A comparative study of the effects of thermal treatments on AlSi10Mg produced by Laser Powder Bed Fusion.

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ABSTRACT

The components produced by laser powder bed fusion (LPBF) generally require specific heat treatments in order to release the residual stresses induced during the additive manufacturing process. Post-processing treatments also play a significant role in obtaining components whose microstructure and characteristics are homogeneous and tailored for specific applications. A comparison of the mechanical features resulting from different heat treatments requires that a material showing the same initial mechanical features and microstructure is investigated. In the present work the effects of a number of different thermal post-processing treatments are compared: AlSi10Mg parts processed by LPBF underwent various thermal treatments such as stress relieving, annealing at high temperature and T6 treatments. The microstructure variation as a function of the applied temperature was correlated to the material mechanical behaviour in term of hardness and tensile strength; impact properties were also evaluated. The thermal evolution of the system was then studied through differential scanning calorimetry and x-ray diffraction analyses.

Keywords. Laser processing; Mechanical Properties; Microstructure; Metallography

1. Introduction

Additive manufacturing (AM) techniques have attracted increasing interest in the scientific community due to the possibility of using a *layer-by-layer* strategy to produce near full-density components with complex geometries. In addition to the advantage of design freedom, this technology saves material and avoids the production of scraps and waste. Different metal AM technologies are currently available and find applications in many fields such as automotive, aerospace, orthopaedic implants etc. [1–4]. Among these technologies, Laser powder Bed Fusion (LPBF), also known as selective laser melting (SLM) is currently one of the most studied. LPBF is a powder bed fusion process that uses a laser source to selectively melt regions of deposited powder layers, according to a computer aided design (CAD) project. One of the most interesting aspects of this process is that it provides a very fine microstructure compared to that obtained using more conventional processing methods. Moreover, it is well known that this particular microstructure greatly influences the mechanical behaviour of the material. LPBF can be applied to different metallic powders such as aluminium alloys, titanium, stainless steel and nickel. Aluminium alloys are the second most used metals, after steels. For this reason, the production of Al components through the LPBF process obtains low cost and high-quality parts for many different applications. Very commonly used aluminium alloys are those based on the Al-Si system, as they are characterized by good castability, specific strength and good corrosion resistance [5–9] . In this context, the hypoeutectic AlSi10Mg alloy is frequently used in both foundry and additive manufacturing technologies; the great interest in this alloy is moreover confirmed by many scientific papers available in the literature.

The components produced by metal additive manufacturing generally need specific heat treatments aimed at releasing the residual stresses coming from the production process. Different kinds of residual stresses (RS) can in fact be present in as-built sample. According to Bartlett et al. [10], they can be classified on the base on the length scale they operate. Macroscopic RS act on the scale of the component geometry and can cause distortion phenomena; they are the most discussed in the literature due to the strong effects they have on mechanical properties of the as-built material. In addition, local stresses which act on individual grain scale or atomic scale stresses can also be present; however, they are only rarely investigated because of the

difficulty to measure them. The stressed state of the material limits its application in the as-built condition. The attention of many researchers has been focused on different thermal treatments according to two approaches: the first one involves the adoption of the well-established heat treatments commonly used for cast materials with similar compositions, while the second one consists in the optimization of thermal treatment conditions (in terms of temperature and duration) as well as in the investigation of the effects of the applied conditions on both microstructure and mechanical properties.

The first approach is questionable because the microstructure of AM components is very different from that of cast parts. Aboulkhair et al. [11] underlines, as an example, the different durations required for solution heat treatments (SHT): for cast alloy the solution heat treatment (SHT) needs a shorter time to obtain a fine and homogeneous microstructure, while for LPBF parts a longer duration is required to stabilize the microstructure and improve the mechanical behaviour of material. According to the second approach, modified thermal treatments have been proposed in the literature with the aim of finding experimental conditions suitable for LPBF components.

In as-built AlSi10Mg parts processed by LPBF, unlike cast parts, silicon is mainly solubilized within the aluminium matrix before the thermal treatment. The annealing cycle allows the system to move toward thermodynamic equilibrium conditions; so, the residual stresses arising during LPBF process, which can cause distortions or microcracks, are relieved. While annealing is generally used to promote alloy ductility, the strengthening of alloys is reached through a precipitation-hardening mechanism. Strengthening is achieved by a solution heat treatment followed by quenching and artificial ageing (AA) that induces the precipitation of intermetallic compounds from the metastable supersatured solid solution of alloying elements in aluminium. The duration of the solution and ageing treatments is critical; under-ageing and over-ageing must be avoided because they can cause a decrease of hardness [11] and reduce mechanical properties.

Many authors focus their attention on low temperature annealing [12–14], stress relieving [15–17] or T6 treatments, but the proposed conditions for these thermal cycles are quite different in term of both maximum temperature reached and duration [16,18–23]. In addition, different mechanical features (mainly hardness and tensile strength) were considered by different authors to evaluate the treatment effectiveness. However, other mechanical properties (such as toughness, fatigue and creep) have been less frequently investigated [24,25]. It is moreover important to take into account that the mechanical properties of LPBF pristine parts depend on the adopted processing parameters; the thermal treatments investigated in the literature were generally carried out on materials that showed different characteristics in the as-built condition. For all these reasons, a comparison of data reported in the literature could be confusing.

Many papers report the mechanical behaviour of LPBF AlSi10Mg components but, according to the authors' knowledge, few studies [26–29] are focused on impact behaviour and this was only rarely investigated after performing a thermal treatment. Some authors [26,27] have studied the impact behaviour of AlSi10Mg alloy in as-built condition as function of the building orientation or of the different surface finishing. However, only few studies report variation of the impact properties after heating the material at high temperature: Girelli et al. [28] evaluated the effect of T6 heat treatment (solution at 540°C, quenching and ageing) and Hot Isostatic Pressing on impact behaviour of AlSi10Mg parts. Moreover, Fulcher et al. [29] compared the mechanical properties of AlSi10Mg and Al6061 alloys processed by DMLS after stress relieving and after other heat treatments (HIP, solution, ageing); however, no information about the temperature and duration of the applied thermal treatments were provided.

To summarize, in spite of the wide literature on the thermal treatments of Al-Si alloys processed by LPBF it is still puzzling to definitely quantify their effect on the mechanical features. The comparison of experimental results reported in different studies can be difficult due to different factors: they were generally referred to few properties (mainly hardness and tensile properties), they were obtained after manufacturing using different processing parameters, and thermal treatments were carried out in different conditions (such as different isotherm duration). On the contrary, the design of a heat treatment process should consider the same starting microstructure and initial properties in order to evaluate their variation.

The present work aims to present a systematic study which compares mechanical properties after different heat treatments, such as stress relieving, annealing at high temperature and T6 treatments, performed on samples fabricated using the same processing parameters. The microstructure variations resulting from the

performed treatments were correlated to mechanical properties in term of hardness, tensile and impact properties. X-ray diffraction analyses and calorimetric studies were also carried out in order to deeply investigate the evolution of the system after different applied thermal conditions.

2. Materials and Methods

The samples under investigation were manufactured by the LPBF technology starting from gas atomized AlSi10Mg powder provided by EOS (actual composition (wt.%): 89.25 Al, 9.70 Si, 0.44 Mg, 0.38 Mn, 0.20 Fe, 0.01 Ti, <0.01 Cu, < 0.01 Zn). The LPBF process was carried out using an EOS M290 system equipped with a 400W Yb-fibre laser. All the specimens under investigation were produced by using the same processing parameters: a laser beam with a power of 370W was scanned across the powder bed, kept at 165°C, with a speed of 1300 mm/s. The hatching distance between adjacent scan tracks was set to 0.19 mm, whereas the layer thickness was fixed at 30 µm.

