Optimization of heat treatment parameters for additive manufacturing and gravity casting AlSi10Mg alloy

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Abstract. Additive manufacturing of metals is a production process developed in the last few years to realize net shape components with complex geometry and high performance. AlSi10Mg is one of the most widely used aluminium alloys, both in this field and in conventional foundry processes, for its significant mechanical properties combined with good corrosion resistance. In this paper the effect of heat treatment on AlSi10Mg alloy was investigated. Solution and ageing treatments were carried out with different temperatures and times on samples obtained by direct metal laser sintering and gravity casting in order to compare their performance. Microstructural analyses and hardness tests were performed to investigate the effectiveness of the heat treatment. The results were correlated to the sample microstructure and porosity, analysed by means of optical microscopy and density measurements. It was found that, in the additive manufactured samples, the heat treatment can reduce significantly the performance of the alloy also because of the increase of porosity due to entrapped gas during the deposition technique and that the higher the solution temperature the higher the increase of such defects. A so remarkable effect was not found in the conventional cast alloy.

1. Introduction
Additive manufacturing (AM) is a production process whereby products are made through a layer by layer melting of a metallic powder (or wire) with a focused energy source [1-2]. This manufacturing method is largely used for aircraft and automotive fabrication [3]. Additive manufacturing technology has revolutionized components manufacturing and logistics, allowing the production on demand, the reduction of energy consumption [4] and the manufacture sustainability [5]. It provides several advantages such as a reduction of material used [6-7], unrivalled freedom of design [1, 5-6, 8-10], elevated quality [11] and a lightening of the final component [12]. It is also important to consider the lead time reduction [1-2, 13], the possibility to realize shapes specifically designed on the requests of the customer [11] that cannot be obtained by traditional production methodologies [6, 14]. Nowadays, different techniques are available for AM of metals, like: laser beam melting, laser metal deposition or electron beam melting. In particular, the laser beam melting process is also known as selective laser melting (SLM), direct metal laser sintering (DMLS), laser metal fusion etc., mainly depending on the trademark of the machine producer [1]. The common metals for AM are: some Ti, Ni, Al and Co alloys and some steels [1].
Aluminium alloys have been considered as an alternative material to replace steels in automotive industry, owing to increasingly strict standards with respect to fuel efficiency, air pollution, recycling and safety [15-16]. For this reasons, today, many automotive components are made in aluminium alloy, such as engine blocks, cylinder heads, wheels, suspension links, engine heads, pumps, callipers, etc.
Many of these parts are typically produced by foundry processing, like gravity or low pressure die casting, with Al-Si alloys containing age hardenable elements [17]. In these alloys, in fact, the small addition of Mg (in the order of 0.3-0.5 wt.% Mg) induces hardening of the aluminium alloy by forming Mg2Si precipitates upon solution treatment followed by natural or artificial ageing (T4 or T6 respectively) [17].

Lately, due to the above-mentioned advantages, also additive manufacturing has been applied for the production of high performance aluminium parts [1]. In particular, AlSi10Mg alloy is one of most widely used aluminium alloys in AM, as well as in foundry processing, because of its high mechanical properties, low thermal expansion and good corrosion resistance [17]. Typically, this alloy undergoes T6 treatment in order to enhance its performance. Some authors have already reported the temperatures for both solution and ageing treatment, ranging between 500-545°C and 150-180°C respectively [19-20]. The durations are normally based on studies carried out on conventionally cast parts, which are characterized by different microstructure compared to AM, thus showing a different response. Aboulkhair et al., for instance, reported that a conventional T6 performed on selective laser melting (SLM) AlSi10Mg results in softening the materials, raising the question of the validity of conventional heat treatments on AM parts [20]. Similarly, Thijs et al. showed that the as-built SLM AlSi10Mg alloy presents a hardness comparable to the high-pressure die-cast alloy in aged condition [21].

Notwithstanding these findings, SLM AlSi10Mg parts are usually heat treated. This happens because, after solution, quenching and ageing treatment, the former anisotropy in the microstructure, caused by the layer by layer manufacturing, is dissolved [1] and an improved ductility is gained [22]. Hence, as already outlined by some authors [19-20], it is fundamental the definition of a new set of heat treatment procedures, specifically tailored for AM parts.

