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EFFECT OF HEAT TREATMENT ON THE PROPERTIES OF ADDITIVELY MANUFACTURED TYPE 316L STAINLESS STEEL

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ABSTRACT
Thin wall test articles were made using Fusion Powder Bed processing of Type 316L stainless steel. This hollow structure was fabricated using a Renishaw AM250. The material was characterized in the as fabricated condition using optical, scanning electron microscopy, and tensile testing. The tensile behavior indicates high initial residual stress due to the higher than annealed material yield strength. A series of thermal treatments were investigated to determine the effect on mechanical properties and microstructural evolution. In order to determine these effects, miniature flat tensile test samples and metallographic samples were wire electrical discharge machined from the structure. These samples were vacuum heat treated at 870, 1040 and 1080ºC for 30 to 120 minutes and metallographically examined to determine the microstructural evolution. The samples were tensile tested and the data are evaluated based on the power-law type relationships. AM Type 316L SS exhibits less strain hardening than wrought stainless steel. In addition, the ductility is somewhat lower in the as-fabricated condition compared to the heat-treated condition. The results of the testing and the rationale for these different behaviors will be discussed.

INTRODUCTION
Additive Manufacturing (AM), the fabrication of components in a layer by layer manner using from a computer aided design (CAD) file that is parsed into processing layers and solidified using powder feedstock and a heat source, is becoming a mainstream approach to fabricate high value components that are difficult to produce using conventional means (1-4). For instance, General Electric is using AM to fabricate fuel nozzles that were comprised of dozens of parts joined together and now can be fabricated directly. AM can be accomplished using several different approaches and heat sources. These include Laser Powder Bed Fusion (L-PBF), electron beam powder bed fusion (EB-PBF) and directed energy powder or wire processes. The process chosen will depend on a variety of factors that include the required fidelity of the part, the requisite surface condition, the desired properties, the availability of feedstock, etc. (5). L-PBF processing conditions have primarily been developed for titanium, aluminum and stainless steels.

Austenitic stainless-steel (SS) alloys, like Type 316L SS, are good candidates for AM since they have well behaved solidification modes, are routinely laser welded and have well defined processing parameters (6-8). AM can generate internal defects, however, from providing insufficient energy to melt and fuse the layers resulting in lack of fusion defects, or keyhole defects from excessive energy density and rapid cooling rates trapping porosity within the structure. Despite the possibility of producing these defects, 316L SS is widely used to fabricate AM components and test articles. In fact, L-PBF has been proposed to fabricate heat exchanger tubing. The materials, both actual and surrogate have been studied extensively at Savannah River National Laboratory (9-11). The efforts to date have been on the optimization and characterization of the stainless steel in the as-fabricated condition. Although there is interest in stress relief heat treatments and hot isostatic pressing to achieve full density.

As-fabricated austenitic SS when processed by L-PBF often-times exhibit dislocation structures that are suggestive of significant levels of cold work. These dislocations are caused by the rapid cooling and levels of constraint during solidification and cooling. The dislocation tangles and loops and density of dislocations manifest themselves in the relatively high yield strength, compared to annealed wrought SS, of as-fabricated materials. The appearance of cold work type defects is also observed in the lower elongation to failure measured in some AM materials (12-14).
LB-PBF AM results in significant levels of residual stress. The effect of the residual stress can result in lower ductility and potentially lower fracture toughness. This study was undertaken to understand the effects of heat treatment on the tensile properties of L-PBF processed Type 316L SS. Samples were removed from surrogate materials that were prepared as part of the fabrication of a coil component. Samples were heat treated at a subcritical annealing temperature to annealing temperatures and the properties are compared to the properties of wrought Type 316L SS. The details of the results are discussed.

RESULTS AND DISCUSSION
Figure 3 shows the samples as they were cut from the center of the square tubes. Note that they are parallel to the fabrication direction. The metallographic samples were taken from these remnants.