Samples were prepared with suitable shape and size for the different mechanical tests: hardness, tensile and impact tests. Some specimens were submitted to different thermal treatments to investigate their effect on both microstructure and mechanical features. Stress relieving was performed at 300°C for 2 hours, as recommended by LPBF powder producers, with the aim to relieve residual stresses developed during the manufacturing process [30]. Annealing were carried out at 500°C and at 550°C; the maximum temperatures were maintained for two or six hours. For stress relieving and annealing treatments air cooling by removing samples from the furnace was adopted. T6 treatment involved solution at 520°C or 550°C for 2 hours, water quenching and artificial ageing at 160°C or 180°C for 30 minutes, 1, 3, 6, 9, 12, 15 and 18 hours. After each selected ageing time, the samples were removed from the furnace and cooled down to room temperature on air. For all the heat treatments a heating rate of 200°C/h was adopted. All the treatments were performed in the same oven (Naberthem GmbH RHTC 80-710/15, Lilienthal, Germany) under flowing argon atmosphere in order to prevent any oxidation of the samples.

The sample microstructure before and after thermal treatments was investigated: low magnification images were obtained by using an optical microscope (OM, Leica DMI 5000 M), while field-emission scanning electron microscopy (FE-SEM, ZEISS MERLIN equipped with EDS Oxford INCA) was used to examine fracture surfaces and the microstructure of polished sections at high magnification. In this case, a chemical etching with Keller's reagent for 30 s was used to put in evidence the microstructure of the material.

A digital image analysis method was used to study the material porosity. This method quantified the average surface fraction of pores from the analysis of cross-section views of as-polished (not etched) samples. Twenty optical micrographs at fixed magnification were taken in different parts of the cross-section, then they were converted to binary imagines, where the black pixels represented the pores. The images were processed by means of Image J. software. The results were averaged, and the standard deviation values were calculated.

The presence of crystalline phases was identified by using X-ray diffraction (Panalytical X'PERT PRO PW3040/60, Cu Kα radiation at 40 kV and 40 mA, Panalytical BV, Almelo, The Netherlands). The lattice parameter of Al in as-built alloy and after performing different heat treatment was calculated by using $\cos 2\Theta/\sin\Theta$ extrapolation method.

Differential scanning calorimetry (Pyris 1 DSC, Perkin Helmer Italia Spa, Milano, Italy) was used for better understanding phase transformations and precipitation phenomena occurring when material is heated at high temperature. Samples of about 30 mg were placed in an aluminium crucible and heated to a temperature range from 100°C to 450°C in argon atmosphere. Calibration was done by using pure In and Zn standards and verifying that their melting point experimentally measured in DSC was 156.6 °C ± 0.3 °C and 419.5°C ± 0.3°C respectively.

The non-isothermal integral isoconversional method proposed by Flynn and Wall [31,32] and Ozawa [33] was adopted to study the kinetics of phase transformation in AlSi10Mg alloy. This method is based on DSC temperature runs, during which samples are heated at constant heating rates and the transformations involving exothermal or endothermic effects are observed. For each DSC run and for each involved transformation, the temperature corresponding to a pre-fixed transformed fraction of the material under investigation (α) is a function of the heating rate according to the equation (1):

$$\log \nu = -0{,}4567 \cdot \frac{E}{R \cdot T} + \text{costant} \qquad (1)$$

Where ν is the heating rate, E is the activation energy of the phase transformation investigated, R is the universal gas constant, and T is the temperature which corresponds to the pre-fixed fraction of transformed material α. For instance, the temperature of the maximum of symmetrical DSC peaks corresponds to α=0.5. According to Ozawa [33], the constant is equal to $-2{,}315 + \log\frac{Z \cdot E}{R} - \log g(\alpha)$, where the integral function g(α) assumes a constant value for the considered α value. When the heating rate in logarithmic form is plotted as a function on 1/T (T corresponding to a fixed fraction of material conversion; in this study, a temperature corresponding to a conversion fraction of 0.5 was used), a straight line is obtained; from its slope the activation energy is determined. The activation energy for transformations observed in DSC traces was calculated by submitting samples to DSC runs with heating rates of 5, 10, 20 and 30 °C/min. To this purpose, the DSC analysis was performed on specimens in different conditions: as-built, solution treated at 550°C for 2 hours and water quenched, solution treated/quenched and aged at 180°C for 6 hours.

Cubic samples with size 20mm x 20mm x 20mm were prepared in order to investigate the microstructure and evaluate their hardness. After performing different kinds of thermal treatments (stress relieving, annealing, precipitation hardening), the samples were polished by using SiC abrasive papers up to 1200 grit and their hardness was measured on the cross sections both parallel and perpendicular with respect to the building direction Z. In order to investigate the microstructure of AlSi10Mg alloy, a further polishing of samples surfaces with SiC papers up to 4000 grit and diamond pastes, with size of 3 μm and 1 μm respectively, was performed. The effect on AlSi10Mg alloy of thermal treatments and ageing duration was investigated through Brinell hardness measurements, which were performed on polished cross sections using an EMCO TEST M5U-030 durometer (Prüfmaschinen GmbH, tungsten carbide indentator with a diameter of 2.5mm). The tests were carried out applying a load of 62.5 kg maintained 10 s. Five measurements were done on each cross section, and hardness values were then averaged. Hardness results were also used as indicator of the strength of the material.

Cylindrical samples were built and then machined in order to obtain standard specimens for tensile tests with a total length of 48 mm, gauge length of 40 mm and a diameter of 8 mm (BS EN ISO 6506-1:2014). These samples were built both vertically and horizontally within the work chamber (samples respectively labelled as Z and XY in the text). The two kinds of samples mainly differed because the sequence of the stacked layers and the layer interfaces were perpendicular to the sample length (that is to the growing direction) in "Z" samples, while both layer sequence and interfaces were parallel to the sample length in "XY" samples. Tensile tests at room temperature were performed by using a Zwick 1474 dynamometer (Ulm, Germany), with a strain rate of 0.00025 s^{-1}. Sets of 5 samples were tested for each treatment condition: as-built, after stress relieving, after annealing at 500°C, and after a selected T6 thermal treatment (solution at 550°C for 2 hours, quenching in water and artificial ageing at 180°C for 6 hours).

Additional specimens were produced (according to ASTM E23-18 standard) directly in the shape required for impact Charpy test: V-notch specimens with length of 55mm, width and thickness of 10 mm were prepared.

Charpy impact tests were performed on sets of 5 samples as well (in as-built conditions and after each investigated heat treatment) by using an instrumented pendulum with an available maximum energy of 25 J (ATS FAAR Industries, Segrate, Italy).

3. Results and discussion

3.1 Microstructure

The peculiar microstructure of AlSi10Mg cubic samples produced by LPBF is shown in Figure 1. These images obtained using optical microscope show samples in as-built conditions; the microstructure was observed on sections taken in directions parallel and perpendicular to the building direction Z.

Revised Figure 1

The microstructure of the parallel cross section (Figure 1A) shows typical melt pools with a semi-circular profile due to the gaussian distribution of laser beam energy involved during the production process. It is possible to observe an overlapping between the pools in each single layer scanned by the laser and between

the pools placed inside adjacent layers. In fact, in order to well consolidate the layer-by-layer structure, it is required a partial re-melting of a solid material portion which lies just below the powder layer interested by the laser scanning path.

