ARTICLES
MECHANICAL BEHAVIOURS OF SELECTIVE LASER MELTING 316L STAINLESS STEEL

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1.0 INTRODUCTION

316L stainless steel is famous for its outstanding corrosion resistance [1-3]. It has been adopted in several applications of biomaterial and chemical industries [4-7]. In the present, 316L stainless steel parts are fabricated using traditional routes such as forging, extrusion or casting. However, these routes are not allowed for complex parts' preparation, and the last part has to be welded or machined into final form. Therefore, these processes are time-consuming. In addition, welding may effect intergranular corrosion in the 316L stainless steel [8], while, the material is relatively costly for machining. Hence, while looking for an alternative, new innovative technologies for additive manufacturing have been fast developed. For instance, the Laser Engineered Net Shaping (LENS) and Selective Laser Melting (SLM) processes can be used for the production of stainless steel parts [9-14].

SLM is one of the innovative approaches of the laser additive techniques for the manufacture of near-net-shape parts. This technique has been explored to make production even more productive and economical [15-21]. Through this technique, the near-net-shaped part can be produced in a single step, that cut down time and costs connected with intermediary processes and the quantity and type of tools used in traditional technology. The fabrication processes involved in SLM technology are similar to the binder jetting process. Briefly, the thermal source is employed to induce fusion between powder particles to a specific region of a build area, via layer-by-layer deposition, to produce a precise shaping of three-dimensional structure geometry. Despite this, the SLM fabricated elements exhibit a particular micro and macro structures that affect the properties which are a bit dissimilar from those conventionally manufactured parts [22-24]. The unusual behaviour of the materials processed using SLM requires the necessity to establish the association between structures, process parameters and properties [23-27].

SLM technique is presently being verified for direct fabrication of complex metal parts from a wide-ranging of engineering materials including cobalt-based alloys, titanium-based alloy, stainless steels and nickel-titanium [28-31]. Findings show that this technique enhances the materials functional and structural properties matched to materials gained using traditional methods [24, 32, 33]. For instance, the formation of finer grains obtained by the SLM parts increased both yield and ultimate tensile strength of the 316L stainless steel as compared to the traditionally processed [34-36]. Moreover, the part of 316L stainless steel formed using the SLM technique shows an ultimate tensile strength of 640 MPa and a yield strength of 490 MPa [36]. A literature data discussed mainly to the features that optimise the fabrication process namely porosity reduction and geometrical precision [34, 37-39]. The SLM parts, porosity was found to correlate with hatching parameters such as the laser power, scanning velocity and building angle.
It was found that the increase of scan speed from 100 to 300 mm/s, elevated the size and the quantity of the pores of the SLM 316L stainless steel parts [42]. Another research on SLM 316L stainless steel suggested that formation of porosity cause an inverse function of hardness property [43]. The mechanical properties and phase composition of one of the laser additive-manufactured 316 stringers were accessed [44]. They pointed out that, besides some cracks and pores, the tensile properties were akin to the results of other literature. However, the microstructure analysis done exhibited the existence of only the austenite phase. On the other hand, the SLM 316L stainless steel revealed the existence of the martensitic phase in the austenite (γ) matrix [44-46].

The microstructure of materials is affected by recrystallisation, phase transformation, and precipitation. In terms of heat treatment of austenitic stainless steels, it does not give a vital effect on strength and hardness [47, 48]. This is because strengthening of the materials usually depends on characteristics of steels' phase transformations, precisely the austenite-martensite. However, this phase transformation is almost impossible and tough to develop since stainless steels have low carbon content, i.e., less than 0.01% [49, 50]. Nevertheless, strength and hardness of the stainless steels can be increased through the formation of a large dislocation density at their grain and cell boundaries via crystallization [50], through rapid solidification induction [51], and through the existence of in-situ established oxide nano-inclusions [52-54]. In a recent study by [55] reported that at a temperature higher than 1100 °C, a dual austenite-ferrite phase assembly is formed from an almost pure austenite phase. It is notable that the mechanical properties of parts processed by SLM show an increased yield strength compared to wrought 316 L stainless steel [56, 57]. Heat treatments impacts the SLM-ed parts have been reported by [58], [59], [15] and [60]. However, stress relieving treatments do not show a major change in mechanical properties and grain size. The nature of SLM exhibits the continuous process of rapid heating and cooling of the alloy powders. Thus, left the as-built SLM produced parts with high residual stresses [61-63]. These internal stresses are believed to affect the mechanical and corrosion properties. Hence, in order to relieve these stresses, heat treatments are applied after the SLM process.