The as-received microstructures both parallel and perpendicular to the build direction are shown in Fig. 4. The as-received microstructure parallel to the build direction, Figs. 4a-c exhibits a “fish scale” structure due to the melt pool solidification in the vertical orientation. Periodic horizontal lines are visible at low magnification, Fig. 4a, where the Renishaw controls software changes the melt pattern. The grains and melt lines are visible and the grains appear to extend across the melt pool lines, Figs. 4b and 4c. The presence of isolated porosity is evident in these images at high magnification. The microstructure perpendicular to the build direction is shown in Figs 4d-4f. It exhibits the “basket weave” microstructure, clearly shown in Fig. 4d at low magnification, associated with the scanning pattern typically used for L-BPF. Evidence of the melt pool is visible in Fig. 4e. Randomly oriented grains are apparent at high magnification in Fig. 4f. The sample also some tiny porosity.

The samples were heat as indicated above and were examined metallographically. All of the samples exhibited microstructural evolution during the heat treatment with some conditions more clearly causing changes. The microstructure evolution for each temperature is shown in Figures 6-8. These images indicate that recrystallization has occurred and the grains are growing. In addition, the evidence of the prior melt boundaries are eliminated. This feature can be seen at temperatures as low as 870°C for 30 minutes, Figure 6a. The presence of the melt interfaces is not observed at all for the 1040°C / 30 minute exposure. 870°C is considered below the heat treatment temperature of the 300 series stainless steels.

An attempt to measure grain growth for each of the exposure conditions was made; these results are not conclusive since the microstructure is complex and the presence of the grain boundaries is not obvious in many of the micrographs, but a typical grain boundary is shown by the arrows. The results are presented in Table 2. The standard deviations are listed and are fairly large. However, there is some correlation of increased grain size with increased time and temperature, as expected.
The tensile properties of a forged Type 304L stainless steel were tested as part of another project; the results for a typical orientation of this material are shown in Figure 8 for reference. This material exhibits a fairly low strain hardening curve which is consistent with the amount of cold work associated with the forging process and yield strength. The material exhibits tensile elongations that are greater than 50%.

The tensile curves for the AM type 316L SS in the as-fabricated condition is shown in Figure 9. These samples also exhibit a low hardening exponent. As a simple method to compare the hardening exponents, the ratio of the UTS to the YS was calculated. Using this value, it is apparent that the baseline (BL) sample has the lowest effective hardening. This result is likely due to the extensive dislocation network and relatively high residual stresses present in SLM produced AM structures (16,17).

The tensile properties after the 870°C heat treatment exhibit varying results as shown in Fig.10. There was a significant increase in the yield strength of one of the samples with an increase in the ratio of UTS to YS (hardening factor) in all of the samples compared to the baseline. It is surmised that the increased yield strength is associated with changes to the grain size of the individual samples, although metallographic examination of the associated samples was inconclusive.

Figure 11 shows the tensile curves after the 1040°C heat treatment. All the samples exhibit similar yield and tensile strengths. The relative hardening factors are also similar for these samples. This temperature is consistent with the suggested annealing temperature so the significant reduction in yield strength results are consistent with what is expected for traditionally produced Type 304L materials as indicated in Table 2, annealed bar (B) and plate (P), however, the UTS and ductility are lower. Since the samples were cut from as fabricated material, and there are both pores and some surface defects, the UTS and elongation may be adversely affected by these conditions. Other researchers have shown that internal defects and build defects adversely affect ductility measurement.

Figure 12 compares the tensile properties of the samples heat treated for 2 hours at the three temperatures. These data indicate a modest decrease in yield strength with increasing temperature which may ultimately be attributable to grain size effects if additional characterization is conducted. The slight increase in elongation with temperature may be due to the reduced dislocation density or the random nature of porosity in these materials. Further these effects may be potentially convoluted with surface effects and random porosity in the samples.

