established subtractive manufacturing processes [1]. Among them, laser powder bed fusion process (L-PBF), commonly defined Selective Laser Melting (SLM), is the most widespread for processing metals [2,3]. In SLM layers of atomized powder are spread on a preheated building platform and melted locally by a laser beam according to a CAD model of the designed object. Once the pattern of the first layer is complete, the building platform drops one step down, the build is recoated with new powder, and the process is repeated until an entire object is made additively. The interaction of the moving laser beam with the metal powder produces a melt pool which is rapidly quenched by the pre-solidified layers. This processing route has been extensively studied in the past years for Al-Si alloys [4].

Eutectic and hypoeutectic Al-Si alloys (e.g. AlSi12, AlSi10Mg and A357) have good castability, low shrinkage and low melting temperature, ideal for conventional casting, and were selected by several authors as material to test powder bed AM in which the morphology and amount of refined eutectic Si are acknowledged to be significant factors influencing the mechanical properties of Al-Si alloys [5–10].

Although the production and characterization of AM samples has provided substantial information on processing parameters for these materials [6,11–15], detailed knowledge of the mechanism of formation of microstructure during cooling has not been fully described yet. After fast melting the crystal growth front moves rapidly across the melt pool producing a microstructure made of cells (or columns) and fine eutectic. The primary phase is apparently supersaturated and the eutectic coupled growth is confined in thin volumes around the cells [12,16,17]: the amounts of trapped solute and of retained eutectic are crucial parameters to be clarified.

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Al-Si alloys have been studied since the early development of rapid quenching techniques: the main objectives being the extension of Si solubility in Al and surface hardening. Microstructural features of alloys, change in lattice parameter of Al, the thermodynamics of metastable phases and the kinetics of precipitation were reported for splat quenched, melt spun and atomized samples [18–26]. Moreover, dendritic growth under rapid solidification conditions employing laser melting was studied both with *ad hoc* experiments and through modelling [25,27–30].

This work aims at advancing in the interpretation of the microstructure of SLM Al-Si alloys on the basis of the available information on rapid solidification and new experiments performed by using current alloys. A correlation is sought for microstructures produced by mean of three rapid solidification techniques (Copper Mould Casting (CMC), Melt Spinning (MS), and SLM) to span a large range of cooling rates. The experimental results on supersaturation, calorimetric responses, and dimension of the microstructural features, and the consolidated literature allow proposing mechanisms for processes occurring in different ranges of cooling rates. Dendrite growth modelling validates the results on the microstructure found in AM.

2. Experimental

All samples were produced using gas atomized powders provided by EOS GmbH whose chemical composition is reported in Table 1.

SLM cubic samples with side of 15 mm were fabricated with an EOSINT M270 Dual Mode version employing the process parameters reported in Ref. [8] and repeated for clarity in Table 1 of Supplementary Information.

Samples were analysed both in the as built condition (AM_AB) and after annealing for stress relieving at 300 °C for 2 h (AM_SR). Pellets of powder were produced by gentle pressing and used for MS and CMC. Induction melting was performed with the help of a susceptor, i.e. a Ta foil surrounding the respective silica and boron nitride crucibles. AlSi10Mg melt spun ribbons were obtained by ejecting the melt onto a copper wheel rotating at either 10 m/s or 15 m/s (MS_N10 and MS_N15 respectively). CMC samples were obtained by ejecting the molten alloy into a conical mould of diameter varying from 5 mm to 1 mm.

For microscopy samples were embedded in conductive resin, mechanically polished, and etched for 10 s using Keller's solution. Secondary electron imaging was carried out both in SEM and FESEM mode using a LEICA STEREO SCAN 420 and a ZEISS SUPRA TM 40.

