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# 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

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

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

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

71 Many authors focus their attention on low temperature annealing [12–14], stress relieving [15–17] or T6  
72 treatments, but the proposed conditions for these thermal cycles are quite different in term of both maximum  
73 temperature reached and duration [16,18–23]. In addition, different mechanical features (mainly hardness  
74 and tensile strength) were considered by different authors to evaluate the treatment effectiveness. However,  
75 **other** mechanical properties (**such as** toughness, fatigue and creep) have been less frequently investigated  
76 [24,25]. It is moreover important to take into account that the mechanical properties of LPBF pristine parts  
77 depend on the adopted processing parameters; the thermal treatments investigated in the literature were  
78 generally carried out on materials that showed different characteristics in the as-built condition. For all these  
79 reasons, a comparison of data reported in the literature could be confusing.

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

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

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

100 performed treatments were correlated to mechanical properties in term of hardness, tensile and impact  
101 properties. X-ray diffraction analyses and calorimetric studies were also carried out in order to deeply  
102 investigate the evolution of the system after different applied thermal conditions.  
103

## 104 2. Materials and Methods

105 The samples under investigation were manufactured by the LPBF technology starting from gas atomized  
106 AlSi10Mg powder provided by EOS (actual composition (wt.%): 89.25 Al, 9.70 Si, 0.44 Mg, 0.38 Mn, 0.20  
107 Fe, 0.01 Ti, <0.01 Cu, <0.01 Zn). The LPBF process was carried out using an EOS M290 system equipped  
108 with a 400W Yb-fibre laser. All the specimens under investigation were produced by using the same  
109 processing parameters: a laser beam with a power of 370W was scanned across the powder bed, kept at  
110 165°C, with a speed of 1300 mm/s. The hatching distance between adjacent scan tracks was set to 0.19 mm,  
111 whereas the layer thickness was fixed at 30 µm.

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

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

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

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

138 Differential scanning calorimetry (Pyris 1 DSC, Perkin Helmer Italia Spa, Milano, Italy) was used for  
139 better understanding phase transformations and precipitation phenomena occurring when material is heated  
140 at high temperature. Samples of about 30 mg were placed in an aluminium crucible and heated to a  
141 temperature range from 100°C to 450°C in argon atmosphere. Calibration was done by using pure In and Zn  
142 standards and verifying that their melting point experimentally measured in DSC was 156.6 °C  $\pm$  0.3 °C and  
143 419.5°C  $\pm$  0.3°C respectively.

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

$$150 \log v = -0,4567 \cdot \frac{E}{R \cdot T} + \text{costant} \quad (1)$$

151 Where  $v$  is the heating rate,  $E$  is the activation energy of the phase transformation investigated,  $R$  is the  
152 universal gas constant, and  $T$  is the temperature which corresponds to the pre-fixed fraction of transformed  
153 material  $\alpha$ . For instance, the temperature of the maximum of symmetrical DSC peaks corresponds to  $\alpha=0.5$ .  
154 According to Ozawa [33], the constant is equal to  $-2,315 + \log \frac{Z \cdot E}{R} - \log g(\alpha)$ , where the integral function  
155  $g(\alpha)$  assumes a constant value for the considered  $\alpha$  value. When the heating rate in logarithmic form is  
156 plotted as a function on  $1/T$  (T corresponding to a fixed fraction of material conversion; in this study, a  
157 temperature corresponding to a conversion fraction of 0.5 was used), a straight line is obtained; from its  
158 slope the activation energy is determined. The activation energy for transformations observed in DSC traces  
159 was calculated by submitting samples to DSC runs with heating rates of 5, 10, 20 and 30 °C/min. To this  
160 purpose, the DSC analysis was performed on specimens in different conditions: as-built, solution treated at  
161 550°C for 2 hours and water quenched, solution treated/quenched and aged at 180°C for 6 hours.

