

THERMAL TREATMENTS OF AlSi10Mg PROCESSED BY LASER BEAM MELTING

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Abstract

Recent studies have shown that AlSi10Mg processed by Laser Beam Melting (LBM) exhibits a much finer microstructure when compared to its cast counterpart as a consequence of the much faster cooling rates imposed in the LBM process. Such microstructural refinement causes a significant increase in strength and hardness, to such an extent that as-fabricated LBM AlSi10Mg was reported to present hardness value of 127 ± 3 Hv0.5, similar to the hardness of high pressure die cast AlSi10Mg in the aged condition (i.e. 130-133 Hv). Yet, little attention has been given so far to the influence of thermal treatments on the microstructure and mechanical behavior of LBM AlSi10Mg. The present work hence aims to investigate the effect of two different types of heat treatments – i.e. (i) stress relief and (ii) solutionizing and ageing – on the microstructure, hardness and tensile properties of LBM AlSi10Mg.

Introduction

The steadily increasing demand for Al parts with complex shapes, controlled porosity and/or lattice structures brings a strong motivation for the development of processing routes offering a high geometrical flexibility [1-3]. The additive manufacturing process known as Laser Beam Melting (LBM) or Selective Laser Melting (SLM) is among the most promising candidates in that respect, and so far, the hypo-eutectic Al alloy AlSi10Mg has been among the most used Al alloys for additive processing [2-4].

Additive Manufacturing (AM) techniques for metallic materials are characterized by extremely fast heating and cooling cycles, and as a consequence, metallic parts processed by AM are sensitive to the build-up of internal stresses. They also tend to exhibit much finer microstructures when compared to their counterparts processed by more traditional processing routes such as casting [5, 6]. This microstructural refinement may cause a significant increase in strength and hardness. As-fabricated LBM AlSi10Mg was even reported to present a hardness value of 127 ± 3 Hv0.5, similar to the hardness of High Pressure Die Cast (HPDC) AlSi10Mg in the aged condition (i.e. 130-133 Hv) [2].

Thanks to the addition of a small amount of Mg, AlSi10Mg is indeed prone to age hardening due to the precipitation of Mg_2Si [7-9]. However, little attention has been given so far to the influence of post-processing thermal treatments on the microstructure and mechanical behavior of LBM AlSi10Mg, possibly in view of the excellent mechanical behavior of as-built LBM AlSi10Mg [2]. Moreover, the few studies available on this subject may appear confusing. Investigations into the ultra-fast solidification of Al-Si alloys have shown that the solid solubility

of Si in Al could be drastically increased at very high cooling rates [10], similar to the cooling rates of 10^3 - 10^7 K/s that may be attained during Laser Melting [11]. Consequently, attempts were made to age harden as-built LBM AlSi10Mg at 170°C for 1 hour without carrying out a prior solutionizing step [12], but this treatment did not bring any significant change in hardness. Brandl et al. [3], on the other hand, reported a significant effect of a solutionizing (at 525°C for 6 hours) and ageing (at 165°C for 7 hours) treatment on the fatigue properties of LBM AlSi10Mg, but the effects of this thermal treatment on the hardness and tensile properties were not discussed.

The present paper hence aims to report on the early stages of an investigation into the effect of two different types of heat treatments – i.e. (i) stress relief and (ii) solutionizing and ageing – on the microstructure, hardness and tensile properties of LBM AlSi10Mg.

Experimental methods

Samples were produced from an AlSi10Mg gas atomized powder whose composition is given in table 1, using a MTT SLM 250 laser melting deposition manufacturing system with an argon purged production chamber. The processing parameters were kept constant for all samples i.e. a laser power of 175W, a travel speed of 195 mm/s, a hatch space of 0.19 mm, a focus offset of 1 mm and a powder layer thickness of 60 μ m. The substrate was preheated at 200°C. All tensile samples were built vertically i.e. with their tensile direction parallel to the building direction.

Table 1: Chemical composition of the AlSi10Mg powder (mass %)

Si	Fe	Cu	Mn	Mg	Zn	Ni	Ti	Al
9.6	0.16	0.003	<0.001	0.41	<0.002	<0.005	0.004	Bal.

Part of the samples were heat treated either (i) for stress relief at 250°C for 2 hours, or (ii) solutionized at 510°C for 6 hours and aged at 150°C, 170°C or 190°C for varying times. Preliminary trials using a solutionizing temperature of 530°C had led to a significant deterioration of the surface of the samples (Figure 1). This could be ascribed to the occurrence of a partial melting of the samples, possibly as a result of the addition of 0.41 wt% Mg that would depress the eutectic temperature [13]. The lower temperature of 510°C was thus selected for later tests in order to avoid this problem.