X-ray diffraction (XRD) was performed in Bragg-Brentano geometry with a PANalytical X'Pert PRO diffractometer by Philips, using the K_α emission line of a Cu filament ($\lambda_{Cu} = 1.5418$ Å). Patterns were acquired in the 2θ range from 20° to 140° at steps of 0.0167°. The SLM build was analysed on the top (AM_AB top and AM_SR top), side (AM_AB side and AM_SR side), and inner surface after cutting (AM_AB in and AM_SR in). For all samples, including an alloy portion cooled from 660 °C to room temperature at 3 °C/min representing the solidification in near equilibrium condition, the face centred cubic (fcc) Al lattice parameter was computed with the cosθcotθ method.

Thermal analyses were made with a TA Q100 Differential Scanning Calorimeter (DSC) in the temperature range from 50 to 450 °C at the heating rate of 5 and 20 °C/min equilibrating the heat flux at both temperatures.

Table 1
Chemical composition of the AlSI10Mg powder used in this study (wt%).

Alloy	Si	Fe	Cu	Mn	Mg	Zn	Ti	Al
AlSi10Mg	9–11	≤0.55	≤0.05	≤0.45	0.2–0.45	≤0.10	≤0.15	Bal.

3. Results

3.1. Rapid solidification microstructures

Fig. 1 reports images of the microstructures obtained in AlSi10Mg samples produced by means of different rapid solidification techniques. CMC samples were analysed after sectioning the cone along its vertical axis. A thin zone (<1 μm) near the outer surface is almost featureless due to faster solidification occurring in contact with the mould. Then, cells and columns are seen, surrounded by fibrous eutectic (Fig. 1-a). Their size increases from 1 to >5 μm from the tip to the base and from the outer surface to the core of the cone (Fig. 1-b).

It is well established [31] that the microstructure of ribbons changes from the wheel side to the outer side (Fig. 1-c). Here it starts almost featureless, becoming than primary plus eutectic with subdivided fine Si particles and ending with primary cells surrounded by fibrous eutectic. The length scale of the cells is finer with respect to CMC samples (cell size of 1–2 μm) (Fig. 1-d).

As already reported in literature [11,13,32–34] the microstructure of the SLM as built AlSi10Mg part has a transition from very fine cellular-dendritic to a coarser dendritic structure going from the centre to the border of the melt pools. This is clearly visible in Fig. 1-e, where the primary Al cells are surrounded by fibrous Si eutectic and change in size because of heat flux generated during subsequent laser scans. Looking across a melt pool, three zones appear: a finer microstructure in the centre (mpc), a coarser cellular microstructure in the transition zone from the centre to the border (mpb), and coarser Si particles in the heat affected zone around the melt pool in the layers deposited previously (haz). The cellular-dendritic structure is to some extent columnar along the building direction following the path of heat extraction via the substrate [5,16,32,35]. As evident in Fig. 1-f, the cells contain occasional Si particles (white contrast) and voids of the same size (dark grey contrast) at points where Si particles were removed by etching.

From the set of images it is apparent that the amount of Al-Si eutectic differs among various samples. Its percentage was determined by careful image analysis and averaging the results obtained for more than 10 micrographs taken at different magnification. It is compared with the expected equilibrium value calculated using the lever rule in Table 2. It is underlined that the microstructure changes when process parameters are changed, since the final result depends on the parameters defining the strategy for the SLM process which results in varied temperature gradient, solidification and cooling rate. Therefore, the results reported here on the amount of eutectic are quantitatively valid for the processing parameters and instrumentation employed for the production of the present samples. However, the methodology applied in this work appears well applicable to other sets of parameters.

After annealing for 2 h at 300 °C for stress relieving samples produced by means of the AM route, Si particles become coarser and precipitation of new Si particles inside the primary phase occurs, Fig. 1-h. Moreover, the heat treatment has an homogenizing effect on the size of Si particles in the matrix, Fig. 1-g.

3.2. XRD

Fcc Al and diamond cubic Si phases were identified in XRD patterns of all samples. Occasionally, minor reflections of Al_2O_3 were found, i.e. the oxide produced by coalescence into fine particles of the surface skin of the starting powder particles and,