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Influence of stress relieving thermal cycles on AISI10Mg specimens produced by selective laser melting

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Abstract: The AlSi10Mg alloy is known for its castability, corrosion resistance, high heat conductivity, low weight and good weldability. Applications of the aluminium alloy components includes heat exchangers that are subjected to high temperatures and corrosive environments. The production process of AlSi10Mg alloy using selective laser melting has been extensively explored because of its hypoeutectic composition of Al and Si. However, it has been observed that stress relieve has a negative **consequence** on tensile properties of SLM built samples, while retaining and improving ductility. The thermal behaviour of AlSi10Mg alloy fabricated by selective laser melting with the specimen heat treated at 300°C for 2 hours investigated in this work. The specimens exhibited mass gain during the thermalgravimetric analysis as a result of the presence of oxygen as one of the elements present as detected by the elemental analysis. The fatigue results post thermal treatment demonstrated improved fatigue resistance qualities comparable to die-casted AlSi10Mg.

1. Introduction

Conventional methods such as casting for component production are slowly being substituted by additive manufacturing (AM) methods which are currently under investigation especially in the fabrication of multifaceted structures [1]. As a category of the AM technology for the manufacturing of the metal components from powder, selective laser melting (SLM) makes an interesting study for aluminium alloys. These alloys are becoming the most desirable metals to different industries due to its properties such as high thermal and conductivity of electricity, low specific weight, low power radiation, high plasticity and ductility, high weldability as well as toxicity [2]. AlSi10Mg as one of the composite matrix, is acquiring a reputation in the automobile, aerospace and rail applications to substitute the monolithic counterparts [3]. Components such as heat exchangers and propellers, which are used in numerous mechanical engineering systems have depended on conventional manufacturing methods for production, which due to manufacturing confinements have affected the designs complexities [4] [5, 6]. These components can be produced by AM as an alternative manufacturing process. However, one of the main drawbacks of AM is the need for post built thermal treatment to relieve the inherent residual stresses experienced during the rapid heating and cooling cycles of the process.



In this case, the modifying of the mechanical properties of SLM materials by thermal treatment customarily is based on the techniques which have been established for materials that are manufactured using conventional methods [7]. Oxidation of aluminium alloys during AM processing has also been studied [8], where it was determined that the oxygen in the chamber is capable of forming thin oxide films on the molten and solid material which may lead to porosity in the material. Brandl et al. suggested that when aiming to increase fatigue resistance, combining different heating profiles such as stress relieving and peak-hardening is a good idea [9-11]. Francolin et al. [12], used thermal gravimetric analysis (TGA) to prove the influence of oxygen on physical and mechanical properties of scaffold sheets. However little information is available on the TGA analysis of SLM produced AlSi10Mg.

Since some components like heat exchangers are subjected to high temperatures and cycling loads, the kind of temperatures and fatigue conditions that these sample can withstand, as well as the improvements that needs to be done, will be studied. Therefore, this study aims to investigate the thermal and fatigue behaviour of stress relieved AlSi10Mg manufactured by selective laser melting.

2. Experimental Procedure

2.1. Samples production

The AlSi10Mg samples were built using the SLM Solutions M280, with fixed parameters of; 150W power, 1000 mm/s scan speed, 50 μm hatch spacing and 50 μm powder layer thickness (see [13] for base plate images). The samples were built in three different orientations namely, 0° horizontal (A), 45° oblique (B) and 90° vertical (C). The samples were stress relieved using a vacuum furnace at 300°C for 2 hours, and furnace cooled to room temperature in an inert atmosphere, which is a casted AlSi10Mg parts heat treatment profile.

2.2. Characterization

JEOL JSM 6010 Plus/LA, scanning electron microscope (SEM), fitted with Electron Diffraction Spectroscopy (EDS) detector, was used for EDX analysis at an accelerating voltage of 20 kV. The samples were cut, mounted, polished and etched using Keller's reagent prior to SEM and EDX characterisation. For thermal analysis, samples of 20 mg filings were prepared for the Perkin Elmer Pyris 1 TGA thermogravimetric analyser, under conditions of 50 to 900 °C at a rate of 10 °C/min for thermogravimetric analysis in a Nitrogen environment. The 20kN Zwick/ Roell Tensile tester was used applying ASTM E466 standard for axial fatigue cycle tests, with a load varied from 80 – 120 MPa, and 0.1 stress ratio. The number of fatigue cycles ranged from 230 000 to 2 000 000 at the frequency of 10 Hz. The samples were stopped after 2 million cycles.