The overall quality and soundness of the parts was assessed by comparing their actual density with the theoretical density of alloy AlSi10Mg i.e. 2.68 g/cm³ [14]. The actual density of as-built specimens was determined by combining a measurement of the mass of the sample using a high precision weighing scale and a measurement of the volume of the same sample using an Accupyc 1340 Helium pycnometer from Micromeritics. The relative density was found satisfactory with an average value of 98%. Samples for metallographic examinations were embedded in resin and polished following standard practices. Detailed microstructural observations were carried out after etching with Flick's reagent i.e. 10 ml HF + 90 ml H₂O [15], using a SEM-FEG FEI X130 scanning electron microscope.



Figure 1: Deterioration of the samples' surface following solutionizing heat treatment at 530°C for 6 hours.

Hardness was measured by Rockwell B tests under a load of 1 kg. Uniaxial tensile tests were performed according to the ASTM E 111-04 standard on samples 3 mm thick, 7 mm wide and with an initial gauge length of 20 mm. An Instron 4500 tensile machine was used at a constant displacement rate of 2 mm/min. To ensure for sufficient statistics, ten samples have been tested for each condition. Average values of the yield stress, the ultimate tensile strength and of the maximum elongation have been obtained from the tensile stress-strain curves. The fractured surfaces have also been observed using SEM, in order to gain a deeper insight into the mechanisms leading to failure.

Results and discussion

As-built samples

Figure 2 shows the HRB hardness of as-built AlSi10Mg specimens, in comparison with hardness values available in literature [2] for the same alloy in the as-fabricated LBM condition (i.e. 127 ± 3 Hv0.5) and in the high pressure die cast and aged condition (i.e. 130-133 Hv). For the sake of comparison, Vickers hardness values were converted to approximate Rockwell B hardness according to Table 9 in [16]. As-built LBM samples of the present study exhibits a lower hardness when compared with data taken from [2]. The conversion of Vickers hardness values from [2] into Rockwell B values may of course introduce an error in the comparison. However, it is also likely that differences in LBM processing parameters may lead to differences in microstructures hence explaining the different hardness between the data of Thijs et al [2] and the properties recorded for the specimens of the present study. In particular, preheating the substrate up to 200°C may induce slightly lower cooling rates in the present case.

Figure 3 shows that the tensile properties of as-built LBM samples of the present study, on the other hand, do compare quite well with the tensile properties announced for AlSi10Mg in materials data sheets [17]. Indeed, the yield stress (figure 3(a)), the ultimate tensile strength (figure 3(b)) and the elongation at break (figure 3(c)) of the as-built LBM specimens are all slightly higher than the corresponding values for the reference material [17].

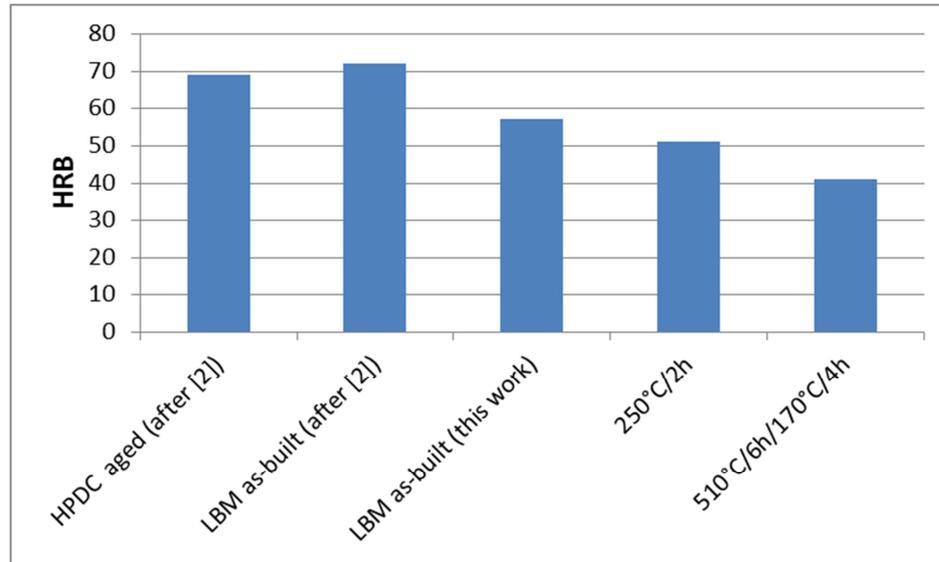


Figure 2: HRB Hardness (i) as-built, (ii) after stress relief at 250°C for 2 hours, (iii) solutionized at 510°C for 6 hours and aged at 170°C for 4 hours. Data for LBM as-built samples and HPDC and aged samples [2] are shown for comparison.