Figure 13 shows the summary tensile strength data for AM, forged and comparison data. These results clearly indicate that the YS of AM and annealed AM thin blades lie within the nominal properties while the UTS is somewhat lower than expected (7, 18 & 19). Again, lower UTS may be attributed to the presence of the defects that cause lower ductility. The summary data are presented in Table 3.

CONCLUSIONS

Simple heat treating of SLM AM Type 316 L SS does not affect the internal defects formed during additive manufacturing. Heat treatment reduces the yield strength of the material as dislocations and other strengthening mechanisms for austenitic SS are removed from the microstructure. The ultimate tensile strength is moderately affected. The elongation properties of AM materials are dominated by the residual porosity and by the surface conditions.

Heat treatment can be used to decrease the yield strength, but it has less effect on the elongation since the defect structure dominates. Other measures of ductility, reduction in area, for instance, may provide a more complete measurement of the effects of heat treatment as well as the defects on AM materials.

Full annealing is accomplished under conditions that are similar to those used for conventional materials, i.e., 1040°C for 2 hours.

ACKNOWLEDGMENTS

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### TABLE 1. FABRICATION PARAMETERS FOR THE SAMPLE BUILD.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Renishaw 316L SS Build 4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Powder size</td>
<td>15-45µm</td>
</tr>
<tr>
<td>Laser Power used</td>
<td>120 W</td>
</tr>
<tr>
<td>Layer thickness</td>
<td>50 µm</td>
</tr>
<tr>
<td>Scan Pattern</td>
<td>Stripe—5 mm</td>
</tr>
<tr>
<td>Beam Size</td>
<td>75 µm</td>
</tr>
<tr>
<td>Point distance</td>
<td>60 µm</td>
</tr>
<tr>
<td>Point exposure</td>
<td>80 µs</td>
</tr>
<tr>
<td>Effective Velocity</td>
<td>0.64 m/s</td>
</tr>
<tr>
<td>Hatch Spacing</td>
<td>100 µm</td>
</tr>
<tr>
<td>Volume Power</td>
<td>200 W</td>
</tr>
</tbody>
</table>

### TABLE 2. MEASURED GRAIN SIZES OF THE SAMPLES AFTER HEAT TREATMENT.

<table>
<thead>
<tr>
<th>Condition</th>
<th>Grain Size (µm)</th>
<th>Standard Dev.</th>
</tr>
</thead>
<tbody>
<tr>
<td>BL</td>
<td>26</td>
<td>4.4</td>
</tr>
<tr>
<td>870°C/120 m</td>
<td>31</td>
<td>5.2</td>
</tr>
<tr>
<td>1040°C/120 m</td>
<td>33</td>
<td>6.0</td>
</tr>
</tbody>
</table>
TABLE 3. TENSILE AND COMPARISON DATA FOR THE AM AND FORGED MATERIALS COMPARED TO THE STANDARDS (18 & 19)

<table>
<thead>
<tr>
<th></th>
<th>YS</th>
<th>UTS</th>
<th>Ef</th>
<th>UTS/YS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Forged</td>
<td>477</td>
<td>703</td>
<td>0.5</td>
<td>1.47</td>
</tr>
<tr>
<td>BL</td>
<td>390</td>
<td>506</td>
<td>0.23</td>
<td>1.30</td>
</tr>
<tr>
<td>870/30</td>
<td>417</td>
<td>643</td>
<td>0.28</td>
<td>1.54</td>
</tr>
<tr>
<td>870/60</td>
<td>344</td>
<td>521</td>
<td>0.33</td>
<td>1.51</td>
</tr>
<tr>
<td>870/120</td>
<td>333</td>
<td>503</td>
<td>0.22</td>
<td>1.51</td>
</tr>
<tr>
<td>1040/30</td>
<td>313</td>
<td>498</td>
<td>0.30</td>
<td>1.59</td>
</tr>
<tr>
<td>1040/60</td>
<td>304</td>
<td>501</td>
<td>0.35</td>
<td>1.65</td>
</tr>
<tr>
<td>1040/120</td>
<td>299</td>
<td>484</td>
<td>0.27</td>
<td>1.62</td>
</tr>
<tr>
<td>1080/120</td>
<td>282</td>
<td>476</td>
<td>0.32</td>
<td>1.69</td>
</tr>
<tr>
<td>Annealed B</td>
<td>235</td>
<td>640</td>
<td>0.76</td>
<td>2.72</td>
</tr>
<tr>
<td>Annealed P</td>
<td>330</td>
<td>590</td>
<td>0.64</td>
<td>1.79</td>
</tr>
</tbody>
</table>