3. Results and discussion

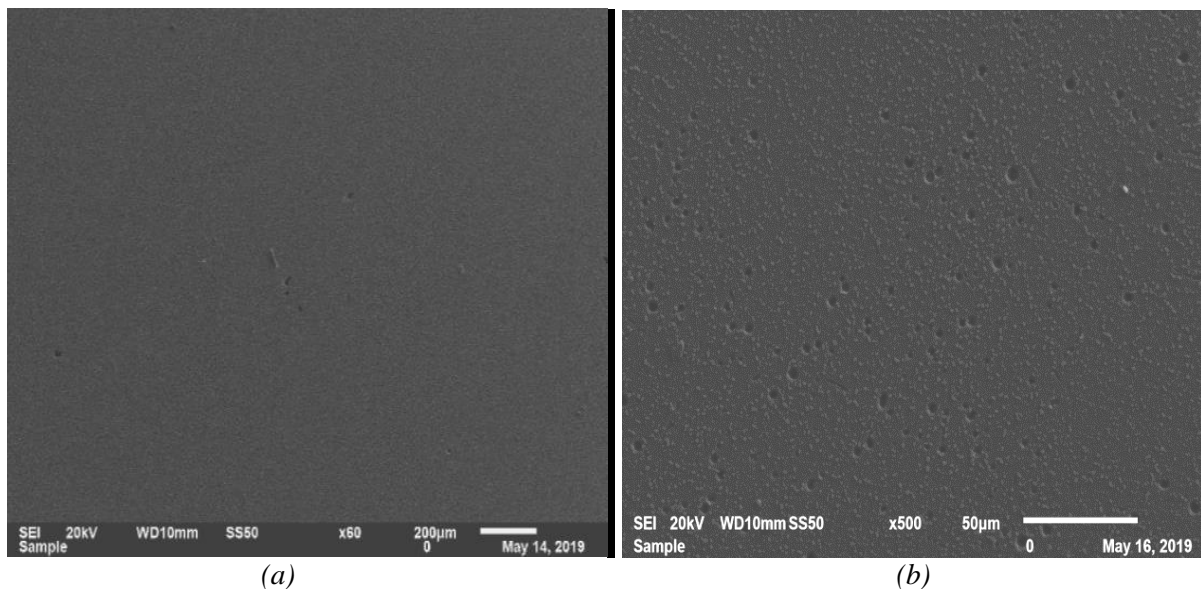
3.1. Energy Dispersive Spectroscopy (EDS)

The EDS results presented in table 1 demonstrates the atomic percentages of the elements present in the samples. Some of the areas of the samples also comprised of oxygen as one of the elements present in them. Louvis [8], attributed this presence of as a result of a system chamber that is not completely oxygen free. This oxygen level of 0.1-0.2% is high enough to affect aluminium alloys, [2], and capable of forming oxide layers on two different scanned tracks.

Table 1: EDS results of the SLM produced AlSi10Mg samples showing atomic% of element content in each microstructure (AB-as-built, SR-Stress relieve)

ELEMENTS	ATOMIC %					
	A-AB	B-AB	C-AB	A-SR	B-SR	C-SR
Mg	1.07	1.22	1.21	1.00	1.39	1.02
Al	89.9	92.14	92.14	85.10	89.53	89.47
Si	7.72	5.66	5.49	12.3	9.09	9.51
O	1.33	0.99	1.16	1.60	0.00	0.00
TOTAL	100.00	100.00	100.00	100.00	100.00	100.00

Figure 1 and 2 presents the SEM images and EDX spectra respectively, of the samples before and after stress relieving, which were focused on certain areas of the microstructure at 60X and 500X magnification. It was observed that silicon is the second major element in all the samples. It was also observed that post thermal treatment, there is an increase in silicon elemental composition which precipitate to the surface while aluminium composition decreased. This is because during the cooling and solidification, silicon contracted and during post thermal treatment, it precipitated as silicon and Mg₂Si [14, 15]. Aboulkhair [1], stated that during rapid cooling, α -aluminium is permitted to solidify first into a cellular structure while residual silicon is left to form at the boundaries of the grain. The silicon is only allowed to disperse during the coarsening of the microstructure subsequent to post thermal treatment.