The aim of this work is to analyse systematically the effect of different temperatures and durations, of both solution treatment and artificial ageing, on hardness and microstructure evolution of AM AlSi10Mg parts, considering in particular the combined effect of porosity. The same investigation was performed on AlSi10Mg gravity cast parts to compare the different behaviour.

2. Experimental procedure

A commercially available AlSi10Mg powder was used in this study for the production of samples by direct metal laser sintering technique (DMLS). The manufacturer used proprietary process parameters to produce the parts.

Cast samples were obtained from a component produced by gravity casting (GC) in a permanent mould. Attention was paid to cut the samples from areas with the same section width in order to ensure the same solidification condition and the same microstructural characteristics. The samples chemical composition is shown in Table 1 for both these manufacturing techniques.

|            | Si   | Mg  | Fe  | Cu  | Mn | Ni  | Zn  | Ti  |
|------------|------|-----|-----|-----|----|-----|-----|-----|
| AlSi10Mg   | 9–11 | < 0.55 | < 0.55 | < 0.05 | < 0.45 | < 0.05 | < 0.12 | < 0.15 |

For both the considered production processes, some samples were tested in as cast condition, while the others were heat treated in a laboratory furnace. Several heat treatment conditions were applied by changing temperature and time. Particularly, solution treatment was performed at 510°C and 540°C for 6 hours. Then, samples were water quenched at 65°C. After each solution treatment and quenching, half samples were aged at 160°C, while the others at 170°C. Ageing time ranged from 1 to 10 hours in order to obtain a complete ageing curve. Between solution and ageing treatment, samples were kept at -20°C in order to avoid natural ageing.

The specimens were grinded and polished with waterproof paper and diamond cloths up to mirror finishing. Microstructural observations were carried out by optical microscope Leica DMI 5000 M equipped with LAS software; DMLS microstructure was also analysed after Keller etching for 20 s.
The density of the all samples was measured by hydrostatic weighing method using a proper balance (Gibertini E42-B) and holding system both before and after heat treatment. According to this method, samples were weighted in two different media, air and water, and the sample density $\rho_{\text{sample}}$ was calculated with the following formula:

$$\rho_{\text{sample}} = \rho_{\text{water}} \cdot \frac{W_{\text{air}}}{W_{\text{air}} - W_{\text{water}}}$$

where $W_{\text{air}}$ and $W_{\text{water}}$ are the weight of the sample in air and water, respectively, and $\rho_{\text{water}}$ is the water density.

Finally, material hardness was measured by Brinell method with a Galileo Ergotest Comp25 apparatus (LTF Galileo Italy) applying a load of 613 N for 15 s and using a ball of 2.5 mm of diameter as indenter. The measure was repeated 5 times for each sample. Hardness values were used to evaluate the effectiveness of the heat treatments. In addition, the influence of porosity on this mechanical property was investigated in order to completely characterize the materials according to the specific manufacturing process.

3. Results and discussion
In Figures 1-2 the microstructure of DMLS and GC samples is reported in as-produced condition, as quenched at 510°C and after ageing at 160°C for 2 hours, as an example of the analysed conditions.

GC samples show the typical microstructure of casting products, characterized by the dendritic Al-rich phase (light grey in the images) surrounded by the modified eutectic, where some micro-shrinkage and hydrogen porosities were detected (Fig. 1a). The solution treatment, as predictable, induces spheroidization and coarsening of the Si particles as visible in Fig. 1b. The ageing does not remarkably affect the microstructure at this magnification. Similar evidences were gathered in the case of solution performed at 540°C and of ageing at 170°C.

Concerning the DMLS samples, the microstructure reveals the typical pools of semi-circular section along the building direction (Fig. 2a) and elongated in the deposition plane, according to the laser beam scanning paths. At higher magnifications, a very fine Al-rich cellular grain can be observed as well as very fine Si particles at the borders, as extensively described in literature [1, 23-24]. During the solution treatment, Si forms larger particles in the matrix (Fig. 2b), preferentially at the former pools boundaries, that now are no more visible [1]. Small porosities can be frequently detected in the DMLS samples, which can be due to the powder quality, hydrogen absorption, oxide inclusions, melt splashing, Marangoni flow during laser scans, etc. [4, 25-29]. The microstructure of DMLS samples after aging does not revealingly change (Fig. 2c).