Figure 4 allows to compare the microstructure of as-built LBM AlSi10Mg with the typical microstructure of its cast counterpart. As can be seen in figure 4 and as was previously reported [2, 5], the extremely high cooling rates involved in LBM cause a strong microstructural refinement. Si precipitates, that can be up to 10-20 μm in size in cast AlSi10Mg, are replaced by an extremely fine lamellar eutectic structure in as-built LBM AlSi10Mg (figure 5) [5]. Microstructural refinement may hence be the cause of the improvement of the tensile properties – both in strength and ductility – of the as-built LBM AlSi10Mg specimens when compared to their cast reference counterparts. The SEM fractograph of figure 6 shows that the fracture mechanism of as-built LBM AlSi10Mg was mostly ductile. However, it is also worth noting that the fracture surface exhibits what Prashanth et al. [18] described as "*a step-like morphology*" while they were investigating the LBM of an Al-12Si alloy. They found that the average step-size was roughly similar to the size of the laser tracks, which also appears to be true in the present case. Prashanth et al. [18] further suggested that an excess of Si-rich precipitates had formed in the hatch overlap regions, and that these regions consequently acted as preferential sites for damage nucleation. However, textural effects may also exert a strong influence on the tensile properties of LBM AlSi10Mg parts [2]. Hence, at this stage, it is unclear whether the hypothesis of Prashanth et al. [18] might also apply to the as-built LBM AlSi10Mg samples of the present study.