**Figure 1:** SEM-EDS area images of the SLM produced AlSi10Mg samples showing (a) 60X and (b) 500X magnifications

Due to the higher electron backscattering coefficient, pores and oxide particles are easily distinguished from one another through the brightness threshold which makes oxide particles appear brighter than pores [16]. In Figure 1b, silicon is observed to have precipitated to the surface after stress relieve (indicated by the light spots on the surface).

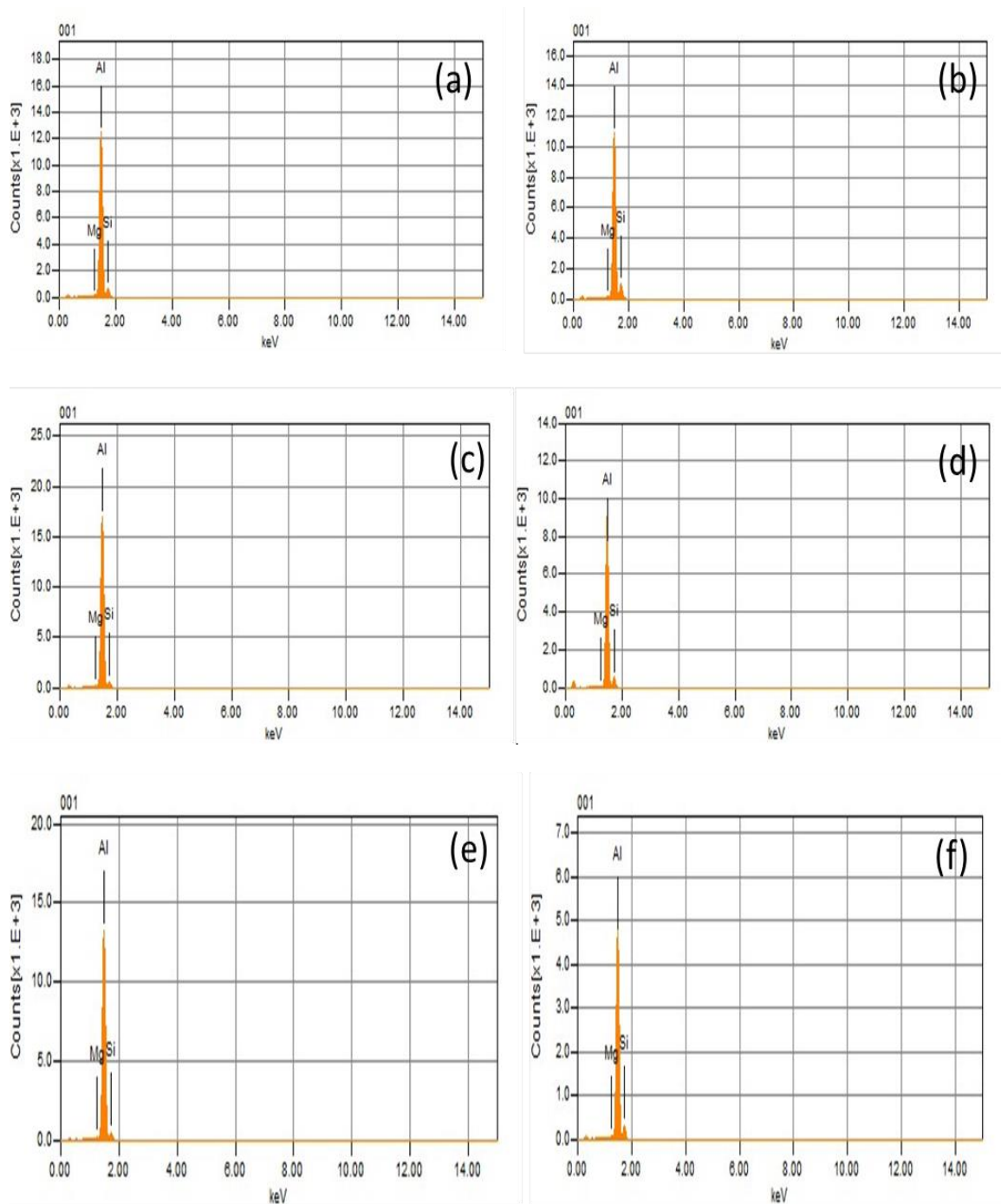


Figure 2: SEM-EDS results of the SLM produced AlSi10Mg samples showing (a,c,e) as-built and (b,d,f) post thermal treatment

3.2. Axial Fatigue

The stress relieved samples for batch A (0°) and C (90°) were further analysed for axial fatigue in order to determine the influence that the thermal treatment process has on the properties. Figure 3 a&b presents the dog-bone samples used in this test. It should be noted that not all samples fractured during the test, some samples were run out after 2 000 000 cycles. The axial fatigue results are presented in Figure 4, with the data in this work indicated in yellow, showing the number of cycles as a function of the maximum stress. The results were compared to the literature data of AlSi10Mg and T6 heat treated AlSi10Mg, [17]. Comparing the obtained results with the literature values for SLM processed AlSi10Mg (red) and die-casted Al6061 that was T6 heat treated (blue), it was observed that the samples