Considering the perpendicular plane (Figure 1B), the microstructure looks different, showing overlapped scan tracks with elongated shape and with different orientations; this is due to the adopted laser scan strategy, which involves the rotation of 67° between two adjacent layers subsequently stacked. The width of the scan tracks is in the range between 100 and 200 µm, which corresponds to the extent of melt pools. Figures 1C and 1D show, for the two cross sections, a single melt pool at higher magnification after etching with Keller's reagent. The microstructure in Figure 1C is the result of a directional solidification. During the LPBF process, in fact, the interaction between the laser beam and the powder causes the formation of a thermal gradient due to the lower temperature of the underlying powder. The melted metal cools down very fast (~10^6 °C/s) leading to the formation of a very fine structure constituted by Al-rich cells surrounded by Si-rich eutectic structures in the intercellular regions (Figures 1C and 1D). The size of cellular α-Al grains decreases moving from the edge to the inner part of the melt pool due to the different cooling rate. The so called "fine cellular zone" consists of a finer microstructure which is characterized by cells in the range from 0.4 µm to 0.8 µm; the "coarse cellular area" presents cells double in size respect to the previous ones (from 1 to 2 µm). This dimensional variation is due to both the gaussian distribution of laser beam energy and the cooling rate, which is different moving away from the previously solidified layer. According to Fousovà et al. [14] magnesium was detected by EDS preferentially within the intercellular network, where it can react mainly with Si. In binary systems the solubility of silicon in solid aluminium decreases from 1.65 wt.% at 577°C to about zero at room temperature, while that of Mg decreases from 15 at. % at 450°C to less than 1% at room temperature. However, when considering the Al-Mg-Si ternary system the solubility of Mg and Si in solid Al, which is just above 1 at. % at 627°C, becomes negligible at 180°C under equilibrium conditions (less than 0.05 at. %) [34]. Therefore, there are not thermodynamic reasons for the presence of both silicon and magnesium inside the cellular α-Al grains after solidification. On the other hand, selective laser melting involves dramatically high cooling rate from the molten state, and then deviations from equilibrium conditions are expected; nonetheless, the eutectic liquid solidifies after the primary α-aluminium cells, and for this reason the eutectic structure contains more alloying elements (magnesium and silicon).
In both microstructures little black spots can be observed, which reasonably correspond to small porosities, that are currently present in parts produced by LPBF. According to some authors [35–38], they can be caused by hydrogen absorption, melt splashing or Marangoni flow occurring during the building process. The porosity evaluated by image analysis on the section parallel to the building direction was slightly higher with respect to that observed for the perpendicular one, the two sections showing porosity values of 0.204 % ± 0.091 and 0.135 % ± 0.062 respectively. Anyway, the porosity seemed rather homogeneously distributed and the porosity level rather low. Tradosky et al. [39] also measured (by using a similar images analyses method) a higher porosity degree in the section parallel to the build direction with respect to that observed within the perpendicular one (about 1.7% and around 1% respectively).
Tentatively, it can be inferred that pores form more easily at the interfaces between layers that subsequently are molten by the laser action and therefore, they are preferentially located at melt pool boundaries.

Revised Figure 2.

After performing a stress-relieving heat treatment at 300°C for 2 hours, only slight microstructure variations can be appreciated with respect to the as-built sample. The micrograph at low magnification (Figure 2A) does not put in evidence any significant differences: evident traces of pre-existing laser scan tracks are still present. Moreover, at higher magnification (Figure 2B), the treated material still shows a cellular-like microstructure, but the continuity of the eutectic network, previously observed in as-built samples, is now interrupted. In fact, it is possible to observe the precipitation of silicon grains uniformly dispersed within the aluminium matrix, with size in the range from 100 to 300 nm. This microstructure change is very likely due to the growth of both aluminium and silicon crystals, which was promoted by the hold at 300°C. At this temperature the system evolves towards equilibrium conditions, which entails the decrease of the amount of silicon present inside the aluminium lattice and the precipitation of this element

along the primary Al-eutectic zone interface. A similar microstructure has been previously observed by Fiocchi et al. [12] for AlSi10Mg parts which were isothermally treated at 263°C, 294° and 320°C. Also in this case, the initial cellular structure was maintained to some extent despite the silicon segregation and the grain growth.

The annealing performed at higher temperature (500°C) for the same duration (2 hours) induces more significant segregation of Si from the supersatured aluminium matrix and grain growth. The melt pools coming from the laser scan tracks can be hardly distinguished (Figure 2C). The resulting microstructure shows Si particles with polygonal shape which are homogeneously dispersed in the Al matrix (Figure 2D and E). The size of Si precipitates (from 1µm to 4µm) is one order of magnitude larger with respect to that observed for stress relieved samples. Moreover, the presence of few acicular precipitates can be observed. EDS analyses performed on these particles evidence the presence of not negligible amount of iron in addition to Al and Si (Figure 2F). The strengthening precipitate in Al-Si-Mg alloys is Mg_2Si, but also Al-Si-Fe based precipitates were previously observed in heat treated AlSi10Mg alloys, and their role in the enhancement of the mechanical properties have been previously discussed [16].

A little increase of annealing temperature from 500°C to 550°C causes the further increase of the size of Si particles (average size from 2 µm to 5 µm) because coalescence and Ostwald ripening occur [12]. This growth of Si grain size is consistent with the evolution of the microstructure of as-built samples (made of supersaturated solid solution of silicon inside the aluminium lattice) toward a thermodynamic equilibrium condition. In fact, increasing the annealing temperature promotes a quicker and important silicon precipitation and the coalescence and growth of silicon grains inside the aluminium matrix [20]. The progressive precipitation of silicon particles also results in the decrease of their concentration, that can be clearly appreciated by comparing the microstructure observed after stress relieving at 300°C (Figure 2B) with the microstructures resulting from annealing treatments performed at 500°C (Figure 2D).
The above-mentioned transformations modify the very fine microstructure of as-built samples; after thermal treatments it in fact becomes more similar to that observed for the AlSi10Mg alloy processed by conventional casting methods.

Figure 3.

Figure 3 compares microstructures observed after a solution treatment at temperature of 550°C for 2 hours, quench in water to room temperature without (Figure 3A and 3B) and with (Figure 3C and 3D) a following artificial ageing at 180°C for 6 hours (which allows to obtain the hardness peak, as reported in section 3.4). Microstructures observed after the same kind of treatments, but at lower solution temperature of 520°C are very similar. Figure 3 shows that after SHT or T6 treatments the microstructure is very similar, consisting of Si particles with polygonal shape which are homogeneously dispersed within the Al matrix. In addition, the size of silicon grains (around 2-5 µm after SHT) does not significantly change during the ageing step. In every case, the silicon precipitates dimension was comparable to those observed after annealing at 520°C, which means that two hours of hold at above 500°C allow to complete the silicon precipitation and to bring the material to equilibrium conditions.

In addition to silicon polygonal particles, also acicular particles were found after the solution treatment. After solution and quenching precipitates are few micrometers long and less than 500 nm wide; they retain these dimensions during the artificial ageing, which only causes the increase of their concentration. EDS analyses on these precipitates showed that they are aluminium based and rich in Fe and Si, while the Mg presence was not found. It can be inferred that these precipitates can strengthen the matrix. The formation of Mg-Si strengthening phases (like Mg_2Si), expected after the solution, quenching and ageing of Al-Si-Mg alloys, was not appreciated by SEM-EDS probably because only Guinier-Preston zones or precipitates semi-coherent with aluminium form.

3.2 X-ray diffraction

Figure 4A compares XRD spectra of AlSi10Mg cubic samples in as-built condition (Figure 4a and 4b) and after the different heat treatments under investigation (Figure 4 c-g). All spectra are normalized with respect to the highest intensity peak of Al (111).

Only two crystalline phases are always present in these spectra: Al and Si, which are respectively the main component and the main alloying element of AlSi10Mg alloy. No additional phases were observed after the thermal treatments of stress relieving, annealing, solution and ageing, whatever the thermal cycle experienced by the material was. This is not surprising because the amount of strengthening precipitates formed during annealing or ageing is small, and very likely below the detectability limits of XRD technique. In the case of as-built specimens, XRD spectra were obtained on cross-sections placed perpendicularly and parallel with respect to the building direction (Figure 4a and b). The ratio between intensities of aluminium (111) and (200) peaks changes in these two XRD patterns. In particular, the intensity of (200) peak is appreciably higher than that of (111) peak in the spectrum collected on the perpendicular section, while this trend is not evident in the pattern recorded for the parallel section. Some authors [13,40–42] reported the presence of texture in Al-Si alloys produced by LPBF as a consequence of the layer-by-layer structure obtained by LPBF and the resulting fast cooling rate. The directional solidification occurring during LPBF process causes, not only a morphological texture involving the development of the characteristic melt pools, but a crystallographic texture too. In aluminium grains formed during LPBF process, the (100) crystallographic direction is parallel to the main axis of columnar grains and therefore to the grain growing direction as well. As a consequence, (200) crystallographic plane of Al is preferentially placed perpendicular with respect the growing direction of the crystals; this results in an increase of the intensity of the relevant peak in the XRD pattern of the perpendicular cross-section.