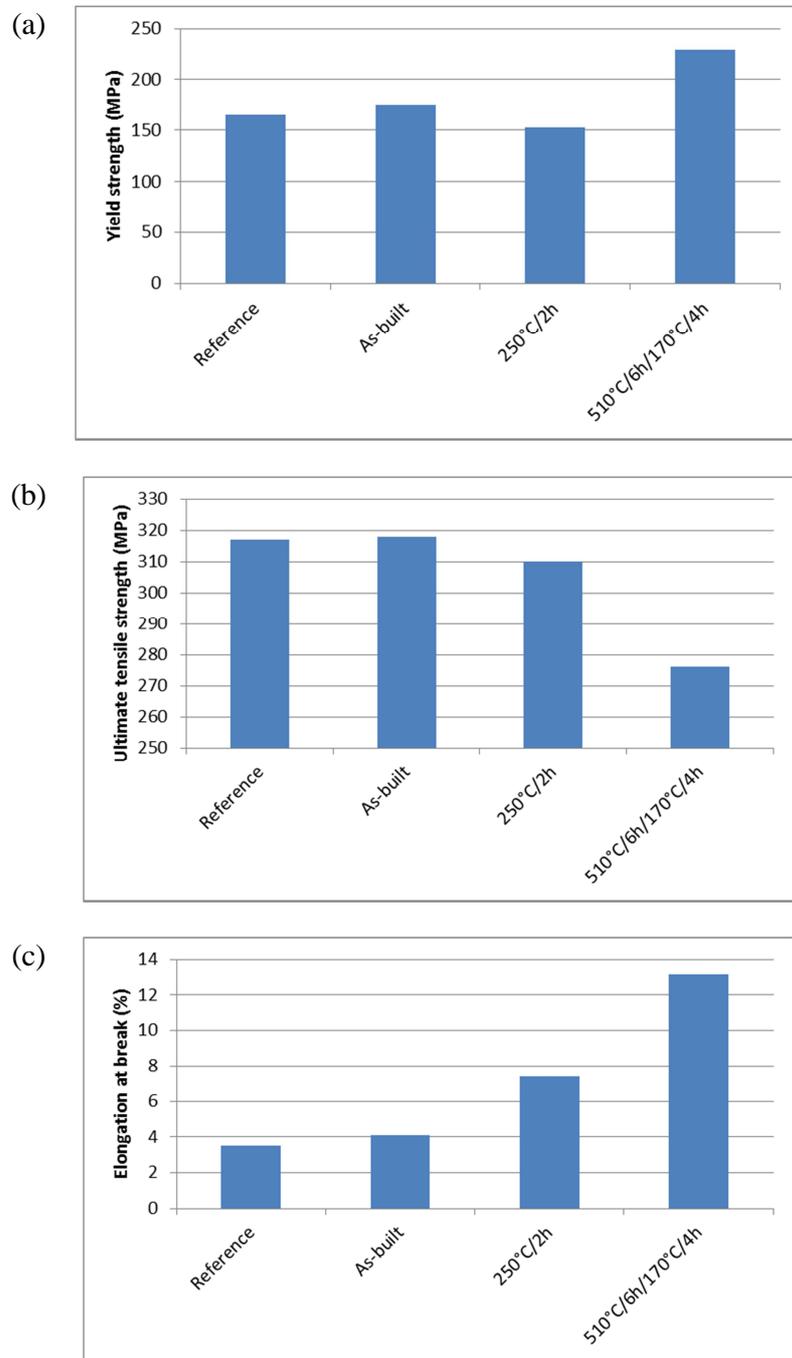


Figure 3: Tensile properties of (i) alloy AlSi10Mg Die Cast (Reference, after [17]), (ii) LBM as-built, (iii) LBM after stress relief at 250°C for 2 hours, and (iv) LBM solutionized at 510°C for 6 hours and aged at 170°C for 4 hours. (a) Yield stress (MPa), (b) Ultimate tensile strength (MPa), and (c) Elongation at break (%).

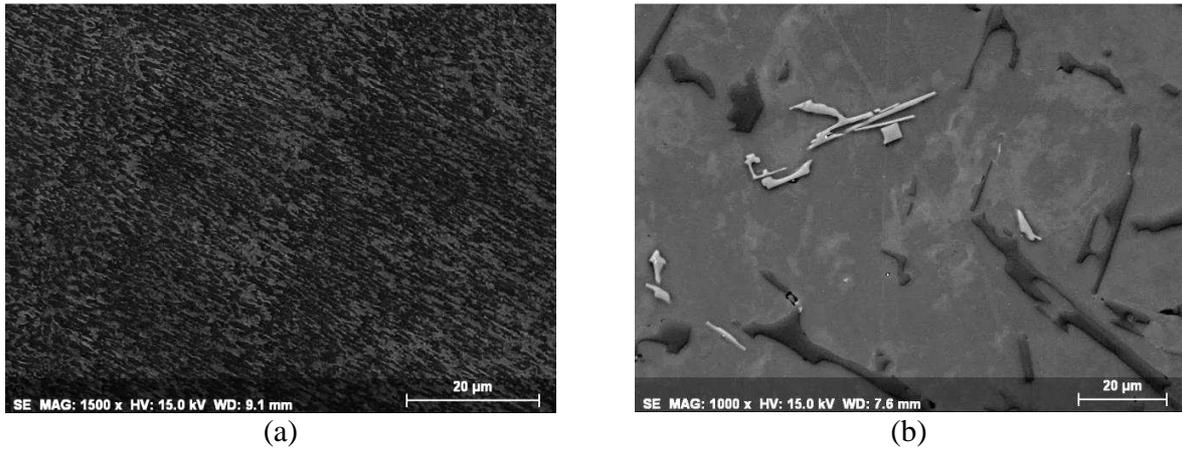


Figure 4: SEM micrographs of (a) LBM AlSi10Mg as-built, and (b) cast AlSi10Mg, after etching with Flick's reagent.

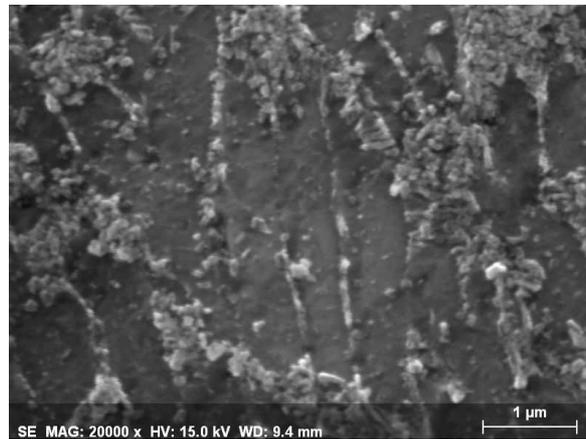


Figure 5: SEM micrograph of LBM AlSi10Mg as-built after etching with Flick's reagent (high magnification)

