Investigating the effects of post-heat treatment on residual stress in AlSi12 parts processed with Selective Laser Melting

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Abstract. Residual stresses are the stresses that remain on a part after the original cause of any stress has been removed. In Selective Laser Melting (SLM), an additive manufacturing process, residual stress occurs in the created part due to rapid melting and rapid solidification; these stresses are responsible for distortion, which must therefore be minimised or eliminated. Residual stresses can be reduced in SLM built parts by the application of post heat treatment, and this process is investigated in this study. X-ray diffraction was employed for the experimental analysis of the residual stresses in AlSi12 parts processed by SLM that were then subjected to heat treatments at different temperatures, ranging from 200 °C to 500 °C, in order to investigate how residual stresses and microstructure changes vary with heat treatment temperatures.

Keywords: SLM, AlSi12, Residual stress, Heat treatment

1. Introduction

Selective laser melting (SLM) is now one of the leading additive manufacturing (AM) processes for fabricating metal parts utilising layer by layer deposition techniques. It is a type AM powder bed process in which a layer is built up by melting metal powder with a scanning laser beam [1, 2]. Residual stress formation occurs in SLM built parts, however, due to the rapid heating and cooling cycle caused by high laser power and other parameters such as scan speed, layer thickness, and scan strategy. The thermal gradient mechanism is the key influencing factor in residual stress formation. In the SLM process, the thermal stresses accumulate in the layers as the top layer (the layer above the previously printed layer) is heated and cooled down repeatedly. Expansion and shrinkage in existing layers are blocked by the already solidified layers, leading to residual stresses, and residual stress may thus cause deformation of parts as soon as the part is removed from anchors on the build plate. Furthermore, residual stress can affect the performance of a part in service by causing superimposition of applied stresses, especially under subsequent dynamic or fatigue loading, that may result in unexpected failure. Tensile residual stress may also cause stress corrosion cracking on exposed surfaces [3].

The most common techniques for reducing residual stress in SLM are the pre-heating of the build plate or post process stress relief treatment of built parts. Certain SLM process parameters have also been found to have a significant role in affecting residual stresses [1]. Several researchers have thus studied...
the phenomenon of residual stress development in aluminium and other metals produced by SLM and various additive manufacturing processes, developing techniques to reduce such stresses.

Mercelis and Kruth [4] studied the development of residual stress in 316L stainless steel under selective laser melting, noting that residual stresses can be identified as those stresses that remain within the SLM built part once it has reached thermal equilibrium with the environment. Residual stress is generally of two types, tensile and compressive. In the top and bottom layers of SLM built parts, tensile stresses are observed while in between them, compressive stresses are observed. Alongside the thermal gradient mechanism, the cool down phase in SLM induces residual stress. Ali and Mumtaz [5] carried out a study on the effect of scanning strategy on residual stress in titanium alloy Ti6Al4V processed using SLM, reporting that an 90° alternative scan strategy yielded the least residual stress. Buchbinder et al. [6] adopted a preheating strategy to reduce distortion in SLM-made AlSi10Mg parts, noting that a preheating temperature of 250 °C completely eliminated distortion in built parts. Yadroitsave and Yadroitsava [7] investigated residual stress in stainless steel and titanium alloys, similarly finding that preheating the build plate minimised residual stress. Prashanth et al. [8] conducted investigations on AlSi12 tensile samples produced using SLM to examine the effects of heat treatment at various temperatures ranging from 200 °C to 450 °C for 6 hours on mechanical properties. They found that the crystalline size of Si begins to increase after the annealing temperature reaches 200 °C, and that the mechanical properties of AlSi12 alloy thus also changed after the annealing temperature exceeded 200 °C.

The published literature thus reveals that heat treatment can reduce residual stresses in SLM processed metal parts; however, few studies are available on the effects of heat treatment on residual stress on SLM produced aluminium alloys [9, 10], and none have focused specially on SLM-processed AlSi12 alloy. Additively manufactured complex aluminium alloy parts are, however, finding increasing application in the automotive, aerospace, and biomedical sectors due to their excellent mechanical properties. A more in-depth understanding of how residual stresses are affected by post heat treatment of SLM-made aluminium alloys, especially AlSi12 alloy, which has received relatively less attention among SLM processed alloys, is thus required.

This study presents an investigation of the measurement and variation of residual stresses of SLM-processed AlSi12 samples subjected to a range of heat treatment temperatures. The aim was to identify at what heat treatment temperatures residual stresses can be minimised. The residual stress in SLM samples was thus measured experimentally using an X-Ray Diffraction (XRD) technique. This method is particularly useful when near-surface residual stress measurements are required, being generally used for measuring residual stresses arising from machining, peening, or heat treatment of a part. In this study, the post-heat treatment on AlSi12 parts was carried out at 200 °C, 300 °C, 400 °C, and 500 °C. Heat treatment relieves residual stress, and the effect of increases in heat treatment temperature on residual stress was thus investigated to offer a guideline for selecting the appropriate temperature for such heat treatment to minimise residual stress.