Revised Figure 4

Table 1. Percentage ratio between the intensities of Si (111) and Al (111) peaks and FWHM (Full Width at Half Maximum) after different thermal treatments.

Thermal treatment	**Si (111) / Al (111) [%]**	**FWHM [°] Si(111)**	**Si grain size distribution**	**Al lattice parameter (nm)**
As-built	2.9	0.610	< 100 nm	0,40489
Stress relieving 300°C_2h	6.7	0.267	100-300 nm	0,40510
Annealing 500°C_2h	23.5	0.094	1-4 µm	0,40512
Annealing 550°C_2h	26.5	0.100	2-5 µm	0,40515
SHT 550°C_2h	21.4	0.100	2-5 µm	0,40513
SHT 550°C_2h + AA 180°C_6h	18.9	0.119	2-7 µm	0,40513

Table 1 shows that the ratio between the intensity of (111) peak of silicon and that of (111) peak of aluminium is higher in samples that experienced higher temperatures during the thermal treatment; this is also put in evidence from the variation of intensity of (111) Si peak in Figure 4B. This outcome is due to the effect of high temperature treatments that promote the modification of the microstructure towards conditions of thermodynamic stability; these are reached more quickly at high temperatures.
Actually, the solubility of silicon in the aluminium lattice in ternary Al-Si-Mg alloys becomes negligible when temperature decreases [34]. For this reason, almost all silicon contained in the AlSi10Mg alloy (namely 9.70 % wt.) must segregate (at every temperature between the melting/solidification point and room temperature) from the aluminium-based supersaturated solid solution, when suitable conditions for silicon diffusion and nucleation of silicon crystal exist. In as-built samples an important fraction of the total silicon is contained within the aluminium crystal lattice as the Si/Al peaks intensity ratio is about 2.9% only, but this ratio increases up to about 6.7% after stress relieving at 300°C and up to 26.5% after two hours of annealing at 550°C because of the progressive formation of silicon crystals. This precipitation occurs not only during the isothermal annealing, but also during the slow cooling at the end of the treatment. On the contrary, a higher cooling rate after annealing (quench) limits the silicon precipitation, as shown in Table 1 where a Si/Al peaks intensity ratio of about 21.4% after the quench is reported. The Si/Al intensity ratio peak slightly decreases after ageing at 180°C for 6 hours, since during ageing silicon is engaged in the formation of strengthening precipitates (namely Mg_2Si and acicular Al-Si-Fe).

This is also confirmed by the determination of lattice parameter of α-Al phase after different thermal treatments. According to the literature [43] the lattice parameter for AlSi10Mg alloy in equilibrium conditions is equal to 0.40515±0.0003 nm. In addition, the lattice parameter decreases when Si dissolves in the Al lattice and then, the lattice parameter of the solid solution depends on the Si concentration**.** In fact, the lattice parameter increases after stress relieving because the treatment at 300°C for 2 hours involves the precipitation of silicon from Al phase. More important precipitation of silicon particles from the supersatured solid solution occurs after treatments at higher temperature (annealing, solution treatment and quenching, and T6 treatment).

In addition, the FWHM (Full Width at Half Maximum) of the (111) Si peak has been considered in order to highlight the connection between the size of Si peak found from microstructural observation and the shape of XRD peaks. The value of FWHM is maximum in the XRD patterns of as-built samples, where very fine Si crystals are present in the microstructure. The decrement of FWHM value after treatments at high temperature is in good agreement with progressive growth of the silicon particles observed by microscopy.

3.3 Differential Scanning Calorimetry

It is generally accepted that the ageing of Al–Si–Mg alloys occurs according to the following precipitation sequence [6,8,44,45] :

α-SSS → GPZ → β'' (GPZ II)→ β' → β (stable Mg_2Si)

where α-SSS represents the supersaturated solid solution, GPZ are primitive Guinier–Preston zones, β'' corresponds to grown GP zones and β' is a semi-coherent metastable phase. The formation of these precipitates, with needle-like and rod-like shapes respectively, occurs by nucleation and their growth is diffusion-controlled. β is the equilibrium Mg_2Si phase.

The DSC technique proved to be a powerful tool for investigating the ageing sequence of Al-Mg-Si alloys with rather low silicon content processed by casting [6,46]. In fact, the whole precipitation sequence can be appreciated in the DSC traces obtained after solution treatment and quench in water.These DSC traces show very weak phenomena for primitive GP zone formation and reversion just over 100°C and 200°C respectively, a sharp exothermal peak just above 250°C due to β" precipitation, an exothermal peak at around 300°C of β' formation (semi-coherent Mg_2Si) and an exothermal effect at higher temperature related to the formation of stable Mg_2Si. The grown GPZ (called GPZ II or β") are considered the main hardening phase, that can be detected by TEM after artificial ageing at the hardness peak.

The AlSi10Mg alloy presently under investigation is expected to show even a more complex DSC trace in as-built condition because of the very high silicon content (about 10%) and the precipitation of silicon grains from the supersatured solid solution. In addition, a comparison between DSC experiments performed on materials with different compositions does not seem simple as a different composition can affect the position of peaks in the DSC trace.

The precipitation of strengthening phases in AlSi10Mg alloy was presently investigated by DSC, and by testing samples in different metallurgical states. In particular, the different DSC traces corresponding to the as-built condition, solution at 550°C for 2 hours followed by water quenching, and precipitation hardening achieved by a final artificial ageing at 180°C for 6 hours were compared. Figure 5 shows the DSC scans of these samples obtained at a heating rate of 10°C/min. The curves are shifted along the Y-axis in order to avoid their overlap. Moreover, Table 2 and Table 3 report the peaks recorded for the three kinds of samples at different heating rates and the activation energies calculated on the base of Ozawa's method. The correlation coefficient (R^2 in Table 3) resulting from the fitting of experimental points in the Ozawa's plot can be taken as an indicator of the experimental error in the calculation of the activation energy.

Revised Figure 5

Table 2. DSC peak temperatures recorded at different heating rates for AlSi10Mg samples in as-built condition, after SHT and after SHT followed by ageing.

	SHT 550°C			SHT 550°C + AA 180°C		AS-BUILT	
Heating rate	**A**	**B**	**C**	**D**	**E**	**F**	**G**
[°C/min]	[°C]	[°C]	[°C]	[°C]	[°C]	[°C]	[°C]
5	216.4	271.7	330.2	264.5	317.0	245.5	320.7
10	232.2	286.8	350.5	276.7	336.8	259.4	329.4
20	247.4	302.6	362.4	293.0	351.0	273.0	339.3
30	259.3	314.2	371.1	301.4	357.6	279.8	345.7

Table 3. Activation energy associated to different exothermal phenomena in DSC thermograms.

SHT 550°C			SHT 550°C + AA 180°C			AS BUILT		
Peak	Activation energy [kJ/mol]	R^2	Peak	Activation energy [kJ/mol]	R^2	Peak	Activation energy [kJ/mol]	R^2
A	88	0.9996	**D**	119	0.9799	**F**	118	0.9912
B	96	0.9996	**E**	120	0.0092	**G**	211	0.9978
C	127	0.9902						

3.3.1. Comparison of DSC traces of AlSi10Mg samples after solution treatment / quenching (curve 1), and after solution / quenching and artificial ageing (curve 2).

The DSC curve of as quenched AlSi10Mg samples shows three peaks (curve 1 of Figure 5): the one with highest intensity (peak A) has a maximum between 216.4°C and 259.3°C depending on the different heating rates; the associated activation energy is 88 kJ/mol. According to literature data [6,46] it seems reasonable to attribute this exothermal phenomenon to the formation of the strengthening β''phase containing Mg and Si. In addition, this peak disappeared in the DSC trace recorded after solution, quenching and artificial ageing at 180°C for 6 hours which cause the precipitation of the strengthening β" phase (curve 2 in Figure 5).