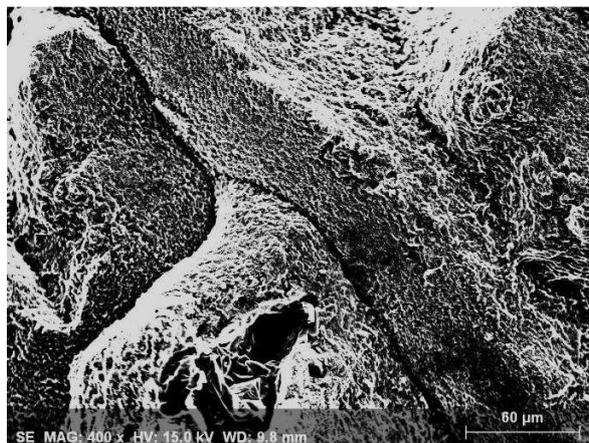


Figure 6: SEM fractograph of as-built LBM AlSi10Mg tensile specimens.

Effect of a thermal treatment at 250°C for 2 hours

Figures 2 and 3 show the effect of a thermal treatment at 250°C for 2 hours, respectively on the hardness and on the tensile properties of LBM AlSi10Mg. In comparison with the as-built samples, heat treating at 250°C for 2 hours results in a decrease of the hardness by 10%, of the yield stress by 12%, and in a slight decrease of the ultimate tensile strength by 2%. The elongation at break of heat treated specimens, on the other hand, exhibits an increase by 80% when compared to the as-built samples. So far, microstructural examinations on samples heat treated at 250°C for 2 hours did not reveal any significant microstructural changes in comparison with as-built samples. It thus appears that the improvement in ductility brought about by the heat treatment at 250°C for 2 hours might be essentially ascribed to the relief of internal stresses. It is also worth noting that the improvement in ductility observed here is not as spectacular as the improvement by 270% reported by some of the present authors for LBM Ti-6Al-4V [19]. This might be explained at least in part by the fairly high thermal conductivity of alloy AlSi10Mg making it somewhat less sensitive to the build-up of internal stresses [2, 19].

Effect of the solutionizing and ageing treatment

Figure 7 shows the effect of the ageing temperature and time (after solutionizing at 510°C for 6 hours) on the HRB hardness of LBM AlSi10Mg. When ageing at 150°C, the hardness increases slowly and steadily up to a value of 53 HRB after ageing for the longest investigated holding time of 10 hours. The hardness increases rapidly during the 4 first hours of ageing at 170°C. It then remains at a constant value of about 40 HRB for the following 6 hours at 170°C. When ageing at 190°C, the hardness goes through a maximum of 38 HRB after 1 hour, after which it decreases slowly and steadily (likely because of an excessive coarsening of the precipitates [20]). In conclusion, among the investigated condition, the highest hardness is obtained after ageing at 150°C for 10 hours. However, a good compromise between hardness improvement and holding time is reached for ageing at 170°C for 4 hours. It is these latter ageing conditions that will be investigated further in the present study as they provide a fairly large processing window. Indeed, further increasing the holding time up to 10 hours at 170°C does not cause any significant decrease in hardness.

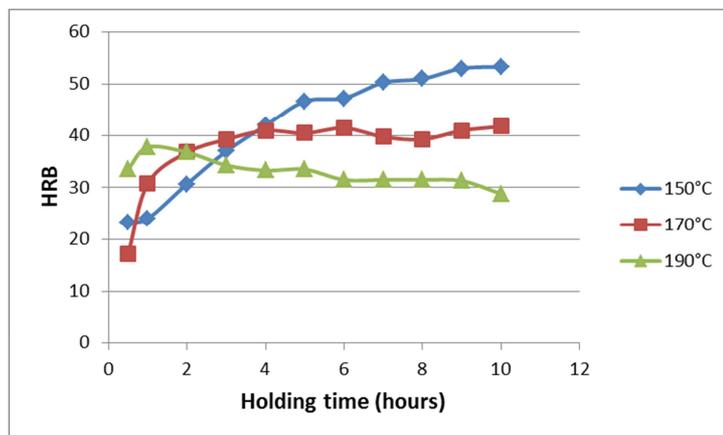


Figure 7: HRB hardness as a function of ageing temperature and time, after solutionizing at 510°C for 6 hours