In this research, a more comprehensive approached was adopted. The SLM system was used to manufacture 316L stainless steel parts for different energy density, building orientation, and heat treatments. Such comprehensive work has not been reported in the literature before this. The properties of the fabricated SLM parts are investigated. The resultant microstructure and its effect on the physical property, tensile strength, microhardness and chemical properties of the obtained material are methodically discussed.

2.0 EXPERIMENTAL

3.1 Powder Characterization

As-received nitrogen gas atomised 316L stainless steel powder supplied by SLM Solution GmbH, Germany was used in this study. The chemical composition of the as received 316L stainless steel consisted of C, Mn, P, Cr, Ni, Mo, Si and Fe is shown in Table 1. The as-received powder morphology is examined for its shape and size by using scanning electron microscopy (SEM) and laser scattering particle analyser, respectively.

SEM examination of the as received 316L stainless steel powder exhibits the powder particle morphology as shown in Figure 1. Mainly, spherical-shaped particles with some smaller satellite particles attached can be observed with a mean powder particle size of 30.6 µm. The powder size distribution is considered favourable from the viewpoint of SLM technology as it is within 12-63 µm [64]. This ensured an excellent flowability of the powder.

<table>
<thead>
<tr>
<th>Elements</th>
<th>Chemical Composition (wt%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>0.01</td>
</tr>
<tr>
<td>Mn</td>
<td>0.99</td>
</tr>
<tr>
<td>P</td>
<td>0.02</td>
</tr>
<tr>
<td>Cr</td>
<td>16.7</td>
</tr>
<tr>
<td>Ni</td>
<td>10.3</td>
</tr>
<tr>
<td>Mo</td>
<td>2.2</td>
</tr>
<tr>
<td>Si</td>
<td>0.69</td>
</tr>
<tr>
<td>Fe</td>
<td>Balance</td>
</tr>
</tbody>
</table>
3.2 Powder Characterization

The fabrication of 316L stainless steel samples with standardised geometry for the physical characterisation, static mechanical testing and chemical rate testing were carried out on SLM machine SLM®125HL. The samples were produced under the argon atmosphere in order to prevent the probability of powder contamination with nitrogen or oxygen. In this study, two sets of parameters were used to fabricate different sample conditions as shown in Table 2. Different sets of processing parameters represent different sets of energy density which are 60 (low) and 65 (high) J/mm³. The energy density (J/mm³) was determined from Equation 1. Note that the LOW energy density applied high power input which is > 200 W, while the HIGH energy density applied low power inputs, typically ≤ 200 W.

A flat dog-bone shape sample was designed according to ASTM D638- Type V standard and generated using SOLIDWORKS 2013 software. The samples were designed to be built with three different building orientations which are 0, 45, and 90° orientation for each set as in Figure 2. Following the SLM fabrication process is the post-processing of the as-built 316L stainless steel samples to detach the samples from the mild steel building plate using Electrical Discharge Machining (EDM) wire-cut. The remaining support structures on the produced samples were removed. The samples were prepared for another set of samples conditions. The samples were either left with no heat treatment or subjected to three different heat treatment (HT) cycle as shown in Table 3 before the testing. In this study, the “as-built” (AB) condition is referred to as the untreated SLM-ed samples.

Energy Density = Laser Power
(Scanning Speed x Hatch distance x Layer Thickness)

Table 2. SLM processing parameters for 316L stainless steel samples.

<table>
<thead>
<tr>
<th>Condition</th>
<th>HIGH-BO0-AB</th>
<th>HIGH-BO45-AB</th>
<th>HIGH-BO90-AB</th>
<th>LOW-BO0-AB</th>
<th>LOW-BO45-AB</th>
<th>LOW-BO90-AB</th>
</tr>
</thead>
<tbody>
<tr>
<td>Energy density (J/mm³)</td>
<td>65</td>
<td>65</td>
<td>65</td>
<td>60</td>
<td>60</td>
<td>60</td>
</tr>
<tr>
<td>Laser power (W)</td>
<td>175</td>
<td>175</td>
<td>175</td>
<td>275</td>
<td>275</td>
<td>275</td>
</tr>
<tr>
<td>Scan speed (mm/s)</td>
<td>750</td>
<td>750</td>
<td>750</td>
<td>760</td>
<td>760</td>
<td>760</td>
</tr>
<tr>
<td>Hatch spacing (mm)</td>
<td>0.12</td>
<td>0.12</td>
<td>0.12</td>
<td>0.12</td>
<td>0.12</td>
<td>0.12</td>
</tr>
<tr>
<td>Layer thickness (µm)</td>
<td>30</td>
<td>30</td>
<td>30</td>
<td>50</td>
<td>50</td>
<td>50</td>
</tr>
<tr>
<td>Building orientation (°)</td>
<td>0</td>
<td>45</td>
<td>90</td>
<td>0</td>
<td>45</td>
<td>90</td>
</tr>
</tbody>
</table>
Table 3. Heat treatment condition to SLM-ed 316L stainless steel samples.