The peak B (curve 1 of Figure 5) in the temperature range from 271.7°C to 314.2°C with an activation energy of 96 kJ/mol can be associated to the precipitation of β' phase that does not form during artificial ageing in the conditions here adopted. In fact, it does not disappear in the DSC pattern of the AlSi10Mg sample after ageing at 180°C (see peak D in curve 2 of Figure 5). However, the position of peak D (temperature ranging between 264.5°C and 301.4°C) slightly changed with respect to that of peak B. Nonetheless, in our experience, the temperature at which a precipitation phenomenon occurs can be also affected by the previous thermal history of the sample, and therefore the precipitation of a specific phase can be affected by the sample state before the DSC run. Very likely precipitation can occur more easily (at lower temperature) if the sample microstructure previously advanced through the precipitation sequence. For this reason, the precipitation of β' could occur at slightly different temperatures for a solution treated-quenched sample and for a sample aged to the hardness peak. In the first case all the precipitation sequence has to occur during DSC run while in the second case the precipitation of β" completely occurred before the DSC test and the β' formation entails a re-arrangement of the β" previously formed. Finally, a very weak signal in the range between 330.2°C and 371.1°C was also observed.

These results (curve 1 of Figure 5) are also in good agreement with the literature dealing with DSC traces of solution and quenched AlSi10Mg alloy produced by additive manufacturing. Girelli et al. [20] recorded a similar DSC trace for AlSi10Mg samples treated at 540°C for 1 hour and then water quenched. Also these authors observed the presence of three exothermal peaks for so treated samples: the first one at 240-284°C corresponds to peak A of β'' formation in curve 1 of Figure 5 (in the range between 216.4°C and 259.3°C); the second at 298-329°C is about in the same temperature range of peak B of β' formation (271-314°C); the third peak was observed at 321°C – 368°C, that is in the same range of peak C (at 330-371°C), and was attributed by Girelli et al. to the formation of β-Mg_2Si phase.

The formation of the stable Mg_2Si phase should occur during the DSC run of samples after solution and quenching as well as during the DSC run performed after artificial ageing. In Figure 5, the peak C (observed in the quenched sample, curve 1) and peak E (present in the DCS trace of artificially aged specimens, curve 2) show their maximum in the range from 330.2°C to 371.1°C and 317°C to 357.6°C respectively and show a similar activation energy of 127 kJ/mol and 120 kJ/mol respectively. Therefore, they could be attributed to the precipitation of β phase.

Conclusively, literature data about the precipitation of β", β' and β well agree with the present experimental results, which show that after solution and quenching the LPBF AlSi10Mg alloy presents the same ageing path of similar alloys processed by casting.

The possible presence in the DSC curves of both solution treated/quenched or artificially aged AlSi10Mg of a significant thermal effect related to silicon precipitation is doubtful; in fact, the activation energy values for Si precipitation which are reported in some literature are in the range from 124 kJ/mol to 165 kJ/mol [12,20,41] or even higher [47]. These activation energy values are higher than that experimentally observed for peaks C and E (that we attribute to β formation instead). In addition, X-ray diffraction and microstructure investigations discussed in the previous sections show that after solution treatment/quenching as well as after artificial ageing only little amount of silicon is still present in the solid solution, and therefore during the DSC scan of samples in these treatment conditions only little amount of silicon could segregate from the solid solution to form silicon grains. The relevant thermal effect very likely could be overlapped to the peak for β formation. On the contrary, a well different scenario occurs when as-built samples are submitted to DSC analysis, as discussed in the following.

3.3.2. DSC traces of as-built AlSi10Mg samples (curve 3).

In as-built samples silicon is almost completely contained in the aluminium supersaturated solution (see Figure 4 and Table 1); therefore, the diffusion of silicon from this solution during heating and the formation of increasing amounts of silicon grains are expected to occur during the DSC run.

Actually, two exothermal peaks were found in the DSC thermogram of as-built specimens (Figure 5, curve 3): the first one has its maximum at about 259.4°C and an activation energy of 118 kJ/mol, while the second peak is placed at 329.4°C and shows an activation energy of 211 kJ/mol. Both these phenomena are exothermal, but Figure 5 shows as the second event is much more significant with respect to the first one. In the literature there is no agreement about the attribution of these two exothermal effects to specific transformations.

All the thermograms of as-built AlSi10Mg samples manufactured by LPBF show the presence of two peaks placed at temperatures which are similar to those presently observed [12,20,43,48]; however, the first peak at about 250°C is frequently stronger than the second one, while we observed the contrary.

The temperature and the activation energy for peak F in curve 3 (Figure 5) are close to those associated by different authors [6,49,50] to the precipitation of β'' phase in Al-Si-Mg alloys produced by casting processes. However, the temperature of the maximum of peak F and its activation energy do not match well with those we observed for β'' in the DSC traces of AlSi10Mg after solution and quenching; in fact, the temperature is intermediate between that of peak A, which was attributed to β'' formation, and that of peak B, which was attributed to β'. The shift of the peak temperature for β'' formation with respect to the values observed for the precipitation of this phase after solution and quenching can be tentatively attributed to the very different microstructure of the as-built sample, which is characterized by very high residual stresses and the very high concentration of silicon in the supersaturated solid solution. These modifications of the crystalline structure could well have some influence on the diffusion processes.

Also Girelli et. al [20] reported in the DSC of as-built samples the presence of two peaks at 240°C - 284°C and 298°C – 329°C (on the base of the heating rate), and calculated activation energies of 89 kJ/mol and 163 kJ/mol respectively. On the base of both these data, they attributed the first exothermal event to the precipitation of β'' phase. Fiocchi et al. [12] observed for the as-built material the presence of two peaks in the temperature range from 226°C to 270°C and from 295°C to 342°C respectively. Also these authors, on the base of both peak temperature and activation energy (110 kJ/mol) assigned the first event to the precipitation of β''. Therefore, from our experimental results and these literature data it seems sensible to attribute the weak peak F to β'' formation, which is the first step of the precipitation sequence. Moreover, the

intensity of this peak is very weak and well lower than that observed for the precipitation of β'' in a sample after solution and quenching (peak A, curve 1), therefore it can be inferred that the amount of β'' precipitated from the as-built sample is lower than that formed after solution and quenching. This is not surprising since, according to Fousova et al. [14] Mg_2Si phase is already present in the intercellular network observed in the as-built sample, and therefore only a fraction of the total magnesium is still available for β'' formation in the course of DSC run of the as-built material. In addition, according to Rao et. al [51] electron microscopy investigations, silicon precipitation is the main phenomenon occurring when a Al-Si-Mg alloy fabricated by LPBF is directly submitted to ageing, while formation of clusters containing Mg and Si and then the formation of β'' is strongly hindered.

The results reported by Fousovà et al. [14] and Rao et al. [51] are in agreement with our DSC traces showing a very weak thermal effect (4.6 J/g) for the F peak and a much important thermal effect (41.5 J/g) for peak G. As a consequence, peak G can be attributed to the more important effect of Si precipitation. In addition, the activation energy of peak G (211 kJ/mol) is much higher than those calculated in the present work (Table 3) or reported in the literature for the formation of β'', β' and β phases [6,12,20,52], but very close to the activation energy for the surface self-diffusion of silicon [47].

Also Fiocchi et al. [12], on the base on both position and activation energy, attributed the second exothermal peak in the DSC of as-built sample to the diffusion of silicon.

However, other interpretations can be found in the literature for the DSC trace of as-built AlSi10Mg processed by metal AM. According to Girelli et. al [20] the attribution of the second peak to a specific phenomenon cannot be done, as it could be assigned to β' formation on the base of its position, but shows an activation energy value more consistent with the silicon diffusion. Marola et al. [43] attributed the first peak placed at 250°C to silicon precipitation on the base of its enthalpy, and the second peak to β formation. Yang et al. [48] attributed the first peak (at 250°C) to the precipitation of small Si grains from Al lattice and the second one (at 310°C) to the precipitation of β' phase.

All these literature outcomes confirm the difficulty to give an interpretation of DSC curves obtained for AlSi10Mg as-built by LPBF process. However, experimental results (peak position, activation energy and enthalpy) suggest that peak F should be attributed to β'' formation and peak G to silicon precipitation.