Figures 2 and 3 also show the effect of the solutionizing and ageing treatment (510°C / 6 hours / 170°C / 4 hours) on the hardness and tensile properties. In comparison with the as-built samples, solutionizing and ageing results in a decrease of the hardness by 28%, and of the ultimate tensile strength by 13%. The yield stress, on the other hand, is increased by 30%, and the elongation at break of aged samples exhibits a spectacular increase by 220%. This latter effect can be better understood in view of the SEM micrographs of figure 8. Figure 8(b) shows that fracture was completely ductile. Moreover, the steps that characterized the fractured surface of the as-built samples (figure 6) have disappeared. Figure 8(a) shows that the microstructure was significantly altered as a result of the solutionizing and ageing treatment. Most importantly, the very fine lamellar eutectic structure of the as-built samples (see figure 4(a) and figure 5) is now completely globularized. A similar observation has been reported by Brandl et al. [3] after solutionizing LBM AlSi10Mg parts at 525°C for 6 hours and ageing at 165°C for 7 hours. The complete globularisation of the fine eutectic lamellar structure requires a long range diffusion of atoms. It is hence not likely to take place during the ageing step, but is rather expected to take place during the solutionizing step which thus appears to play a key role in determining the mechanical properties of solutionized and aged LBM AlSi10Mg. Although further work is still needed in order to reach a deeper understanding of the microstructural change occurring during the solutionizing step and to fully optimize the treatment conditions, a solutionizing and ageing heat treatment thus appears very promising in view of tailoring the tensile properties – particularly the yield stress and the ductility – of LBM AlSi10Mg parts.

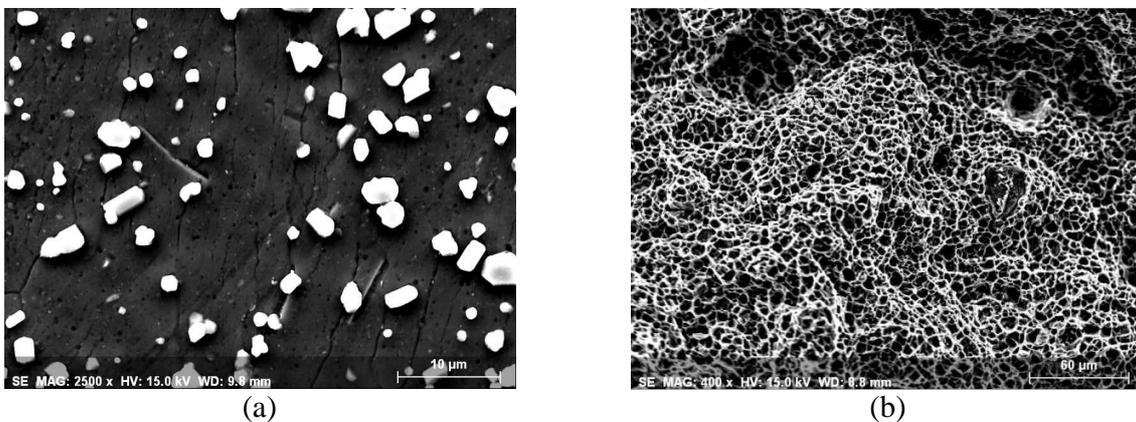


Figure 8: (a) SEM micrograph of LBM AlSi10Mg solutionized at 510°C for 6 hours and aged at 170°C for 4 hours (after etching with Flick's reagent), and (b) SEM fractograph of a solutionized and aged LBM AlSi10Mg tensile specimen

Conclusions

- The effects of two different heat treatments on the microstructure, hardness and tensile properties of LBM AlSi10Mg have been investigated.
- Heat treating at 250°C for 2 hours brought an increase of the elongation at break by 80%, at the expenses of a decrease of the hardness by 10%, of the yield stress by 12%, and of a slight decrease of the ultimate tensile strength by 2%. This effect might be ascribed mainly to the relief of internal stresses.
- In comparison with as-built samples, solutionizing and ageing results in a decrease of the hardness by 28%, and of the ultimate tensile strength by 13%. The yield stress, on the other hand, is increased by 30%, and the elongation at break of aged samples exhibits a spectacular increase by 220%.
- The improvement of the ductility could be linked to the fact that the very fine lamellar eutectic structure of the as-built samples was completely globularized after the solutionizing and ageing treatment. This microstructural modification may be expected to take place during the solutionizing step which hence appears to play a key role in determining the mechanical properties of solutionized and aged LBM AlSi10Mg.
- Although further work is still needed to fully optimize the treatment conditions, a solutionizing and ageing heat treatment thus appears very promising in view of tailoring the tensile properties – particularly the yield stress and the ductility – of LBM AlSi10Mg parts.

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