162 Cubic samples with size 20mm x 20mm x 20mm were prepared in order to investigate the microstructure  
163 and evaluate their hardness. After performing different kinds of thermal treatments (stress relieving,  
164 annealing, precipitation hardening), the samples were polished by using SiC abrasive papers up to 1200 grit  
165 and their hardness was measured on the cross sections both parallel and perpendicular with respect to the  
166 building direction Z. In order to investigate the microstructure of AlSi10Mg alloy, a further polishing of  
167 samples surfaces with SiC papers up to 4000 grit and diamond pastes, with size of 3  $\mu\text{m}$  and 1  $\mu\text{m}$   
168 respectively, was performed. The effect on AlSi10Mg alloy of thermal treatments and ageing duration was  
169 investigated through Brinell hardness measurements, which were performed on polished cross sections using  
170 an EMCO TEST M5U-030 durometer (Prüfmaschinen GmbH, tungsten carbide indentator with a diameter of  
171 2.5mm). The tests were carried out applying a load of 62.5 kg maintained 10 s. Five measurements were  
172 done on each cross section, and hardness values were then averaged. Hardness results were also used as  
173 indicator of the strength of the material.

174 Cylindrical samples were built and then machined in order to obtain standard specimens for tensile tests  
175 with a total length of 48 mm, gauge length of 40 mm and a diameter of 8 mm (BS EN ISO 6506-1:2014).  
176 These samples were built both vertically and horizontally within the work chamber (samples respectively  
177 labelled as Z and XY in the text). The two kinds of samples mainly differed because the sequence of the  
178 stacked layers and the layer interfaces were perpendicular to the sample length (that is to the growing  
179 direction) in “Z” samples, while both layer sequence and interfaces were parallel to the sample length in  
180 “XY” samples. Tensile tests at room temperature were performed by using a Zwick 1474 dynamometer  
181 (Ulm, Germany), with a strain rate of 0.00025  $\text{s}^{-1}$ . Sets of 5 samples were tested for each treatment condition:  
182 as-built, after stress relieving, after annealing at 500°C, and after a selected T6 thermal treatment (solution at  
183 550°C for 2 hours, quenching in water and artificial ageing at 180°C for 6 hours).

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

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

### 190 191 3. Results and discussion

#### 192 3.1 Microstructure

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

#### 196 197 Revised Figure 1

198 199 The microstructure of the parallel cross section (Figure 1A) shows typical melt pools with a semi-circular  
200 profile due to the gaussian distribution of laser beam energy involved during the production process. It is  
201 possible to observe an overlapping between the pools in each single layer scanned by the laser and between



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

205 Considering the perpendicular plane (Figure 1B), the microstructure looks different, showing overlapped  
206 scan tracks with elongated shape and with different orientations; this is due to the adopted laser scan  
207 strategy, which involves the rotation of  $67^\circ$  between two adjacent layers subsequently stacked. The width of  
208 the scan tracks is in the range between 100 and 200  $\mu\text{m}$ , which corresponds to the extent of melt pools.  
209 Figures 1C and 1D show, for the two cross sections, a single melt pool at higher magnification after etching  
210 with Keller's reagent. The microstructure in Figure 1C is the result of a directional solidification. During the  
211 LPBF process, in fact, the interaction between the laser beam and the powder causes the formation of a  
212 thermal gradient due to the lower temperature of the underlying powder. The melted metal cools down very  
213 fast ( $\sim 10^6$   $^\circ\text{C}/\text{s}$ ) leading to the formation of a very fine structure constituted by Al-rich cells surrounded by  
214 Si-rich eutectic structures in the intercellular regions (Figures 1C and 1D). The size of cellular  $\alpha$ -Al grains  
215 decreases moving from the edge to the inner part of the melt pool due to the different cooling rate. The so  
216 called "fine cellular zone" consists of a finer microstructure which is characterized by cells in the range from  
217 0.4  $\mu\text{m}$  to 0.8  $\mu\text{m}$ ; the "coarse cellular area" presents cells double in size respect to the previous ones (from 1  
218 to 2  $\mu\text{m}$ ). This dimensional variation is due to both the gaussian distribution of laser beam energy and the  
219 cooling rate, which is different moving away from the previously solidified layer. According to Fousovà et  
220 al. [14] magnesium was detected by EDS preferentially within the intercellular network, where it can react  
221 mainly with Si. In binary systems the solubility of silicon in solid aluminium decreases from 1.65 wt.% at  
222  $577^\circ\text{C}$  to about zero at room temperature, while that of Mg decreases from 15 at. % at  $450^\circ\text{C}$  to less than 1%  
223 at room temperature. However, when considering the Al-Mg-Si ternary system the solubility of Mg and Si in  
224 solid Al, which is just above 1 at. % at  $627^\circ\text{C}$ , becomes negligible at  $180^\circ\text{C}$  under equilibrium conditions  
225 (less than 0.05 at. %) [34]. Therefore, there are not thermodynamic reasons for the presence of both silicon  
226 and magnesium inside the cellular  $\alpha$ -Al grains after solidification. On the other hand, selective laser melting  
227 involves dramatically high cooling rate from the molten state, and then deviations from equilibrium  
228 conditions are expected; nonetheless, the eutectic liquid solidifies after the primary  $\alpha$ -aluminium cells, and  
229 for this reason the eutectic structure contains more alloying elements (magnesium and silicon).