3.4 Hardness

The hardness of AlSi0Mg samples in as-built conditions and after annealing thermal treatments carried out at different temperature and for different periods are compared in Figure 6; the measurements were performed on both the parallel and the perpendicular sample faces in order to investigate the possible material anisotropy.

Hardness values of 131.7 ± 3.1 HB and 115.4 ± 4.0 HB were measured on the sections of as-built cubic samples which are perpendicular and parallel to the building direction, respectively; the different hardness value shows that the not treated material has an anisotropic behaviour, with a higher hardness on the face which is perpendicular respect to the growth direction. This anisotropy can be explained considering that on the perpendicular face the hardness values are referred to the material layer which lastly solidified, while the hardness on the parallel face involves several layers subsequently consolidated and their interfaces. The microstructure on a parallel face or section is characterized by the presence of semi-circular melt pools partially overlapped and then tightly bonded together (Figure 1A). The scan tracks overlapping is due to a partial re-melting and solidification of the layer just below the last one consolidated by laser action. The material laying inside the last layer and placed on the side of each laser track is heated by the laser action, which could contribute to reduce the porosity by sintering. However, the porosity was a bit higher on the plane parallel to the growing direction, probably because of the presence of interfaces between the layers. The enhanced porosity can be responsible for slightly lower hardness values.

Anyway, LPBF parts show higher average hardness values if compared to material processed by conventional method. Maeshima et al. [53] reported that the hardness of AM AlSi10Mg is twice respect to that of material produced by gravity die casting. Moreover, Kempen et al. [26] investigated the hardness of AlSi10Mg alloy produced by high pressure die casting (HPDC), which is considered one of the casting process giving the best properties for this alloy, and found that the Vickers hardness of these specimens is

about 30 HV lower than that of samples processed by LPBF. According to Zou et al. [54], also wrought AlSi10Mg shows lower hardness compared to the same alloy processed by LPBF .

Revised Figure 6.

The stress relieving treatment performed at 300°C for 2 hours caused a reduction of hardness values down to 91.0 ± 0.9 HB and 85.6 ± 1.0 HB for the perpendicular and parallel planes respectively. On the base of the previously reported microstructure, the difference between these two values can be explained considering that the material, after stress reliving, still partially retains the microstructure and then the anisotropy characteristic of as-built specimens.

The exposition at the higher temperature of 500°C for 2 hours caused an additional remarkable decrement of the hardness, with values of 45.6 ± 0.9 HB and 44.3 ± 1.0 HB in the perpendicular and parallel sections respectively. There are no more significant differences between the two considered sections; this can be ascribed to the homogenization of the microstructure previously reported for annealed samples. Moreover, it can be noticed that the extension over 2 hours of the duration of the annealing treatment at 500°C has not a significant impact on the hardness. Also, a slight increase of the annealing temperature up to 550°C caused only a further small decrement of hardness values, irrespectively from the treatment time (2 and 6 hours). According to Aboulkhair et al. [11] the variation of hardness (and therefore of strength) as a function of the treatment temperature can be explained considering its effect on three main hardening mechanisms.

AlSi10Mg material in the as-built condition shows maximum hardness values mainly due to the very fine microstructure of the supersatured solid solution and the intercellular zones, where the final solidification of eutectic occurs; the grain-boundary strengthening effect (mechanism governed by Hall-Patch equation) plays in fact a fundamental role in maximise the properties of as-built parts [11,53]. Moreover, the as-built parts are in a metastable condition because of the fast cooling rates involved in the LPBF process, which reduce the amount of silicon rejected into the liquid phase during the solidification. This greatly extents the concentration of silicon in the aluminium matrix, which exceeds the solubility limit in the equilibrium state and enhances the strength through a solid solution mechanism. According to Maeshima et al. [53] the presence of Si atoms with lower atomic radius with respect to the Al ones (0.118 nm for Si and 0.143 nm for Al, respectively) causes a lattice deformation which favours the development of residual stresses and makes difficult the movements of dislocations. Aboulkhair et al. [11] also assumed that the interaction of dislocations in as-built AlSi10Mg material prevents their motion favouring a dislocation strengthening mechanism. However, this strengthening mechanism is partially inhibited in as-built samples due to the increased volume of grain boundaries.

An isothermal treatment at temperatures of 300°C, 500°C and 550°C, favours stress relaxation and can cause significant modification of the microstructure, which is no longer ultrafine after treatment at 500°C and 550°C, as discussed in previous sections. Moreover, the progressive disruption of Si particle network which surround the α-Al cells and the formation of polygonal Si precipitates with increased size at 500 and 550°C, inhibits both the Hall–Petch strengthening and the solid solution strengthening mechanisms previously reported.

One of the most studied heat treatments for AlSi10Mg alloy is T6 one, which exploits the precipitation-hardening mechanism. In order to evaluate the variation of hardness as function of T6 heat treatment conditions, the samples were solution treated at 520°C and 550°C, quenched and then subjected to artificial ageing at temperatures of 160° C and 180°C. Figure 7 shows the ageing curves of samples, based on the hardness measured on cross-sections perpendicular (Figure 7A) and parallel (Figure 7B) respect to the growing direction.

Revised Figure 7.

The trend of hardness change depends on the solution and ageing temperatures and on the ageing duration. The solution thermal treatment at 520°C causes the reduction of hardness from 131.7 ± 3.1 HB observed for as-built samples to about 74 HB. Afterwards, two different trends can be observed by ageing the samples at 160°C and 180°C respectively. The ageing at the lower temperature results in an increment of hardness values up to about 97,3 HB after 12 hours; this value remains almost constant during additional 3 hours of

ageing. The curves obtained by ageing the samples at 180°C show a maximum hardness value around 105 HB after 6-9 hours; over-ageing occurs for longer treatment time.

The solution treatment at the higher temperature of 550°C allows to obtain hardness values which are intermediate between those measured for samples as-built and aged at 520°C. Also in this case, different trends can be observed if the ageing is performed at 160°C or 180°C. In the first case the maximum hardness value (about 109 HB) is reached after 12 hours and remains constant for longer ageing time. A slightly higher hardness value can be reached by ageing at 180°C for 6 hours (113 HB).

It should be noted that the hardness of AlSi10Mg alloy is significantly different after annealing and SHT both performed at 550°C for 2 hours (hardness around 40 HB and 80HB, respectively). The higher hardness of solutioned samples with respect to annealed ones could be due to the presence of alloying elements in the aluminium lattice (retained during quenching from 550°C, where their solubility in aluminium is not negligible) that strengthen the material through solid solution mechanism and residual stresses developed during water quenching.

The curves obtained by using all the solution and ageing conditions discussed above are not significantly different when hardness measurements are performed on perpendicular or parallel sections; which is due to the fact that the T6 treatment involves the homogenization of the microstructure.

The results described above are in agreement with those reported by Aboulkhair et al. [11]. According to these authors, the hardness increases with the artificial ageing time due to the formations of Mg_2Si phase, even though the maximum hardness always remains lower respect to that of the as-built material. In addition, they observed that higher hardness values of as-quenched specimens can be obtained after by increasing the solution time thanks to the increase of Si particles size.

According to obtained experimental results, the highest hardness value is reached after a solution treatment of 550°C for 2 hours, quenching in water and artificial ageing at 180°C for 6 hours. However, the maximum hardness value resulting from this T6 treatment, for both the investigated cross sections, still remains about 13% lower than that measured for as-built sample.

3.5 Tensile strength

By means of tensile tests the behaviour of as-built material was compared to that of samples after stress relieving, annealing at 500°C for 2 hours or T6 treatment. The annealing conditions were selected considering that a further increment of annealing duration and temperature does not result in any significant changes in the microstructure, but involves a decrement of hardness because of grain growth. Moreover, on the base of the relationship between the microstructure and the hardness variation showed by microscopy investigations and ageing curves, the T6 thermal treatment consisting of a solution at 550°C for 2 hours, quenching in water and artificial ageing at 180°C for 6 hours, was selected.