can withstand higher fatigues and are comparable to literature values of the same load range of 80 – 100 MPa [18].



Figure 3: (a) Dog-bone sample used for axial fatigue tests (b) Fractured fatigue samples after testing.

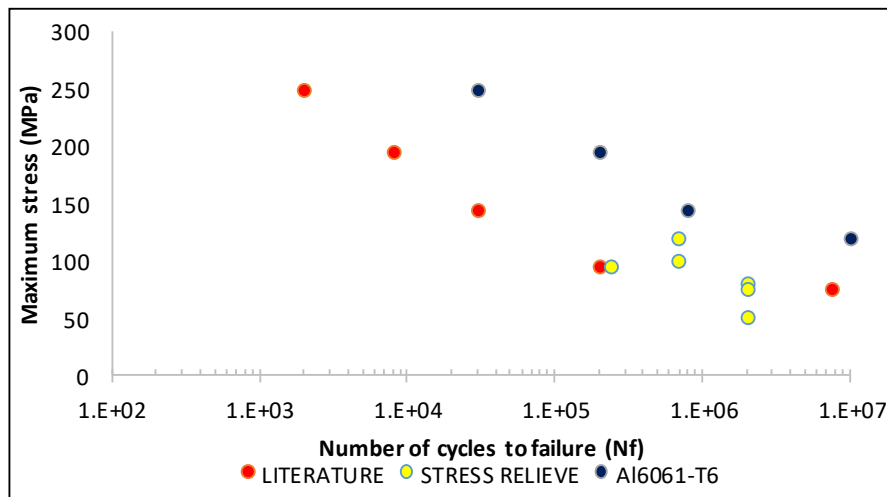


Figure 4: Axial fatigue results of the SLM produced AlSi10Mg samples

These results show that the even though the stress relieving profile used in this case resulted in a decline in strength, it increased the fatigue life of the samples. Most studies on fatigue life are based on the behaviour of cast aluminium alloys such as A360 which was believed to be affected by microstructural defects such as porosity, spacing of cellular and dendrite arms as well as grain size and inclusions [9, 16, 19]. Major and serious fatigue cracks found at the *extension of the defect size dissemination* are found to be the cause of failure in die-casting [20], whereas with AM components defects that lead to probable fractures are generally caused by oxide films. Alumina is easily formed as oxide film on the powder particles since aluminium has a great affinity to oxygen. *Therefore*, the residual porosity exposed on the surface of the component is the substantial to dictate of fatigue life for SLM produced AlSi10Mg [16].