230 In both microstructures little black spots can be observed, which reasonably correspond to small porosities,  
231 that are currently present in parts produced by LPBF. According to some authors [35–38], they can be caused  
232 by hydrogen absorption, melt splashing or Marangoni flow occurring during the building process. The  
233 porosity evaluated by image analysis on the section parallel to the building direction was slightly higher with  
234 respect to that observed for the perpendicular one, the two sections showing porosity values of  $0.204\% \pm$   
235  $0.091$  and  $0.135\% \pm 0.062$  respectively. Anyway, the porosity seemed rather homogeneously distributed and  
236 the porosity level rather low. Tradosky et al. [39] also measured (by using a similar images analyses method)  
237 a higher porosity degree in the section parallel to the build direction with respect to that observed within the  
238 perpendicular one (about 1.7% and around 1% respectively).

239 Tentatively, it can be inferred that pores form more easily at the interfaces between layers that subsequently  
240 are molten by the laser action and therefore, they are preferentially located at melt pool boundaries.

#### 241 Revised Figure 2.

242  
243  
244 After performing a stress-relieving heat treatment at  $300^\circ\text{C}$  for 2 hours, only slight microstructure  
245 variations can be appreciated with respect to the as-built sample. The micrograph at low magnification  
246 (Figure 2A) does not put in evidence any significant differences: evident traces of pre-existing laser scan  
247 tracks are still present. Moreover, at higher magnification (Figure 2B), the treated material still shows a  
248 cellular-like microstructure, but the continuity of the eutectic network, previously observed in as-built  
249 samples, is now interrupted. In fact, it is possible to observe the precipitation of silicon grains uniformly  
250 dispersed within the aluminium matrix, with size in the range from 100 to 300 nm. This microstructure  
251 change is very likely due to the growth of both aluminium and silicon crystals, which was promoted by the  
252 hold at  $300^\circ\text{C}$ . At this temperature the system evolves towards equilibrium conditions, which entails the  
253 decrease of the amount of silicon present inside the aluminium lattice and the precipitation of this element

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

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

268 A little increase of annealing temperature from 500°C to 550°C causes the further increase of the size of Si  
269 particles (average size from 2 µm to 5 µm) because coalescence and Ostwald ripening occur [12]. This  
270 growth of Si grain size is consistent with the evolution of the microstructure of as-built samples (made of  
271 supersaturated solid solution of silicon inside the aluminium lattice) toward a thermodynamic equilibrium  
272 condition. In fact, increasing the annealing temperature promotes a quicker and important silicon  
273 precipitation and the coalescence and growth of silicon grains inside the aluminium matrix [20]. The  
274 progressive precipitation of silicon particles also results in the decrease of their concentration, that can be  
275 clearly appreciated by comparing the microstructure observed after stress relieving at 300°C (Figure 2B)  
276 with the microstructures resulting from annealing treatments performed at 500°C (Figure 2D).