2. Material and Methods
AlSi12 metal powder supplied by 3D Systems was used to prepare cylindrical test samples, which were processed using a ProX200 SLM machine equipped with a 300 W fibre LASER (1,070 nm wavelength). Based on optimised parameters, laser power of 285 W, a scanning speed of 1,000 mm/s, a hatch spacing of 100 µm, a layer thickness of 40 µm, a defocus distance of -40 mm/10, and an alternative scan strategy were adopted. The build chamber, which had dimensions of 140 × 140 × 100 mm, was filled with argon gas to purge oxygen to less than 1,000 ppm. The samples were carefully removed from the substrate through wire electrical discharge machining (EDM) method, which is recommended for SLM parts, as it induces the least amount of residual stress as compared to other machining processes.
Residual stresses were measured using XRD for the as-built AlSi12 samples at room temperature and after heat treatment at 200 °C, 300 °C, 400 °C, and 500 °C, respectively. Cylindrical AlSi12 samples of dimensions Ø 10 mm × 8 mm were printed in the SLM machine. However, due to restrictions on size imposed by the XRD machine, these samples had to be reduced in size, and a cutting allowance of 2 mm was used for Electrical Discharge Machining (EDM) to arrive at standard dimensions of Ø8 mm × 8 mm. The AlSi12 samples post-heat-treatment, which had compressive residual stresses, were also reduced from 8 mm to 4 mm in height, as shown in Figure 1.

![Figure 1 AlSi12 sample at Ø8 mm × 4 mm for X-Ray Diffraction.](image)

The heat treatment was conducted using a Gero furnace, which has an operating temperature range of 30 °C to 3,000 °C, and options to combine vacuum and high temperature heat treatment, which was utilised at 200 °C to 500 °C in this study. The heating rate was 10 °C per minute until reaching the maximum required temperature, as shown in Figure 2, then the samples were held at the maximum temperature for three hours prior to cooling. The cooling rate was initially at 10 °C per minute for the first 60 °C, then the rate was reduced, and furnace cooling was allowed until the vacuum was discharged. Figure 2 shows the temperature vs time profiles for two temperatures, 200 °C and 400 °C, as examples. A similar profile was followed for the other two heat treatment temperatures, 300 °C and 500 °C.

![Figure 2 Temperature vs Time graph for heating at 200 °C and 400 °C.](image)

X-Ray Diffraction (XRD) was used to measure residual stresses in SLM-processed AlSi12 samples. As shown in Figure 1, while exposed to X-ray beam, the test points, shown in yellow, positioned at a distance of 3 mm, are scanned and the stress in the X-direction measured. The residual stress is calculated as the average value of stresses measured at the three test locations on the top surface of the
The X-ray interacts with the crystal lattice on the sample and causes diffraction patterns, with the stress measured using the distance between crystal planes (d-spacing). A D8 ADVANCE X-Ray Diffractometer from Bruker was used in this study, which takes about two hours from mounting to capture the XRD data for each sample. The X-rays in an XRD have limited energy and lower penetrating ability, and thus only obtain residual stresses at the surface level, which are compressive in nature. This is evident based on the calculations using equation (1), which provide a negative stress value due to the negative slope of \( m \). The incoming X-rays thus obey Bragg’s law.

For residual stress measurement, lattice strains are measured initially, then the stresses are calculated from these. This is achieved by X-ray diffraction using the \( \sin^2 \psi \) method, where the \( \psi \) angle refers to the tilt angle, ranging between 0° to 45°, of the sample mounted in the diffractometer. According to Anderoglu [11], the residual stresses are then obtained using gradient \( m \) of the linear curve between \( d \) and \( \sin^2 \psi \), which is obtained by measurement of the inter-planar spacing.

The residual stress \( \sigma_\phi \) is then calculated using equation (1), as given in [11]:

\[
\sigma_\phi = \frac{m \left( E \sin^2 \phi \right)}{do}
\]  

(1)

where \( m \) is the slope of \( d \) and the \( \sin^2 \psi \) curve, \( d \) is the distance between lattice spacing, \( do \) is the unstressed interplanar spacing, \( E \) is the Young’s Modulus (73 GPa), and \( \nu \) is the Poisson’s ratio (0.33). The values of \( m \) differ for different heat treatment temperatures.

In this study, the SLM samples for obtaining residual stress were post-machined using electrical discharge machining, allowing the induction of residual stress to be minimised. The prepared AlSi12 sample was then mounted in the XRD machine and measurements obtained for a 90° sample rotation. Based on Poisson’s ratio, when compressive stress is applied, the lattice spacing decreases. The lattice spacing was thus measured from the diffraction angle, and the X-Ray wavelength determined using Bragg’s law [11].