<table>
<thead>
<tr>
<th>Samples</th>
<th>Heat Treatment Cycle</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-built (AB)</td>
<td>No heat treatment</td>
</tr>
<tr>
<td>650 HT</td>
<td>650 °C for 2 hours, furnace cooling</td>
</tr>
<tr>
<td>950 HT</td>
<td>950 °C for 2 hours, furnace cooling</td>
</tr>
<tr>
<td>1100 HT</td>
<td>1100 °C for 2 hours, furnace cooling</td>
</tr>
</tbody>
</table>

### 3.3 Materials and Method

Standard microstructure procedure was used for the microstructure observation. Samples for the microstructure observation were prepared and etched with 7.5 mL of nitric acid, 5.0 mL of hydrofluoric acid, and 37.5 mL of distilled water for 5 minutes. An optical microscope (OM) was used for the microstructure examination is MEIJI-MT 7100 optical microscope. Image J software was used to carry out quantitative image analysis. The 316L stainless steel powder and SLM representative samples were subjected to XRD diffraction to identify the phase composition and possible preferential crystallographic orientations influenced by the SLM process. While, high resolution of scanning electron microscope SEM SU5000, equipped with an EDS analysis system and Hitachi ion milling were used to observe higher magnification images.

An analytical balance with ±0.0001 g precision was used to determine the density of SLM-ed samples based on Archimedes’ principle. This method considers the whole volume of the samples. Three SLM-ed samples for each condition were weighed in air and distilled water, following the standard of ASTM B962-15. Relative density of the sample was calculated by the ratio of the differences in the sample density to the density of the reference material.

The tensile samples in the as-built and heat-treated (650, 950, 1100 °C/2 hours/furnace cooling) condition were tested for their yield strength, ultimate tensile strength, and elongation at fracture. The test was carried out using the Instron 5900 Universal Testing Machine. Vickers hardness machine MATSUZAWA Type MMT X7 was used to perform the hardness test using the Zwick Roel tester. Corrosion tests were conducted using WPG100e Potentiostat/Galvanostat with saturated calomel electrode (SCE) reference electrode and a platinum counter electrode. The exposed surface area of the samples was 0.25 cm² and performed in Ringer’s solution at 37 °C. The generated polarisation curves were used to get the estimated quantitative data using Tafel plots.

### 3.0 RESULTS AND DISCUSSION

#### 3.1 Structure analysis of the SLM-fabricated 316L stainless steel

The nominal density of 316L stainless steel, 7.99 g/cm³, was referred to calculate the relative density of SLM-build samples as illustrated in Figure 3. The combination of process parameters for different energy densities (60 and 65 J/mm³) showed that a total number of 16 samples with high relative densities (>98%) were obtained in this research work. The Archimedes’ method used provides a good approximation of internal porosity, but not the distribution of the pores in each volume of the sample. Generally, the HIGH energy density shows higher density compare to LOW energy density.

![Figure 3. Relative density of the 316L stainless steel parts processed by SLM at various energy densities for as-built conditions.](image-url)
HIGH energy density shows a little inconsistency of density values which could be contributed by spattering phenomena. Usually, any small spatter particles would be dissolved into a layer after a next scanning. However, big spatter particle might not be able to dissolve well during the next scanning, thus, leaving it partially melted and an assortment of molten metal that cause defects in the form of irregular pores formation. The edge of the 45° building orientation samples leave partial of the layers exposed. When the spattering occurs at this area, the particle lands on the layer surfaces and pores remain open.

The porosity of each of the as-built samples was analysed at the central area of the metallographic part using images analysed using an optical microscope (OM). The OM observations confirmed the presence of macro-pores at the as-built SLMed 316L stainless steel. The porosity quantitative analysis, i.e., involving the percentage of porosity and pores distribution, was generated by ImageJ software.

Based on Table 4, the lowest percent of porosity is shown by 90° BO of HIGH energy density at 0.10%, while the highest is shown by 0° BO of LOW energy density at 0.73%. Generally, the HIGH energy density exhibits a lower percent of porosity than LOW energy density for 0° and 90° BO. It is also shown from Figure 4.8 that the pores are widely scattered. In this research, diverse values of energy densities have been controlled by different laser powers and layer thicknesses. In the case of LOW energy density, the processing parameters involve includes a higher layer thickness of 50µm compares to 30µm for HIGH energy density. Although the laser power used for LOW energy density is 275W which is higher than 175W used for HIGH energy density, the laser is not enough to infiltrate deep into the lower layer during the SLM process. The new and former layers are unable to fuse completely, and thus, cause voids to occur in the interlayer. The existence of voids also causes some particles to trap and unmelt during the SLM process.