277 The above-mentioned transformations modify the very fine microstructure of as-built samples; after thermal  
278 treatments it in fact becomes more similar to that observed for the AlSi10Mg alloy processed by  
279 conventional casting methods.

280 Figure 3.

281  
282 Figure 3 compares microstructures observed after a solution treatment at temperature of 550°C for 2 hours,  
283 quench in water to room temperature without (Figure 3A and 3B) and with (Figure 3C and 3D) a following  
284 artificial ageing at 180°C for 6 hours (which allows to obtain the hardness peak, as reported in section 3.4).  
285 Microstructures observed after the same kind of treatments, but at lower solution temperature of 520°C are  
286 very similar. Figure 3 shows that after SHT or T6 treatments the microstructure is very similar, consisting of  
287 Si particles with polygonal shape which are homogeneously dispersed within the Al matrix. In addition, the  
288 size of silicon grains (around 2-5 µm after SHT) does not significantly change during the ageing step. In  
289 every case, the silicon precipitates dimension was comparable to those observed after annealing at 520°C,  
290 which means that two hours of hold at above 500°C allow to complete the silicon precipitation and to bring  
291 the material to equilibrium conditions.

292 In addition to silicon polygonal particles, also acicular particles were found after the solution treatment.  
293 After solution and quenching precipitates are few micrometers long and less than 500 nm wide; they retain  
294 these dimensions during the artificial ageing, which only causes the increase of their concentration. EDS  
295 analyses on these precipitates showed that they are aluminium based and rich in Fe and Si, while the Mg  
296 presence was not found. It can be inferred that these precipitates can strengthen the matrix. The formation of  
297 Mg-Si strengthening phases (like Mg<sub>2</sub>Si), expected after the solution, quenching and ageing of Al-Si-Mg  
298 alloys, was not appreciated by SEM-EDS probably because only Guinier-Preston zones or precipitates semi-  
299 coherent with aluminium form.

### 301 3.2 X-ray diffraction

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

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

323  
 324 **Revised Figure 4**

325  
 326 Table 1. Percentage ratio between the intensities of Si (111) and Al (111) peaks and FWHM (Full Width at Half  
 327 Maximum) after different thermal treatments.

328  
 329

330 Thermal treatment	331 $\frac{\text{Si (111)}}{\text{Al (111)}} [\%]$	332 FWHM [°] Si(111)	333 Si grain size distribution	334 Al lattice parameter (nm)
335 As-built	2.9	0.610	< 100 nm	0.40489
336 Stress relieving 300°C_2h	6.7	0.267	100-300 nm	0.40510
337 Annealing 500°C_2h	23.5	0.094	1-4 $\mu\text{m}$	0.40512
338 Annealing 550°C_2h	26.5	0.100	2-5 $\mu\text{m}$	0.40515
339 SHT 550°C_2h	21.4	0.100	2-5 $\mu\text{m}$	0.40513
340 SHT 550°C_2h + AA 180°C_6h	18.9	0.119	2-7 $\mu\text{m}$	0.40513

338 Table 1 shows that the ratio between the intensity of (111) peak of silicon and that of (111) peak of  
 339 aluminium is higher in samples that experienced higher temperatures during the thermal treatment; this is  
 340 also put in evidence from the variation of intensity of (111) Si peak in Figure 4B. This outcome is due to the  
 341 effect of high temperature treatments that promote the modification of the microstructure towards conditions  
 342 of thermodynamic stability; these are reached more quickly at high temperatures.