A slight coarsening of the microstructure can be observed increasing the solution temperature as well as the ageing time, in agreement with other findings [22].
As-produced parts are characterized by very different performance compared to the heat treated ones (Fig. 3). In particular, the as-built alloy shows very high hardness, with a mean value of $125.20 \pm 1.05$ HB, while the as-cast one results $63.37 \pm 1.55$ HB. This superior resistance of the as-built material is related to the grain refinement and nano-sized eutectic Si obtained as a consequence of the fast cooling [22]. Additionally, the alloy experiences a “self quenching” phenomenon because of the layer by layer deposition process. In fact, the rapid cooling and the subsequent re-heating during the layer deposition can act like a T6 treatment, promoting the precipitation of Mg$_2$Si [30].

The subsequent solution treatment equalizes the hardness of the DMLS and GC alloys, independently from the tested temperature. In detail, after quenching, the hardness of the GC alloy remains almost the same as before; on the contrary, that of the DMLS alloy drops to about a half of the initial value.

This remarkable reduction in the DMLS alloy hardness is due to the dissolution of the previously formed precipitates combined with the possible increase of porosity defects.

To evaluate the effect of the porosity level on mechanical properties, the density of the samples was measured at each treatment condition. The as-built alloy shows a density of about $2.679 \ g/cm^3$, comparable to that of the as-cast part ($2.669 \ g/cm^3$). After quenching, the density of the GC alloy remains almost the same for both the solution temperatures, as shown in Table 2. For the DMLS, when the solution treatment is performed at higher temperature, a reduction in density of about the 5% is obtained.

**Table 2. Density of GC and DMLS alloys before and after quenching.**

|                  | GC [g/cm$^3$]       | DMLS [g/cm$^3$]  |
|------------------|---------------------|------------------|
| As-produced      | $2.669 \pm 0.006$   | $2.679 \pm 0.003$|
| As-quenched (from 510°C) | $2.672 \pm 0.007$   | $2.638 \pm 0.004$|
| As-quenched (from 540°C) | $2.671 \pm 0.008$   | $2.558 \pm 0.006$|

**Figure 2.** Microstructure of DMLS samples in as-built (a), quenched (b) and 2h-aged condition (c).

**Figure 3.** Brinell hardness of as-produced and as-quenched samples.
This remarks are further supported by the observation of the microstructure of samples in as-produced and quenched conditions. In fact, for GC samples, no significant increase of porosity can be identified, and both micro- and macro-porosities can be found (Fig. 4).

![Figure 4. Micrographs showing different porosity of GC samples in as-cast (a), quenched after solution treatment at 510°C (b), quenched after solution treatment at 540°C (c) condition.](image)

On the other hand, in Figure 5, it is possible to observe a remarkable increase in porosity after solution treatment of the DMLS samples, with a very high number of small pores, especially after treatment at 540°C, which is consistent with density measurements (Table 2).

![Figure 5. Micrographs showing different porosity of DMLS samples in as-built (a), quenched after solution treatment at 510°C (b), quenched after solution treatment at 540°C (c) condition.](image)

An explanation of this decrease in density for DMLS alloy after solution treatment can be found if the characteristics of this innovative manufacturing route are taken into account. As already mentioned, the used technology allows the formation of small porosities in the as-built part. In fact, due to the laser beam, the liquid pool reaches very high temperatures, promoting the dissolution of gas, such as hydrogen coming from the moisture on the powder particle surface as well as dissolved in the powder itself [31]. This results in the formation of pores in the solidified alloy. Additionally, because of the fast solidification and cooling, it is enhanced the entrapment of gas atoms into the aluminium lattice, since they have no time to diffuse.

During solution treatment, the diffusivity of these atoms in Al is known to increase remarkably [32-33] in comparison with that at room temperature. This can lead to the growth of pores size and number, due to their enrichment of gas atoms previously present in solid solution in the bulk, and, therefore, to the measured decrease in density. In particular, between 510°C and 540°C, an increase of about 30% in diffusivity coefficient can be calculated [33], explaining the worse microstructure quality.

A similar pronounced mechanism of porosity increase with heat treatment is instead not possible for cast parts, where the solidification rate is significantly lower and allows the rejection of gas atoms from the metal during solidification itself, resulting in the well-known gas porosities.