Table 4. The percentage porosity of SLM as-built samples.

<table>
<thead>
<tr>
<th>Condition</th>
<th>HIGH-BO0-AB</th>
<th>HIGH-BO45-AB</th>
<th>HIGH-BO90-AB</th>
<th>LOW-BO0-AB</th>
<th>LOW-BO45-AB</th>
<th>LOW-BO90-AB</th>
</tr>
</thead>
<tbody>
<tr>
<td>Percentage porosity (%)</td>
<td>0.41</td>
<td>0.29</td>
<td>0.10</td>
<td>0.73</td>
<td>0.22</td>
<td>0.17</td>
</tr>
</tbody>
</table>

Porosity is a general defect exhibited in metal-AM parts, and it can be influenced by various processing parameters such as layer thickness, laser power, and scan speed. Porosity can be divided into two types, namely process-induced porosity and gas-induced porosity [66]. The gas-induced pores are typically spherical. They could occur during the gas atomisation of the feedstock proceeding to SLM process and maintain their existence in the final parts. Nevertheless, the non-spherical process-induced pores are developed when either: (i) insufficient energy is applied, resulting in a failure to fully melt the powder which causes inadequate fusion between successive layers and between each adjacent layers [67]; or (ii) the energy applied is excessive which cause spatter ejection [68].

In this research, the spherical pores conquered the non-spherical pores as can be seen in Figure 4. The spherical pores can either be produced due to the existence of contamination or moisture on the powder particles’ surface [69]; or through the reactions between the small amount of oxygen and carbon present during SLM processing, hence, cause carbon monoxide or carbon dioxide gases to trap in the produced parts [70]. On the other hand, more non-uniform pores with corner points can be observed in low energy density samples (Figure 3 (d-f)) compared to high energy density samples. These corner points could initiate microcrack and variation in strength, thus, lower the strength of materials. The factors of pores formation could be due to the variant in surface roughness [71], and the layer-wise manufacture way of the AM process. The variant in surface roughness of the low energy samples leads to inconsistency of powder distribution which causes uneven melt flow and an unbalanced molten pool in the succeeding layers [72].

The decrease of porosity tends to cause an increase for relative density and mechanical properties of SLMed 316L stainless steel material as proven by many literatures [73, 74]. Porosity is restricted to such hatch trails, instigation microcracks at the triple points because of stress concentration at those sites, could lead to lesser ductility.
Figure 4. Macro-pores distribution for HIGH energy density at (a) 0°, (b) 45° and (c) 90° building orientations, and LOW energy density at (d) 0°, (e) 45° and (f) 90° building orientations.
### 3.2 Microstructure Analysis

Figure 5 reported the XRD spectra for as-received 316L stainless steel powders and SLM-ed samples. For the SLM-ed samples, they are in as-built condition or heat treated (HT) at different temperature of 650, 950 and 1100 °C. The austenite was identified using JCPDS-ICDD- card No. 33-0397. From the inspection of the diffractometric data, as shown in Figure 4, it is proven that all samples possibly related to the austenitic phase. The 316L stainless steel powder exhibits a typical austenitic phase crystallographic peaks for the following FCC planes of (111), (200) and (220) at 2-theta values at 43.55, 50.72 and 74.60, respectively. For the 316L stainless steel as-built sample, a typical austenitic phase crystallographic peaks for the following FCC planes of (111), (200) and (220) at 2-theta values at 44.05, 51.23 and 75.17, respectively. Meanwhile, for 650°C heat-treated sample, the typical austenitic phase crystallographic peaks for the following FCC planes of (111), (200) and (220) are shown at 2-theta values of 43.92, 50.97 and 74.82, respectively; 950°C heat-treated sample sample at 2-theta values of 43.95, 51.12 and 74.92, respectively; and 1100°C heat-treated sample at 2-theta values of 43.78, 51.08 and 74.92, respectively.