343 **Actually, the solubility of silicon in the aluminium lattice in ternary Al-Si-Mg alloys becomes negligible**  
 344 **when temperature decreases [34].** For this reason, almost all silicon contained in the **AlSi10Mg** alloy  
 345 (namely 9.70 % wt.) must segregate (at every temperature between the melting/solidification point and room  
 346 temperature) from the aluminium-based supersaturated solid solution, when suitable conditions for silicon  
 347 diffusion and nucleation of silicon crystal exist. **In as-built samples an important fraction of the total silicon**  
 348 **is contained within the aluminium crystal lattice** as the Si/Al peaks intensity ratio is about 2.9% only, but  
 349 this ratio increases up to about 6.7% after stress relieving at 300°C and up to 26.5% after two hours of  
 350 annealing at 550°C because of the progressive formation of silicon crystals. This precipitation occurs not  
 351 only during the isothermal annealing, but also during the slow cooling at the end of the treatment. On the  
 352 contrary, a higher cooling rate after annealing (quench) limits the silicon precipitation, as shown in Table 1  
 353 where a Si/Al peaks intensity ratio of about 21.4% after the quench is reported. The **Si/Al intensity ratio** peak  
 354 slightly decreases after ageing at 180°C for 6 hours, since during ageing silicon is engaged in the formation  
 355 of strengthening precipitates (namely Mg<sub>2</sub>Si and acicular Al-Si-Fe).



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

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

369

370

### 3.3 Differential Scanning Calorimetry

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

373  $\alpha$ -SSS  $\rightarrow$  GPZ  $\rightarrow$   $\beta''$  (GPZ II)  $\rightarrow$   $\beta'$   $\rightarrow$   $\beta$  (stable  $Mg_2Si$ )

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

378 The DSC technique proved to be a powerful tool for investigating the ageing sequence of Al-Mg-Si alloys  
379 with rather low silicon content processed by casting [6,46]. In fact, the whole precipitation sequence can be  
380 appreciated in the DSC traces obtained after solution treatment and quench in water. These DSC traces show  
381 very weak phenomena for primitive GP zone formation and reversion just over 100°C and 200°C  
382 respectively, a sharp exothermal peak just above 250°C due to  $\beta''$  precipitation, an exothermal peak at around  
383 300°C of  $\beta'$  formation (semi-coherent  $Mg_2Si$ ) and an exothermal effect at higher temperature related to the  
384 formation of stable  $Mg_2Si$ . The grown GPZ (called GPZ II or  $\beta''$ ) are considered the main hardening phase,  
385 that can be detected by TEM after artificial ageing at the hardness peak.

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

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

400

401

Revised Figure 5

402

403

404

405

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

Heating rate [°C/min]	SHT 550°C			SHT 550°C + AA 180°C		AS-BUILT	
	A	B	C	D	E	F	G
	[°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

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

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

430 *3.3.1. Comparison of DSC traces of AlSi10Mg samples after solution treatment / quenching (curve 1),*  
 431 *and after solution / quenching and artificial ageing (curve 2).*

432 The DSC curve of as quenched AlSi10Mg samples shows three peaks (curve 1 of Figure 5): the one with  
 433 highest intensity (peak A) has a maximum between 216.4°C and 259.3°C depending on the different heating  
 434 rates; the associated activation energy is 88 kJ/mol. According to literature data [6,46] it seems reasonable to  
 435 attribute this exothermal phenomenon to the formation of the strengthening  $\beta''$  phase containing Mg and Si.  
 436 In addition, this peak disappeared in the DSC trace recorded after solution, quenching and artificial ageing at  
 437 180°C for 6 hours which cause the precipitation of the strengthening  $\beta''$  phase (curve 2 in Figure 5).