3. Results and Discussion

Figure 3 shows the intensity vs diffraction angle with respect to the scan of the sample seen in Figure 1. The graph of all the XRD patterns at various heat treatment temperatures shows that the XRD patterns have peaks between the diffraction angles for 40 °C and 50 °C. The AlSi12 samples produced by SLM in the vertical orientation were annealed between 200 °C and 500 °C for three hours, and the XRD patterns reveal Al and Si presence. Moreover, maximum peak intensity is observed at 300 °C during the transformation phase of recrystallisation, while at 400 °C, with regard to the first three peaks, the first and the third peak cannot be detected for this phase.

Chemical phase analysis shows that Al is most significant in terms of element percentage composition. At 300 °C, only one peak, at diffraction angle 44.88°, has the highest intensity; all other peaks, including that for Al, are much smaller. At room temperature, the highest intensity was 960 at a 44.8° diffraction angle, while the XRD data at 200 °C shows that although the Al peak was for the 40° to 50° diffraction angle, the intensity was reduced at 44.66°. This could be attributed to thermal softening from a microstructure perspective.

Figure 4 shows the residual stresses corresponding to the heat treatment temperatures represented in Figure 2. This demonstrates variation of the measured residual stress with the temperatures of the as-built and heat-treated samples. Although three samples were printed for each case, two samples were used at the same temperature conditions.
Figure 4 also shows the corresponding representative values of residual stress at each temperature of heat treatment. In this work, compressive residual stress was shown to be reduced through the annealing process. Based on the as-built sample, residual stress in the AlSi12 sample reached a peak level at 72 MPa, which then remained stable. Residual stress reduces as the annealing temperature is increased from 200 °C to 400 °C, however, while at 500°C, the rate of decrease of residual stress is minimal, becoming 36 MPa from the previous 42 MPa. It should be noted that the recrystallisation of AlSi12 alloy occurs between 200 °C and 400 °C, and thus, heat treatment beyond 300 °C is unlikely to be as effective in reducing residual stress. Similar results have been found by other researchers [12, 13] for AlSi10Mg alloy processed using SLM for as-built and heat treated conditions involving X-ray Diffraction measurement. Zhuo et al [12] showed that heat treatment at 300 °C for two hours, followed by quenching, was an effective method of reducing residual stress on the top surface of AlSi10Mg samples with preheating temperatures of 150 °C, while Jiang et al [13] investigated the effects of heat treatment with a range of heating temperatures ranging from 250 °C to 550 °C, at increments of 50°C, for two hours each, on residual stress and mechanical properties, determining that heat treatment at 250 °C reduced the residual stress at the top surface to 90 MPa from the 115 MPa of the as-built sample of AlSi10Mg.
Figure 5 Microstructures of heat treated AlSi12 samples produced by SLM at (A & B) 200 °C, (C & D) 300 °C, (E & F) 400 °C, and (G & H) 500 °C.
3.1 Microstructure of heat treated SLM-made AlSi12 samples

An Olympus BX61 optical microscope was used to analyse the microstructures of the AlSi12 samples. To reveal these microstructures, NaOH (Sodium hydroxide) and water, in the ratio 10 g of NaOH pellets to 90 ml of water, was used as an etchant for an immersion time of 20 seconds. Figure 5 shows the microstructures of the samples heat-treated at 200 °C, 300 °C, 400 °C and 500 °C at two different magnifications in order to display the details of the microstructure more effectively. The magnified images, B, D, F, and G as shown in Figure 5, display finer grain boundaries, which allows better interpretation. In particular, the grain boundary and Si particle growth are of special interest in terms of understanding the mechanical properties.

Based on the micrographs in Figure 5, the size of Si increases when the annealing temperature is increased to 500 °C, most likely due to the rejection of Si from the AlSi12 matrix. At temperatures such as 200 °C and 300 °C, this transformation of boundaries has not yet occurred, and the Si particles are also smaller in size when compared to well defined morphology seen at 500 °C. The increase in annealing temperature thus clearly causes changes in the size of the Si particles, evident in Figure 4 (400 °C to 500 °C). Moreover, the grain boundaries disappear completely when parts are annealed to 500 °C, while microstructure coarsening occurs simultaneously, which would necessarily cause a reduction in residual stress in the AlSi12 samples produced by SLM.