The result reveals the existence of merely single-phase austenite is found in the as-received powder, the as-built sample, and the various temperature heat treated samples. This can be ascribed to the case that austenitic stainless steels’ phase transformation relies greatly on the cooling rate as well as the alloy’s chemical composition particularly the Cr/Ni ratio. Provided that the Cr/Ni ratio is low, the chance of ferrite formation is concealed [49, 75, 76]. Moreover, during the SLM process, the rapid solidification leads to an increase of high density of dislocation, thus, inhibit the phase transformation to happen [77, 78]. Unexpectedly, no major change of the peak intensities was established between SLM as-built and heat-treated samples. This is contrary to the discoveries in other works reported [55, 79, 80]. The absent preferred orientation can be attributed by the scanning direction rotation after each layer during the SLM process, hence, modify the direction of the thermal gradient of every layer. As a result, it reduces the potential form of strong texture [35]. Nevertheless, the peaks of the as-built and heat-treated samples are wider than that of the as-received powder. This is due to the existence of residual stress and dislocations induced by the SLM process, as reported by [34, 81].

It is well-informed that any stainless-steel welds with only a single-phase austenite microstructure are more crack sensitive compare to those with a few numbers of delta ferrite. The presence of delta ferrite cause the size of austenite grain to refine and the total number of grain boundaries to increase [82], which could hinder crack propagation. With the absence of delta ferrite, this also reveals to be the reason for high elongation of single-phase austenite parts [83].

![Figure 5. XRD diffraction patterns for as-received precursor 316L stainless steel powder and SLM manufactured samples](image-url)
FE-SEM images taken on mechanically polished and ion milled surfaces of the 316L stainless steel shown in Figure 6 and 7 disclosed the formation of grain structures and nano-sized pores for 90° BO for as-built and 950°C heat-treated samples. 950°C heat-treated samples were selected due to the significant differences than as-built samples in terms of mechanical properties values. Figure 6 (a-b) of the as-built samples shows a complex small size of irregular grains, ascribed to the temperature gradient caused by rapid solidification. A similar microstructure can be observed in 950°C heat-treated samples as shown in Figure 7 (a-b), with the only difference being the size and shape of the grains. The grains of the as-built samples tend to be small and equiaxed as shown in Figure 6 (a-b). However, at heat-treated samples, the grains become slightly larger and more equiaxed (Figure 7 (a-b)). It has been reported that preferential grains’ orientation can be achieved through heat treatment [84, 85]. However, this research work shows that the grains of the heat treated samples are still inconstantly oriented and the heat treatments do not contribute in texture formation, as exhibits by XRD in Figure 5.

The FE-SEM images in Figure 6 (c-d) and 7 (c-d) revealed the existence of randomly disseminated small pits-like microvoids (black spots) inside the grain which consist of larger irregular pits-like size and smaller spherical pits-like. For the spherical pits-like, their sizes extend from several tens nanometers to hundreds of nanometers. These are the unique features of the nano-scales structure of SLM 316L stainless steel. Similar characteristics were also reported by [81] and [34]. According to [86], these features might be known as oxide nano-inclusions. The nature of the rapid cooling process in SLM contributes to the formation of oxide nano-inclusions. [86] reported that the nano-inclusions morphology could be associated with an increase of molten silicates viscosity and their inclination to coagulate in a spherical shape to lessen surface tension. The formation of nano-inclusions happen through chemical reactions between an active element in the starting 316L stainless steel powder (like silicon) and the traces oxygen gas exist in the processing chamber at the very high temperatures induced by the laser radiation interacted with the melted powder and form melted oxide droplets [81]. Nano-inclusion is believed to be rich with Mo, Si, O. In order to confirm the small pit-like features, the content of its chemical composition should be checked on the nano-particles. However, since the micro- and nano-voids inside the grain are very small in size, it was impossible to identify the particles; chemical composition via EDS due to the limitation of the EDS measure volume used in this study.

Many recent studies have reported the findings of nano-inclusion structures in SLM-ed 316L stainless steels [34, 52, 81, 87, 88]. They confirmed the existence of the structures as silicon rich oxides nano-inclusions. In addition, another TEM-EDX analysis done by [87] highlighted the existence of 14% of Mn in similar silicates. The well-distributed nano-inclusions are believed to assist pinning dislocation flow during mechanical stress and thus perform as strengthening action. Homogeneous distributions of small pits-like are exposed in the FE-SEM images reported in Figure 6 (c-d) and Figure 7 (c-d) of this study. However, the size and amount of small voids are different. At Figure 6 (c) which represents the as-built sample condition for LOW energy density, the amount and size of the voids are larger when compare to the HIGH energy density (Figure 6 (d)). A similar situation is seen at Figure 7 where a larger amount and size of the nano-voids are observed for the 950°C HT sample condition for LOW energy density compare to the HIGH energy density. The LOW energy density samples consist of a combination of larger prismatic micro-voids ranging from more than 0.5 μm to 1.5 μm, and smaller spherical the small nano-voids with a diameter ≤ 0.5μm; while HIGH energy density consist of mostly smaller nano-voids. Likewise, the amount of nano-inclusions are larger at the as-built samples than the 950°C HT samples for LOW and HIGH energy densities, respectively. The voids size are slightly bigger than the reported silicon and manganese-rich oxide inclusions of SLM samples [52]. [89] reported that large irregular shaped of titanium and aluminum oxide around 10 to 100 μm in 18Ni(300) SLM maraging steel parts detriment the mechanical properties. This can be associated to the micro- and nano-voids features in which the large amount and size of the voids are believed to give demerit for the mechanical properties performances, thus, contribute to the lower value of yield strength, ultimate tensile strength and hardness of the LOW energy density compare to HIGH energy density. Similarly, the value of yield strength, ultimate tensile strength and hardness for heat treated samples deteriorise when compare to as-built samples due to increased numbers of irregular voids.
Figure 6. FESEM images showing the as-built SLMed 316L stainless steel condition for (a) LOW energy density and (b) HIGH energy density at medium magnification. Image (c) and (d) show the higher magnification of specimens for LOW and HIGH energy densities, respectively.
3.3 Microstructure Analysis