Table 4 reports the tensile properties of as-built and thermal treated specimens in term of tensile strength, yield strength, elongation and elastic modulus. Stress-strain curves after different thermal treatments applied to samples with the axis parallel to the Z growing direction or placed on XY plane are also compared in Figure 8, with the aim of investigating the effect of AlSi10Mg sample orientation (with respect to the building platform) on the mechanical features. Tensile strength, modulus of elasticity and elongation at break are similar to those reported in the datasheet of the producer of the powders used for manufacturing the specimens.

The tensile properties resulting from different treatments are in good agreement with the previously observed hardness trend: in fact the maximum hardness value corresponds to the maximum tensile strength and it is observed in the as-built condition; the decrement of hardness with respect to the as-built condition after T6, stress relieving and annealing heat treatments corresponds to a progressive decrement of strength.

Table 4. Tensile properties of AlSi10Mg samples in as-built condition and after different kinds of heat treatments

Heat treatment	Tensile strength [MPa]		Yield strength [MPa]		Elongation at failure [%]		Elastic modulus [GPa]	
	Z	XY	Z	XY	Z	XY	Z	XY
As-built	429 ± 8	418 ± 7	226 ± 7	269 ± 6	4.0 ± 0.3	7.8 ± 0.4	75.5 ± 2	76 ± 2
Stress relieving 300°C/2h	257 ± 1	261 ± 3	159.5 ± 0.6	170 ± 2	18.1 ± 0.5	19.9 ± 1	78 ± 3	73 ± 2
Annealing 500°C/2h	133.4 ± 0.2	138.4 ± 0.2	77.9 ± 0.5	83.5 ± 0.4	29 ± 1	29 ± 2	69± 2	77.1 ± 0.2
SHT 550°C/2h + AA 180°C/2h	321 ± 3	332.9 ± 0.9	270 ± 4	275 ± 3	9 ± 1	12.0 ± 0.6	75 ± 2	74 ± 3

As expected, ductility inversely varies with the strength: the previously observed decreasing of material strength as function of different thermal treatments correspond to a more or less marked increase of elongation at failure with respect to as-built samples.

The tensile and yield strengths of as-built AlSi10Mg are 429.2 ± 8.0 MPa and 226.4 ± 6.9 MPa for samples grown along Z direction; while values of 418.0 ± 7.4 MPa and 268.6 ± 5.5 MPa are observed for specimens placed on the XY plane. These noticeable values of strength can be attributed to the very fine microstructure of the material. The investigated thermal treatments do not show any clear effects on elastic modulus, which in most cases does not seem to depend on the building orientation as well.

The results summarized in Table 4 show that ductility and yield strength of the material in as-built condition are influenced by the building orientation. This trend is in agreement to the results reported by Kempen et al. [26] who also investigated the mechanical behaviour of AlSi10Mg samples built according to different orientations (Z and XY); however, both the tensile strength and elongation values they obtained are lower respect to the experimental results here reported.

According to Kempen et al. [26] LPBF samples show a degree of anisotropy in elongation at break which is caused by the presence of more borderline porosity in Z-oriented samples. The presence of these pores, which formed at the beginning/end of a laser scanning, and the orientation of the interfaces perpendicular to the load direction could negatively affect the ductility of Z samples with respect to XY ones. On the contrary, the influence of orientation was negligible when ultimate tensile strength was considered. The effect of building orientation can be observed mainly in as-built samples, while it is no longer evident after further thermal treatments (stress-relieving, annealing, T6) since they modify the peculiar microstructure arising from the LPBF process (Figures 2 and 3).

Figure 8.

The rather poor elongation of LPBF components can be overcome by performing a stress relief treatment, which, unfortunately, also results in a significant decrease of tensile strength, as previously reported by many authors [14,16,17]. In fact, after stress relieving, the elongation at failure increases up to 18.1 ± 0.5 % and 19.9 ± 1.4 % for samples built along vertical (Z) and horizontal (XY) orientations respectively. On the other hand, stress relieving, involving the exposition of the material to a temperature of 300°C for 2 hours, causes the strong decrease of both tensile strength (decrease of 40% and 37.5% for Z and XY samples) and yield strengths (decrease of about 30% and 37.5% for Z and XY samples). Stress relieving is expected to recover most part of residual stresses and decrease the dislocation density; in addition, this treatment promotes the precipitation of fine silicon particles and lowers the silicon concentration in the aluminium supersaturated solid solution. All these microstructure modifications account for the strength decrease and the improvement of ductility.

The decrease of strength with respect to the as-built condition becomes even more marked after treatments involving the exposure at higher temperatures. In fact, after annealing at 500°C for 2 hours the tensile strength falls down of about 68% for both Z and XY samples. This is related to the coarsening of the microstructure and recrystallization, which entails the reduction of dislocations density. Moreover, at 500°C, the alloy microstructure evolves toward conditions which are closer to equilibrium ones. Silicon particles

segregates from solid solution, thus limiting the solid solution strengthening effect. In addition, silicon is a brittle phase that is expected to reduce the material plasticity [14] and affect the impact behaviour (as discussed in the next section). On the other hand, at the same time, the recrystallization phenomenon occurring during annealing can overbalance the reduction of plasticity caused by Si grains precipitation. In fact, after annealing at 500°C the strain at failure reaches the maximum value of about 28.5%.

The T6 heat treatment allows to obtain mechanical properties which are intermediate between those observed for as-built and stress relieved samples. After this treatment, the tensile strength for the two building orientations (Z and XY ones) was 321 MPa and 333 MPa and the elongation at failure was about 9% and 12%. Iturrioz et al. [16] performed T6 treatment in similar conditions (solution at 550°C and ageing at 180°C for 12 hours) obtaining slightly lower values of tensile strength (307 ± 8 MPa) and similar value of elongation (9 ± 3%). The mechanical properties after T6 treatment can be justified on the base of the microstructural modifications previously discussed. In fact, the T6 heat treatment results in a coarsening of the microstructure, a significant decrease of Si concentration in the aluminium-based solid solution and the precipitation of strengthening phases during ageing. The first two transformations should cause the decrease of strength and the increase of deformation, while precipitation should strengthen the materials because of the pinning of dislocation by precipitates.

Conclusively, the material properties can be tuned by using proper thermal treatments. The properties of the material in the as-built condition should be improved for practical applications because of the high residual stresses and very low ductility. Annealing at 500°C allows for achieving the best ductility, while stress-relieving results in higher strength coupled with rather good ductility. Further improvement of strength with respect to stress-relieving can be observed after T6 treatment, but deformation at failure is around 9% only. Both stress-relieving and T6 treatments, therefore, allow to obtain a compromise between strength and ductility; T6 treatment gives the better strength, but lower elongation at break with respect to stress relieving.

3.6 Impact properties

In spite of several investigations present in the literature about both the mechanical properties of AlSi10Mg parts processed by LPBF , and the influence of thermal treatments conditions on them, impact properties were only rarely studied. The response of specimens in terms of fracture energy caused by rapid load application has been summarized in Table 5.

Table 5. Results of Charpy test for AlSi10Mg parts.

Thermal treatment	Impact energy [J]
As-built	3.4 ± 0.9
Stress relieving 300°C/2h	13.0 ± 0.9
Annealing 500°C/2h	10.6 ± 1.4
SHT 550°C + AA 180°C	5.4 ± 0.7

The impact toughness was investigated only for V-notched specimens showing their axis perpendicular to the Z building direction. According to some literature [26] the sample orientation with respect to the building direction does not significantly affect the impact behaviour, even though in the case of specimens with the axis perpendicular to Z direction the interfaces between the stacked layers are perpendicular with respect to the applied load and then, in principle, they could deviate the crack path. Very likely this possible effect is not important when notched samples are tested, and therefore there is no reason of investigating samples with different building orientation. The as-built material showed the lowest impact energy of 3.4 ± 0.9 J, which is comparable to that obtained by Kempen et al. [26] for similar AlSi10Mg samples.