3.3. Thermogravimetric Analysis (TGA)

Figure 5 presents the TGA results of the as-built and post heat treatment samples. In the Figure 5a, the anisotropy in build direction is observed in the variation of the graphs. The as-built thermal behaviour of orientation B (45°) stands out from A (0°) and C (90°), in the range of temperatures between 25 and 400 °C, showing weight loss that is associated with the material defects decomposition. At 595 °C all the specimens loose mass due to melting, which is the melting point of aluminium. On the other hand, the post stress relieving plots presented in Figure 5b showed no variation as a function of build orientation, which made all the structures homogeneous. The weight loss and subsequent gain was also

observed at the same temperature, as seen for the as-built samples. The specimen showed a weight gain due to aluminium reacting with oxygen and forming aluminium oxide as Tang noted the high affinity they have to one another [16]. In this case, the AlSi10Mg can withstand operational conditions of temperatures above its melting point of 595 °C due to the reaction of aluminium with oxygen to form aluminium oxide which melts at 2072 °C [16].

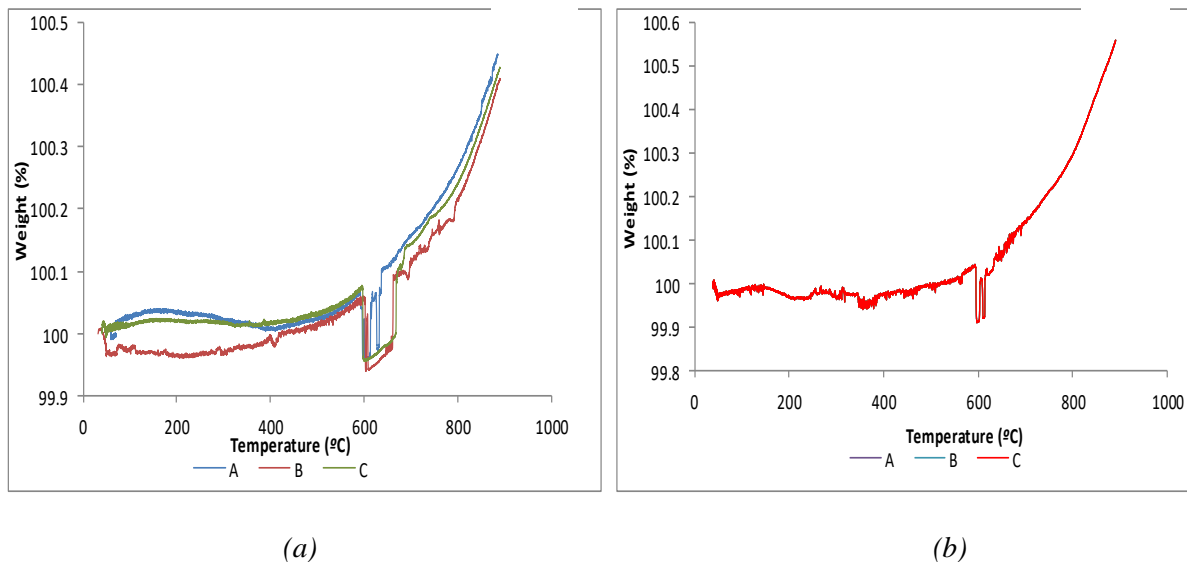


Figure 5: TGA results of the SLM produced AlSi10Mg samples showing (a) As-built and (b) Post thermal treatment

4. Conclusion

The EDX elemental analysis showed a change in the Si and Al content before and after stress relieving, where Si increased and Al decreased. This was also seen from surface morphology of Si. Axial fatigue measurements on the stress relieved samples were comparable to T6 heat treated and die-casted results obtained in literature. The thermal behaviour of the SLM produced AlSi10Mg changed with the post build stress relieving, but there was anisotropy observed only for the as-built samples. In this case, the presence of oxygen content in the microstructure as observed through EDX, contributed a positive influence on the TGA results of SLM produced AlSi10Mg components. It can thus be concluded that although the stress relieving of components results in drastic strength loss, it improved the fatigue life, where the residual stresses were modified.

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