Table 1 shows the residual stress at each temperature investigated, and the corresponding percentage reduction of residual stress as compared to residual stress of the as-built alloy at a room temperature (RT) of 20 °C. It also shows percentage reduction of residual stress from the previous temperature in the table in each case. As the heat treatment temperature rises, a greater reduction in residual stress is observed, yet the rate of reduction from the previous value decreases up to 300 °C before increasing again. Heat treatment conditions for the reduction of residual stress should therefore be carefully tuned, as microstructure and mechanical properties also change with increasing temperatures beyond recrystallisation: the higher the heat treatment temperature, the larger the grain size. The cooling rate was kept as the same for all the samples heated to temperatures of 200 °C to 500 °C. As the heat treatment temperature increased, the Si particles clearly grew larger and the grain boundaries disappeared. This suggests that heat treatment of 200 °C to 300 °C may be more appropriate for reducing residual stress without significantly affecting any mechanical properties.

| Heat Treatment Temperature °C | Residual stress MPa | Reduction from RT (%) | Reduction from previous value (%) |
|-------------------------------|---------------------|------------------------|-----------------------------------|
| 20 (RT)                       | 72                  | 0                      | 0                                 |
| 200                           | 52                  | 28                     | 28                                |
| 300                           | 50                  | 31                     | 4                                 |
| 400                           | 42                  | 42                     | 16                                |
| 500                           | 36                  | 50                     | 14                                |

4. Conclusions

This paper investigated the effect of heat treatment at different temperatures on the residual stress developed in AlSi12 alloy parts manufactured using the SLM additive manufacturing process, based on XRD measurement of residual stress. The XRD results were further strengthened by microstructure examination, which demonstrated an increase in Si particle size with heat treatment temperature, which contributes to the coarsening of the microstructure, and in which the number of Si particles reduces while the size increases. Moreover, these Si particles are deposited in the boundary of the Al-Si matrix. The residual stress measured decreased with increasing temperatures, with reductions of 28% and 31%
at heat treatment temperatures of 200 °C and 300 °C, respectively, from the residual stress in the baseline sample at room temperature.

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References

[1] Ponnusamy P, Masood S, Ruan D, Palanisamy S and Rashid R 2018 High strain rate dynamic behaviour of AlSi12 alloy processed by selective laser melting Int J Adv Mfg Tech 97(1-4) 1023-35.
[2] Ponnusamy P, Masood S, Ruan D, Palanisamy S, Rashid RR, Mukhlis R and Edwards NJ 2020 Dynamic compressive behaviour of selective laser melted AlSi12 alloy: Effect of elevated temperature and heat treatment Addit Manuf 101614.
[3] Zhang W, Fang K, Hu Y, Wang S and Wang X 2016 Effect of machining-induced surface residual stress on initiation of stress corrosion cracking in 316 austenitic stainless steel Corros Sci 108 173-84.
[4] Mercelis P and Kruth JP 2006 Residual stresses in selective laser sintering and selective laser melting Rapid Prototyp J 12/5 254–265.
[5] Ali H, Ghadbeigi H and Muntaz K 2018 Effect of scanning strategies on residual stress and mechanical properties of Selective Laser Melted Ti6Al4V Mat Sci Eng A 712 175-87.
[6] Buchbinder D, Meiners W, Pirch N, Wissenbach K and Schrage J 2014 Investigation on reducing distortion by preheating during manufacture of aluminum components using selective laser melting J Laser Appl 26(1) 012004.
[7] Yadroitsev I and Yadroitseva I 2015 Evaluation of residual stress in stainless steel 316L and Ti6Al4V samples produced by selective laser melting Virtual Phys Prototyp 10(2) 67-76.
[8] Prashanth KG, Scudino S, Klauais HJ, Surredi KB, Löber L, Wang Z, Chaubey AK, Kühn U and Eckert J 2014 Microstructure and mechanical properties of Al–12Si produced by selective laser melting: Effect of heat treatment Mat Sci Eng A 590 153-60.
[9] Ponnusamy P, Masood S, Ruan D, Palanisamy S, Rahman R and Kariem M. 2018 High strain rate behaviour at high temperature of AlSi12 parts produced by selective laser melting. IOP Conf Series: Materials Science and Engineering; 377(1) 012167.
[10] Ponnusamy P, Rahman Rashid RA, Masood SH, Ruan D and Palanisamy S 2020 Mechanical properties of SLM-printed Aluminium alloys: A Review Materials.
[11] Anderoglu O. Residual stress measurement using X-ray diffraction: Texas A&M University; Masters Thesis, 2004.
[12] Longchao Zhuo, Zeyu Wang, Hongjia Zhang, Enhui Yin, Yanlin Wang, Tao Xu, Chao Li 2019 Effect of post-process heat treatment on microstructure and properties of selective laser melted AlSi10Mg alloy. Materials Letters, 234, 196-200.
[13] Xiaohui Jiang, Wenjing Xiong, Lianfeng Wang, Miaoxian Guo and Zishan Ding 2020 Heat treatment effects on microstructure-residual stress for selective laser melting AlSi10Mg Materials Science And Technology 36, 2, 168–180.