Illustrative engineering stress (σ) versus strain responses of both low and high energy densities samples tested are displayed in Figure 8. Tensile properties such as ultimate tensile strength (UTS), yield strength (YS), and elongation at failure elicited from these plots are listed in Table 5. In order to compare, literature records on conventionally manufacture 316L stainless steel are also listed. A significant enhancement (of up to ~40%) in YS due to the SLM process can be distinguished. Contrarily, the improvement in UTS is only borderline at ~10%. These enhancements in strength come at the ductility’s expense, which is lowered by about 13% in 90° building orientation, and even more noticeably by ~60% and ~75% in 0° and 45° building orientations, respectively.

From the mechanical properties data listed in Table 5, it is clearly shown manufacturing of 316L stainless steel works via SLM process contributes to a significant advantage where elevating yield strength can be derived. Nevertheless, the increase in UTS is relatively small than the commercially manufacture alloy. Hence, exhibits the different level of work hardening of SLM alloys compares to commercially manufacture austenitic stainless steel. The difference between the YS and UTS values of the as-built SLM samples than the commercially manufactured 316L stainless steel are contributed by the high dislocation density found in the SLM samples. Another notable decrease in tensile ductility of SLM-ed 316L stainless steel is due to lack of work hardening, in accordance with the necking Considère's criterion.

Reduction of alloys unnecessarily makes an alloy brittle. Indeed, the lowest percentage elongation at failure, at 18.70% for as built with high ED at 45° BD case is regarded as fairly ductile. Furthermore, no additional forming steps such as forging or rolling of the SLM-ed works required as the SLM process is already for the near-net shaped final product. In relation, the ductility reduction does not essentially seem like a serious detriment, especially when it comes to the application potential of the SLM-ed 316L stainless steel works.
Table 5 Mechanical properties of 316L stainless steel fabricated by Selective Laser Melting and conventional manufacturing process.

<table>
<thead>
<tr>
<th>Condition</th>
<th>Yield Strength (MPa)</th>
<th>UTS (MPa)</th>
<th>Elongation (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-Built with low ED</td>
<td>332.3</td>
<td>555.6</td>
<td>22.3</td>
</tr>
<tr>
<td>As-Built with high ED</td>
<td>446.1</td>
<td>552.9</td>
<td>18.7</td>
</tr>
<tr>
<td>Conventionally manufacture 316L SS*</td>
<td>220–270</td>
<td>520–680</td>
<td>40–45</td>
</tr>
</tbody>
</table>

*Source: [56]

Tensile tests were performed for SLM as built and heat-treated samples. Figure 7 represent the stress-strain curves for all tested samples, showing a great combination of ductility and strength. Referring to Table 6 residual stress is believed to play roles in the significant changes of tensile properties. This situation can be proven with the changes of values for the heat-treated samples compare to the as-built condition. The heat treatments which acted as stress reliever shown a marginal impact on yield strength, ultimate tensile strength, and elongation at break, as it encourages strength decline and ductility increase. These phenomena can be associated with the microstructure alterations, i.e. minor cellular coarsening as the result of a stress reliever. The changes in tensile properties are not statistically major between the as built and 650°C heat treatment, but noticeable at 950°C and higher heat treatments temperature. In general, the strength of the samples reduces as the heat treatment temperature increases. There are some probable justifications for this condition. Firstly, it may be due to the existence of immiscible elements at the interface of solid-liquid, that probably solidify at the boundaries [90, 91]. This indicates a very fine cellular microstructure with little sub grains to form along with dense dislocation in each individual grains [92]. Secondly is the presence of misorientations in these sub grains of cellular microstructure [93], that prevents the movement of dislocations and hinder the development of a fracture.