438 The peak B (curve 1 of Figure 5) in the temperature range from 271.7°C to 314.2°C with an activation energy  
 439 of 96 kJ/mol can be associated to the precipitation of  $\beta'$  phase that does not form during artificial ageing in the  
 440 conditions here adopted. In fact, it does not disappear in the DSC pattern of the AlSi10Mg sample after ageing  
 441 at 180°C (see peak D in curve 2 of Figure 5). However, the position of peak D (temperature ranging between  
 442 264.5°C and 301.4°C) slightly changed with respect to that of peak B. Nonetheless, in our experience, the  
 443 temperature at which a precipitation phenomenon occurs can be also affected by the previous thermal history  
 444 of the sample, and therefore the precipitation of a specific phase can be affected by the sample state before the  
 445 DSC run. Very likely precipitation can occur more easily (at lower temperature) if the sample microstructure  
 446 previously advanced through the precipitation sequence. For this reason, the precipitation of  $\beta'$  could occur at  
 447 slightly different temperatures for a solution treated-quenched sample and for a sample aged to the hardness  
 448 peak. In the first case all the precipitation sequence has to occur during DSC run while in the second case the  
 449 precipitation of  $\beta''$  completely occurred before the DSC test and the  $\beta'$  formation entails a re-arrangement of  
 450 the  $\beta''$  previously formed. Finally, a very weak signal in the range between 330.2°C and 371.1°C was also  
 451 observed.

452 These results (curve 1 of Figure 5) are also in good agreement with the literature dealing with DSC traces  
 453 of solution and quenched AlSi10Mg alloy produced by additive manufacturing. Girelli et al. [20] recorded a  
 454 similar DSC trace for AlSi10Mg samples treated at 540°C for 1 hour and then water quenched. Also these  
 455 authors observed the presence of three exothermal peaks for so treated samples: the first one at 240-284°C  
 456 corresponds to peak A of  $\beta''$  formation in curve 1 of Figure 5 (in the range between 216.4°C and 259.3°C);  
 457 the second at 298-329°C is about in the same temperature range of peak B of  $\beta'$  formation (271-314°C); the  
 458 third peak was observed at 321°C – 368°C, that is in the same range of peak C (at 330-371°C), and was  
 459 attributed by Girelli et al. to the formation of  $\beta$ -Mg<sub>2</sub>Si phase.

460 The formation of the stable  $Mg_2Si$  phase should occur during the DSC run of samples after solution and  
461 quenching as well as during the DSC run performed after artificial ageing. In Figure 5, the peak C (observed  
462 in the quenched sample, curve 1) and peak E (present in the DCS trace of artificially aged specimens, curve  
463 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  
464 a similar activation energy of 127 kJ/mol and 120 kJ/mol respectively. Therefore, they could be attributed to  
465 the precipitation of  $\beta$  phase.

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

469 The possible presence in the DSC curves of both solution treated/quenched or artificially aged AlSi10Mg of  
470 a significant thermal effect related to silicon precipitation is doubtful; in fact, the activation energy values for  
471 Si precipitation which are reported in some literature are in the range from 124 kJ/mol to 165 kJ/mol  
472 [12,20,41] or even higher [47]. These activation energy values are higher than that experimentally observed  
473 for peaks C and E (that we attribute to  $\beta$  formation instead). In addition, X-ray diffraction and microstructure  
474 investigations discussed in the previous sections show that after solution treatment/quenching as well as after  
475 artificial ageing only little amount of silicon is still present in the solid solution, and therefore during the  
476 DSC scan of samples in these treatment conditions only little amount of silicon could segregate from the  
477 solid solution to form silicon grains. The relevant thermal effect very likely could be overlapped to the peak  
478 for  $\beta$  formation. On the contrary, a well different scenario occurs when as-built samples are submitted to  
479 DSC analysis, as discussed in the following.

480

481 *3.3.2. DSC traces of as-built AlSi10Mg samples (curve 3).*

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

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

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

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

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

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

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

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

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

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

539

540

### 3.4 Hardness

541 The hardness of AlSi10Mg samples in as-built conditions and after annealing thermal treatments carried out  
542 at different temperature and for different periods are compared in Figure 6; the measurements were  
543 performed on both the parallel and the perpendicular sample faces in order to investigate the possible  
544 material anisotropy.