Both stress relieving performed at 300°C for 2 hours and annealing at 500°C for 2 hours involved a remarkable improvement of impact behaviour respect to the material in the as-built condition. Differently, the T6 treatment only led to a slight increase of impact Charpy energy for V-notched specimens. These outcomes only partially agree with the results reported by Girelli et al. [20], who observed for T6 treated

762 samples peak force and impact energies comparable or lower to that of samples in as-built condition, while
763 annealing resulted in significant increase of toughness, independently from the annealing temperature.
764 The morphologies of the fracture surfaces are compared in Figure 9.
765 The stress relieving treatment proved to exert a great beneficial effect on toughness because of the
766 reduction of residual stresses, even though this treatment had only little effect on the microstructure and the
767 fracture surface morphology, since the microstructure remained fine and the silicon particles did not grow
768 appreciably (Figure 9 E, F, I, L). Also annealing resulted in a significant toughness improvement, as it
769 caused the residual stress relieving and contemporaneously it greatly reduced the amount of silicon inside the
770 aluminium matrix, and then the distortion of the aluminium lattice. These toughening effects seem only
771 partially counterbalanced by the growth of both Al-Si-Fe and silicon precipitates.
772 The superior toughness arising from annealing treatment, over that of the as-built samples, is also evident
773 when the fracture surfaces are examined. In fact, larger dimples, typical of a ductile behaviour, can be seen
774 on the fracture surfaces of the annealed samples (Figure 9 G and M).
775 The material did not show very good resilience after the T6 treatment. Lower toughness with respect to the
776 stress relieved samples can be attributed to the presence of rather large silicon polygonal plates and the
777 precipitation of hardening phases. The precipitation of strengthening intermetallics greatly reduced the
778 ductility with respect to samples in the annealed conditions too. The fracture surface of T6 treated samples
779 show rather large dimples, but rather large particles (silicon platelets) can be observed on it (put in evidence
780 by red arrows in Figure 9 N). Very likely the interfaces between aluminium grains and large silicon platelets
781 can act as crack initiation sites.
782 Revised Figure 9.
783
784 **4. Conclusions**

785 Several combinations of mechanical features can be obtained by performing different thermal treatments on
786 AlSi10Mg components produced by selective laser melting. On the other hand, generally speaking, it is not
787 possible to maximize contemporaneously strength and ductility, and thermal treatments should be tailored
788 for achieving the best combination of properties for each practical application.
789 As-built material shows very high hardness and tensile strength, well over those observed for the same
790 material processed by conventional casting. Nevertheless, residual stresses which come from the production
791 process and are responsible for the material poor ductility and toughness have to be released. In addition, the
792 LPBF fabricated samples show a certain degree of anisotropy, as put in evidence by different hardness and
793 strength values measured on sections both parallel and perpendicular to the sample growth direction. Some
794 other properties, like yield strength and strain at fracture, depends on the orientation of specimens with
795 respect to the building direction; also these properties are expected to vary in the different parts of rather
796 large components with complex shape. This peculiar combination of microstructure features depends on the
797 very fast cooling from the solidification temperature involved in LPBF process, and it is also affected by the
798 presence of more or less weak interfaces between the layers that are subsequently consolidated by the laser
799 action. The fast cooling results in a very fine microstructure, consisting of Al-rich cells surrounded by
800 eutectic Al-Si regions forming a continuous network, and it promotes the formation of a supersaturated
801 aluminium-based solid solution containing large amounts of silicon. The as-built alloy shows a metastable
802 microstructure, that can be easily modified during subsequent heat treatments. Heating mainly results in the
803 diffusion of silicon and the formation of new silicon grains, as demonstrated by DSC investigations. Also
804 mechanical features change as a result of the microstructure modifications.
805 An isothermal treatment at 300°C mainly causes stress relieving and a limited reduction of concentration of
806 silicon in the supersaturated solid solution, while the microstructure still remains very fine. However, the
807 continuity of the network of eutectic structure around the aluminium cells cannot be seen any longer because
808 of the formation of small, but well distinguishable, silicon crystals. Nonetheless, the stress relieving
809 treatment greatly decreases strength and hardness and greatly enhances ductility and resilience. This kind of
810 treatment seems the best choice when, for a specific application, toughness should be maximized.
811 Isothermal treatments at higher temperature, such as annealing at 500-550°C for few hours, entail more
812 important microstructure evolution, which does not remain ultrafine any longer. In fact, annealing causes
813 more significant segregation of Si from the supersatured aluminium matrix and the formation of silicon

grains with larger size, as confirmed X-ray diffraction analyses, showing that the intensity of silicon main peak increases and its width decreases during this treatment. Moreover, Al-Si-Fe acicular precipitates appears in the microstructure of annealed samples. The relaxation of local stress, the reduction of silicon amount inside the aluminium matrix and the coarsening of the microstructure lead to the best improvement of the elongation at failure, while the resilience is just a bit lower than the maximum one observed after stress relieving. These toughening effects are associate with a very strong decrease of strength and hardness, in spite of the formation of intermetallic Al-Si-Fe precipitates. Finally, mechanical features are no more influenced by direction or building orientation as a result of the microstructure homogenization.

When annealing at 520-550°C is followed by water quenching and artificial ageing (that is when a T6 treatment is carried out) a compromise between ductility and strength can be achieved. However, this result can be obtained only if a sufficiently high ageing temperature is adopted. Ageing at 180°C for 6 hours allows to obtain hardness and tensile strength not too far from those characteristics of as-built conditions, while a lower ageing temperature results in lower hardening peak, which is observed only after a rather long treatment time. As a counterpart, after artificial ageing, ductility and toughness are not so good as after stress relieving or annealing, but better than those of the as-built material. During the solution step the microstructure changes in the same manner than during annealing. The subsequent water quenching could cause some residual stresses, that very likely are relieved during ageing. According to the results of microstructure and DSC investigations, artificial ageing is associated with the precipitation of β" phase, which is mainly responsible for the material strengthening, and it also causes the concentration increase of Al-Si-Fe acicular precipitates.

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Figure captions

Figure 1. Microstructure (A, B OM; C, D SEM images) of as-built samples: section parallel to the growing direction (A, C) and perpendicular to the building direction (B, D).

Figure 2. Microstructure of AlSi10Mg samples (perpendicular cross-sections) after stress relieving at 300°C for 2h (A- OM image, B- SEM) and after annealing at 500°C for 2h (C- OM image, D-SEM). EDS analyses performed on polygonal (E) and acicular (F) precipitates observed after annealing at 500°C for 2h.

Figure 3. Microstructure of AlSi10Mg samples after heat treatments. A and B: solution at 550°C for 2h and water quenching (OM and SEM images); C and D: solution at 550°C for 2h, water quenching and ageing at 180°C for 6h (OM and SEM images).

Figure 4. A) XRD spectra (normalized with respect to the most intense reflection of Al peak) of AlSi10Mg samples after thermal treatments: as-built condition (a-parallel cross-section and b-perpendicular cross-section); c: stress relieving at 300°C for 2h; d: annealing at 500°C for 2h; e: annealing at 550°C for 2h; f: solution at 550°C for 2h and quenching; g: solution at 550°C for 2h, quenching and ageing at 180°C for 6h. B) comparison of intensity for (111)Si peak after different thermal treatments.

Figure 5. DSC curves of AlSi10Mg samples (heating rate of 10°C/min; curves normalized by the mass of the samples): curve 1- after SHT at 550°C and quenching, curve 2 - after solution at 550°C followed by quenching and ageing at 180°C for 6 hours, curve 3 – as-built condition.

Figure 6. Hardness of AlSi10Mg samples in as-built conditions or after annealing at different temperatures and for different times.

Figure 7. Hardness of AlSi10Mg samples after solution at 520°C and 550°C, quenching in water and artificial ageing at 160°C and 180°C. A= perpendicular section; B=parallel section.

Figure 8. Tensile stress/strain curves of AlSi10Mg in as-built condition (A) and after different thermal treatments: T6 (B), stress relieving (C) and annealing (D). Effects of building orientation: specimens built parallel to the growing direction (Z samples), specimens placed on a plane perpendicular to the growing direction (XY samples).

Figure 9. Fracture surface of AlSi10Mg samples observed after Charpy impact test. SEM images obtained at different magnification: A, B, C and D at 8x, E, F, G and H at 750x; I, L at 15000x and M and N at 25000x.