FIG. 1. SPECIMEN COIL, FABRICATED FOR OTHER TESTING, AND TOWER, INDICATED BY ARROW, WHICH WAS USED FOR HEAT TREATMENT STUDIES.

FIG. 2. DIMENSIONS OF THE MINI-TENSILE SAMPLES THAT WERE WIRE EDM FROM THE TOWERS. THE THICKNESS OF THE SAMPLES WAS NOMINALLY 0.7 mm.

FIG. 3. APPEARANCE OF THE SQUARE TUBE AFTER MINI-TENSILES WERE MACHINED.
FIG. 4. AS-FABRICATED TYPE 316L SS SHOWN PARALLEL (A-C) AND PERPENDICULAR (D-F) TO THE BUILD DIRECTION; A) LOW MAGNIFICATION, B) MODERATE MAGNIFICATION, C) HIGH MAGNIFICATION. SHOWN PERPENDICULAR TO THE BUILD DIRECTION; A) LOW MAGNIFICATION, B) MODERATE MAGNIFICATION, C) HIGH MAGNIFICATION. D) LOW MAGNIFICATION, E) MODERATE MAGNIFICATION, F) HIGH MAGNIFICATION. NOTE GRAINS EXTENDING ACROSS LASER MELT LINES, AT WHITE ARROWS IN (C). NOTE SMALL PORES AT BLACK ARROW IN (C).

FIG. 5. MICROSTRUCTURE OF AM TYPE 304L SS PERPENDICULAR TO THE BUILD DIRECTION AFTER HEAT TREATING AT 870°C FOR (A) 30 MINUTES (60) MINUTES AND (C) 120 MINUTES.

FIG. 6. MICROSTRUCTURES OF AM TYPE 316L SS PERPENDICULAR TO THE BUILD DIRECTION AFTER HEAT TREATING AT 1040°C FOR (A) 30 MINUTES (60) MINUTES AND (C) 120 MINUTES. NOTE THAT THE LASER MELT POOL LINES HAVE BEEN ELIMINATED, SEE FIGURE FIG. 4E FOR COMPARISON.
FIG. 7. MICROSTRUCTURE OF AM TYPE 304L SS AFTER HEATING TREATING FOR 120 MINUTES AT (A) AS FABRICATED (B) 870°C (C) 1040°C AND (D) 1080°C.

FIG. 8. TENSILE PROPERTIES OF FORGED TYPE 304L STAINLESS STEEL, PARALLEL TO FORGING

FIG. 9. TENSILE PROPERTIES OF AM TYPE 304L SS IN THE AS-FABRICATED CONDITION.

Fig. 10. TENSILE DATA FOR THE SAMPLES HEAT TREATED AT 870°C FOR 30 TO 120 MINUTES COMPARED TO A TYPICAL AS FABRICATED SAMPLE (RED CURVE).
Fig. 11. TENSILE DATA FOR THE SAMPLES HEAT TREATED AT 1040°C FOR 30 TO 120 MINUTES COMPARED TO A TYPICAL AS FABRICATED SAMPLE (RED CURVE).


Fig. 12. TENSILE DATA FOR THE SAMPLES HEAT TREATED AT 870°C, 1040°C, AND 1080°C FOR 120 MINUTES COMPARED TO A TYPICAL AS FABRICATED SAMPLE (RED CURVE).