The most significant difference between the SLM as-built samples and heat-treated samples is the deterioration of both YS and UTS with increasing heat treatment temperatures (Table 6). As mentioned before, no preferred grains’ orientation was observed. Hence, these consequences are likely because of the variations of microstructural that happen upon heat treatment. Additionally, the misorientation of sub grain is inversely proportional to the heat treatment temperature [93]. The dislocations density reduces after heat treatment. In other word, there are insufficient barriers to convey plastic deformation and delay the movement of dislocation. As a result, the mechanical properties of the heat-treated samples are declined.

Table 6 shows that the elongations at fracture for the as built 316L stainless steel samples were about 20%. The elongation did not show significant changes when heat-treated at 650°C, but it rose significantly after been heat treated at temperature 950°C and slightly increase or decrease when heat-treated at 1100°C. The LOW energy density shows the most significant increase of elongation to approximately 50% compared to HIGH energy density to only 22% at the heat treatment of 950°C. The LOW energy density SLM samples that were heated treated at 950°C shows a ductility that is compatible with the conventionally manufacture 316L stainless steel but higher in yield strength and ultimate tensile strength.

![Stress-strain curves of as-built 316L stainless steel after the SLM process at room temperature for low (a) and high (b) energy densities.](image-url)

**Figure 8.** Stress-strain curves of as-built 316L stainless steel after the SLM process at room temperature for low (a) and high (b) energy densities.
The Vickers hardness test measurements obtained for the as-built condition is shown in Figure 9. The more significant value of the as-built samples compares to wrought 316L stainless steel, i.e., with approximately 200.0 HV, can be attributed to the high dislocation concentration in 316L stainless steel. The parts constructed at HIGH energy density are shown stronger than the corresponding parts constructed at LOW energy density. The similar trend is shown by 650°C heat-treated samples condition. At 950°C and 1100°C heat-treated samples conditions, the HIGH energy density exhibits higher hardness values compared to LOW energy density for all BD cases.

The influence of energy density over a varied range of values on the hardness of 316L stainless steel is supported by many literatures. Many researches demonstrate similar findings where for energy density that lies in the range between 48 and 125 J/mm², the hardness tends to increase linearly with energy density [43, 94, 95]. The hardness value is hugely influenced by the relative density and the amount of porosity of the material. In relation, the hardness increases with the decrease of porosity, and contrariwise. As porosity and hardness are conversely associated, the sample with the biggest energy density has the smallest porosity, hence, exhibits the highest hardness, and vice versa. Besides that, the FESEM analysis reveals the existence of micro- and nano-voids in the SLM samples. The larger amount and size of voids features in LOW energy density than HIGH energy density contributed to the decrease of hardness values in LOW energy density.

Table 6. Average values of yield strength, ultimate tensile strength, and elongation at fracture of SLM as-built and heat treated samples.

<table>
<thead>
<tr>
<th>Condition</th>
<th>Yield Strength (MPa)</th>
<th>UTS (MPa)</th>
<th>Elongation at fracture (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Low ED</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>As-built</td>
<td>332.33</td>
<td>554.15</td>
<td>24.9</td>
</tr>
<tr>
<td>650 HT</td>
<td>370.56</td>
<td>567.69</td>
<td>21.7</td>
</tr>
<tr>
<td>950 HT</td>
<td>268.08</td>
<td>535.53</td>
<td>67.8</td>
</tr>
<tr>
<td>1100 HT</td>
<td>253.94</td>
<td>537.84</td>
<td>65.7</td>
</tr>
<tr>
<td><strong>High ED</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>As-built</td>
<td>446.13</td>
<td>552.88</td>
<td>18.7</td>
</tr>
<tr>
<td>650 HT</td>
<td>408.19</td>
<td>568.94</td>
<td>18.4</td>
</tr>
<tr>
<td>950 HT</td>
<td>291.39</td>
<td>526.2</td>
<td>25.97</td>
</tr>
<tr>
<td>1100 HT</td>
<td>274.94</td>
<td>512.91</td>
<td>25.37</td>
</tr>
</tbody>
</table>

The rise of temperature. Consequently, the pinning effect within the dislocations is weakening, thus, lead to a decrease in microhardness of the SLM samples with the increase of heat treatment temperature up to 1100°C. Besides that, higher residual stress level commonly leads to a higher microhardness value. Hence, the constant values decrease after 650°C heat treatment temperatures suggested the probability of the residual stress release at upper temperatures. The reduction of microhardness values after heat treatments can also be contributed by the increase of the amount of the irregular-shape micro- and nano-voids features as mentioned before.
3.4 Corrosion Properties