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

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

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

#### 565 Revised Figure 6.

566  
567  
568 The stress relieving treatment performed at 300°C for 2 hours caused a reduction of hardness values down  
569 to  $91.0 \pm 0.9$  HB and  $85.6 \pm 1.0$  HB for the perpendicular and parallel planes respectively. On the base of the  
570 previously reported microstructure, the difference between these two values can be explained considering  
571 that the material, after stress relieving, still partially retains the microstructure and then the anisotropy  
572 characteristic of as-built specimens.

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

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

596 An isothermal treatment at temperatures of 300°C, 500°C and 550°C, favours stress relaxation and can  
597 cause significant modification of the microstructure, which is no longer ultrafine after treatment at 500°C  
598 and 550°C, as discussed in previous sections. Moreover, the progressive disruption of Si particle network  
599 which surround the  $\alpha$ -Al cells and the formation of polygonal Si precipitates with increased size at 500 and  
600 550°C, inhibits both the Hall-Petch strengthening and the solid solution strengthening mechanisms  
601 previously reported.

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

#### 608 Revised Figure 7.

609  
610 The trend of hardness change depends on the solution and ageing temperatures and on the ageing duration.  
611 The solution thermal treatment at 520°C causes the reduction of hardness from  $131.7 \pm 3.1$  HB observed for  
612 as-built samples to about 74 HB. Afterwards, two different trends can be observed by ageing the samples at  
613 160°C and 180°C respectively. The ageing at the lower temperature results in an increment of hardness  
614 values up to about 97,3 HB after 12 hours; this value remains almost constant during additional 3 hours of



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

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

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

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

631 The results described above are in agreement with those reported by Aboulkhair et al. [11]. According to  
632 these authors, the hardness increases with the artificial ageing time due to the formations of Mg<sub>2</sub>Si phase,  
633 even though the maximum hardness always remains lower respect to that of the as-built material. In  
634 addition, they observed that higher hardness values of as-quenched specimens can be obtained after by  
635 increasing the solution time thanks to the increase of Si particles size.

636 According to obtained experimental results, the highest hardness value is reached after a solution treatment  
637 of 550°C for 2 hours, quenching in water and artificial ageing at 180°C for 6 hours. However, the maximum  
638 hardness value resulting from this T6 treatment, for both the investigated cross sections, still remains about  
639 13% lower than that measured for as-built sample.

640

### 641 3.5 Tensile strength

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

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

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

660

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665

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

Heat treatment	Tensile strength		Yield strength		Elongation at failure		Elastic modulus	
	[MPa]		[MPa]		[%]		[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

677  
678 As expected, ductility inversely varies with the strength: the previously observed decreasing of material  
679 strength as function of different thermal treatments correspond to a more or less marked increase of  
680 elongation at failure with respect to as-built samples.

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

686 The results summarized in Table 4 show that ductility and yield strength of the material in as-built condition  
687 are influenced by the building orientation. This trend is in agreement to the results reported by Kempen et al.  
688 [26] who also investigated the mechanical behaviour of AlSi10Mg samples built according to different  
689 orientations (Z and XY); however, both the tensile strength and elongation values they obtained are lower  
690 respect to the experimental results here reported.

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

699  
700 Figure 8.

701  
702 The rather poor elongation of LPBF components can be overcome by performing a stress relief treatment,  
703 which, unfortunately, also results in a significant decrease of tensile strength, as previously reported by many  
704 authors [14,16,17]. In fact, after stress relieving, the elongation at failure increases up to  $18.1 \pm 0.5$  % and  
705  $19.9 \pm 1.4$  % for samples built along vertical (Z) and horizontal (XY) orientations respectively. On the other  
706 hand, stress relieving, involving the exposition of the material to a temperature of 300°C for 2 hours, causes  
707 the strong decrease of both tensile strength (decrease of 40% and 37.5% for Z and XY samples) and yield  
708 strengths (decrease of about 30% and 37.5% for Z and XY samples). Stress relieving is expected to recover  
709 most part of residual stresses and decrease the dislocation density; in addition, this treatment promotes the  
710 precipitation of fine silicon particles and lowers the silicon concentration in the aluminium supersaturated  
711 solid solution. All these microstructure modifications account for the strength decrease and the improvement  
712 of ductility.