Hardness ageing curves in Figure 6 show the different response to ageing treatment of the two alloys. Values for 0 hours of ageing correspond to the samples condition after solution treatment and quenching.
In case of the GC alloy, the 510°C solution treatment results, in general, less effective than that performed at 540°C, requiring longer ageing time to reach comparable hardness values (Fig. 6a). For instance, after solution treatment at 510°C, a 6 hours ageing at 160°C is needed to obtain the same performance that can be reached after only 2 hours at 170°C when the solution is carried out at 540°C. The maximum hardness is achieved when a 540°C solution followed by a 170°C-8 hours ageing is done. 

The ageing curves of the DMLS alloy appear slightly different (Fig. 6b). In particular, it can be seen that after 1 hour almost the same hardness is obtained, independently from the analysed treatment conditions. The highest hardness is reached after 510°C solution followed by 160°C-6 hours ageing, while the treatment at 540°C-170°C shows the lower values, independently from the ageing time. Furthermore, it has to be noticed that the best heat treatment condition for the GC alloy is the worst for the DMLS one. In fact, the hardness at 540°C solution and 170°C-8 hours ageing treatment results 106.99±3.85 HB and 85.51±0.42 HB for GC and DMLS respectively. The reason of this different behaviour can be correlated to the presence of porosity defects, which were investigated by density measurements on aged samples for all the experimental conditions. Density values as a function of heat treatment parameters are shown in Figure 7.

Considering GC samples (Fig. 7a), the density does not vary remarkably during ageing treatment in comparison with that of the quenched condition. More interesting are the results for DMLS material (Fig. 7b). In fact, density values of aged samples after solution treatment at 510°C are quite constant for all the considered temperatures and durations. This implies that, in this condition, the ageing treatment does not induce a remarkable increase in porosity, with positive contribution to mechanical properties. This is in agreement with hardness data since the highest values were measured after solution treatment at 510°C (Fig. 6b). Instead, as already discussed for quenched condition, noticeable lower density values were found for samples treated at 540°C for all the investigated combinations of ageing time and temperature.
Additionally, samples density is quite constant for treatment at 160°C, while in the case of 170°C, the values vary without a clear trend. It is believed that heat treatment at highest temperatures induces a non-uniform distribution of porosities, which results in this scattered behaviour.

4. Conclusion
In this study, it was investigated the effect of different temperatures and durations of T6 heat treatment on the properties of AlSi10Mg produced by means of gravity casting and direct metal laser sintering techniques.

In the as-produced condition, the hardness of DMLS part results the double of the GC one, as a consequence of the very fine microstructure and of a sort of self-treatment experienced by the alloy during the layer by layer production. After quenching, the hardness of both the samples appeared almost the same, with a strong reduction of performance of the DMLS alloy, compared to the as-built condition. This significant drop in hardness for the DMLS alloy is clearly due to the microstructural changes related to the treatment itself, but also to the remarkable increase in porosity. The evolution of porosity can be ascribed to the pores already present in the as-built component, combined with the atoms of hydrogen in solid solution in the aluminium lattice, unavoidably present because of the manufacturing method. The maintenance at high temperature during the heat treatment allows the diffusion of gas atoms and formation and growth of porosities; moreover, the higher the temperature, the higher the increase of such defects. In fact, the solution treatment performed at 540°C resulted deleterious for hardness and porosity. The density of DMLS decreases of 1.5% after solution at 510°C and quenching and of 4.5% after solution at 540°C and quenching, as a consequence of the increase in hydrogen diffusivity of about 30%. Microstructural analysis confirmed the density measurements. On the other hand, in the case of GC alloy, such reduction was not revealed.

Concerning the temperature and duration of the ageing treatment, it was shown by density measurements and microstructural analyses that they affect less the porosity, for both the studied alloys.

Furthermore, it was noticed that for the DMLS part none of the investigated temperature and duration of the treatment allowed to reach the same values of hardness measured on the as-built samples, supporting the theory of the influence of the porosity on mechanical properties.

Finally, it was observed that the optimized heat treatment parameters for the AlSi10Mg GC alloy result the worst for the DMLS one, showing the need of the definition of proper heat treatment procedures specifically tailored for AM parts.

5. References
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