Figure 10 illustrates the potentiodynamic polarisation curves of as-built and heat treated SLM-ed 316L stainless steel samples in Ringer's solution at 37°C for low and high energy densities. For low energy density (Figure 9 (a)), the corrosion potential of the as-built SLM-ed 316L stainless steel exhibits a value of approximately -0.23 mV vs. SCE. The corrosion potential then reduced by approximately 0.16 mV at 650°C, 0.18 mV at 950°C, and 0.19 mV at 1100°C. Meanwhile, for high energy density (Figure 9 (b)), the corrosion potential of the as-built SLM-ed 316L stainless steel has a value of approximately -0.24 mV vs. SCE. The corrosion potential then slightly reduced by approximately 0.05 mV at 650°C and 0.02 mV at 950°C. At 1100°C, the corrosion potential show more significant decrease by approximately 0.22 mV. In general, the corrosion potentials slightly decrease with the increase of heat treatment's temperatures.

The effect of the heat treatment on the decrease of corrosion potentials can be ascribed to several factors. The existences of large pores with the larger exposed surface area make the SLM-ed 316L stainless steel samples prone to corrosion attack. In a more corrosive environment, smaller pores with a size smaller than 10 μm will lose its passive condition and can be corroded. Such pores make passive films that act as a barrier for the material’s surface from external chemical reaction to be further susceptible to breakdown. In relation to that, the porosity of samples increased to a certain extent after heat treatment. This is proven by the increased amount and size of micro- and nano-voids features as discussed previously. As a result, the passive films are weaker at the increased amount and size of pores of the SLM-ed 316L stainless steel with the increased of heat treatment temperatures, hence, lead to the reduction of corrosion potentials. The reduction of corrosion potentials can be ascribed to the decrease in dislocation density and relief of the compressive residual stresses via the heat treatment [98]. Nevertheless, the underlying relationship between corrosion potentials and residual stress can be inferred from the results in previously mentioned on the effect of heat treatments of the mechanical properties of SLM-ed 316L stainless steel samples.
Figure 10. Potentiodynamic polarisation curves of SLM-ed 316L stainless steel at as-built and different heat treatment temperatures for (a) LOW and (b) HIGH energy densities.

Figure 11 shows the effect of the as-built and heat treated SLM-ed 316L stainless steel process on the corrosion rate. The different of corrosion rate at different energy density is most significant during 650°C HT and 950°C HT which are 85.4% and 167.2%, respectively. While at as-built, 950°C HT and 1100°C HT, the LOW energy density shows higher corrosion rate than HIGH energy density, an inverse trend is exhibited at 650°C HT showing the inconsistency of the effect of energy density on the corrosion rate values. However, at different heat treatment, i.e. at 650°C HT and 950°C HT, a significant effect is observed. In general, the data exhibits a lower corrosion rate than 2.5 x 10^{-4} mm/year, which is the acceptable corrosion rate for metallic implant [99, 100]. This result also reveals the microstructure and pores in the SLM-ed 316L stainless steel could be controlled at a minimum level through suitable SLM process parameters and heat treatment strategies, leading to a susceptible corrosion behaviour in the Ringers’ solution.
4.0 CONCLUSIONS

This paper has presented the mechanical characterisation of fabricated SLM-ed 316L stainless steel samples. Experimental works have been performed with a purpose to study the relationship between energy density and heat treatment with the resultant microstructure, mechanical and chemical properties. High relative densities (>98%) are achieved for all as-built samples condition. The relative density increases with the increase of energy densities. Insufficient laser penetrated into powder layer and its correlation with pore formation leads to the decrease in relative density. The quality of SLM products is crucially affected by energy densities. The highest amount of porosity exhibited leads to the lowest hardness and strength of the low energy density (60 J/mm³) samples. The combination of pores and high residual stress lead to lower hardness and strength of as-built SLM-ed 316L stainless steel samples.

The hardness, yield strength, and ultimate tensile strength declined after heat treatment due to a decrease in dislocation density, compress stress relief, and an increase in number and size of small pits-like features. Improved ductility with elongation at fracture from around 40% to 85% was reached after the heat treatment at 950°C and above for LOW energy density (60 J/mm³) SLM-ed 316L stainless steel samples. The slight reduction of corrosion potential with the increased of heat treatment temperatures are associated with the existence of large pores and the increased of amount and size of small pores, i.e., in the form of small pits-like features; the decline of dislocation density; and residual stress released.

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References


Witt, G. and J. Sehr, Static strength analysis of beam melted parts dependent on various influences. in 21st Annual International Solid Freeform Fabrication (SFF) Symposium, Austin. 2010.