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

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

723 The T6 heat treatment allows to obtain mechanical properties which are intermediate between those  
724 observed for as-built and stress relieved samples. After this treatment, the tensile strength for the two  
725 building orientations (Z and XY ones) was 321 MPa and 333 MPa and the elongation at failure was about  
726 9% and 12%. Iturrioz et al. [16] performed T6 treatment in similar conditions (solution at 550°C and ageing  
727 at 180°C for 12 hours) obtaining slightly lower values of tensile strength ( $307 \pm 8$  MPa) and similar value of  
728 elongation ( $9 \pm 3\%$ ). The mechanical properties after T6 treatment can be justified on the base of the  
729 microstructural modifications previously discussed. In fact, the T6 heat treatment results in a coarsening of  
730 the microstructure, a significant decrease of Si concentration in the aluminium-based solid solution and the  
731 precipitation of strengthening phases during ageing. The first two transformations should cause the decrease  
732 of strength and the increase of deformation, while precipitation should strengthen the materials because of  
733 the pinning of dislocation by precipitates.

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

### 742 3.6 Impact properties

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

747  
748 Table 5. Results of Charpy test for AlSi10Mg parts.

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

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

757 Both stress relieving performed at 300°C for 2 hours and annealing at 500°C for 2 hours involved a  
758 remarkable improvement of impact behaviour respect to the material in the as-built condition. Differently,  
759 the T6 treatment only led to a slight increase of impact Charpy energy for V-notched specimens. These  
760 outcomes only partially agree with the results reported by Girelli et al. [20], who observed for T6 treated  
761

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 supersaturated aluminium matrix and the formation of silicon

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

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

834

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838

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### 980 Figure captions

981  
 982 Figure 1. Microstructure (A, B **OM**; C, D SEM images) of as-built samples: section parallel to the growing direction (A,  
 983 C) and perpendicular to the building direction (B, D).

984

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

988

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

992

993 Figure 4. A) XRD spectra (normalized with respect to the most intense reflection of Al peak) of AlSi10Mg samples  
 994 after thermal treatments: as-built condition (a-parallel cross-section and b-perpendicular cross-section); c: stress  
 995 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  
 996 quenching; g: solution at 550°C for 2h, quenching and ageing at 180°C for 6h. B) comparison of intensity for (111)Si  
 997 peak after different thermal treatments.

998

999 Figure 5. DSC curves of AlSi10Mg samples (**heating rate of 10°C/min; curves normalized by the mass of the samples**):  
 1000 curve 1- after SHT at 550°C and quenching, curve 2 - after solution at 550°C followed by quenching and ageing at  
 1001 180°C for 6 hours, curve 3 – as-built condition.

1002

1003 Figure 6. Hardness of AlSi10Mg samples in as-built conditions or after annealing at different temperatures and for  
 1004 different times.

1005

1006 Figure 7. Hardness of AlSi10Mg samples after solution at 520°C and 550°C, quenching in water and artificial ageing at  
 1007 160°C and 180°C. A= perpendicular section; B=parallel section.

1008

1009 Figure 8. Tensile stress/strain curves of AlSi10Mg in as-built condition (A) and after different thermal treatments: T6  
 1010 (B), stress relieving (C) and annealing (D). **Effects of building orientation: specimens built parallel to the growing  
 1011 direction (Z samples), specimens placed on a plane perpendicular to the growing direction (XY samples).**

1012

1013 Figure 9. Fracture surface of AlSi10Mg samples observed after Charpy impact test. SEM images obtained at different  
 1014 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.

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