INVESTIGATION OF THE PROPERTIES OF DIRECT ENERGY DEPOSITION ADDITIVE MANUFACTURED 304 STAINLESS STEEL

S.E. Hoosain¹*, L.C. Tshabalala², S. Skhosana², C. Freemantle³ & N. Mndebele¹

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One of the main considerations in adopting the additive manufacturing (AM) technology is whether the material properties of the AM-produced part are comparable with the wrought material. This study compared the properties of AM-produced 304 stainless steel via the direct energy deposition (DED) AM process with wrought 304 stainless steel. The samples were studied in their as-built or as-received condition; and the characterisation analysis included micro-structural evaluation, hardness tests, Charpy impact testing, tensile testing, and X-ray diffraction. The results demonstrated that, in general, the strength characteristics of the AM sample exceeded those of the wrought material other than for toughness.

1 INTRODUCTION

Additive manufacturing (AM) is the process of building 3D parts and components directly by adding material layer by layer, based on digital models [1]. AM is specifically suited to low-volume and complex part production, as AM has significantly fewer manufacturing constraints than traditional CNC manufacturing processes [2-4]. This has enabled design engineers to optimise the geometry of a part according to its service environment without the traditional manufacturing restrictions. In the field of metal AM, two different processes are used: powder bed fusion (PBF) and direct energy deposition (DED). DED uses a focused thermal energy in the form of a laser to fuse materials continuously by melting during deposition [5]. It is used to refurbish or to add material to existing parts. DED machines typically use a nozzle that is...
mounted on a multi-axis arm that can move in multiple directions, allowing for variable deposition and thus the manufacture of complex shapes [6].

One of the key considerations when adopting the technology is the material properties of an AM-produced part compared with those of wrought material. Stainless steels are well known for their resistance to corrosion, creep, and high-temperature applications. There is an increasing demand for components made from stainless steel in a variety of applications in the aerospace, automotive, and railway industries [2-3,7]. 304 stainless steel is the most common stainless steel material. It contains both chromium (between 18% and 20%) and nickel (between 8% and 10.5%) metals as the main non-iron constituents. The AISI 304 austenitic stainless steel investigated in this study has excellent weldability, producing products with high strength, high ductility, and good corrosion resistance in the natural environment. A key factor in 304 stainless steel’s final properties is micro-structural evolution during processing: AM produces unique microstructures owing to the rapid heating and cooling rates of the laser process and the layer-by-layer build method [8]. This includes a very fine and highly anisotropic and crystallographically textured microstructure as a result of high solidification rates and possibly non-equilibrium phases in the as-built state [9]. In the DED processing of 316L stainless steel, it is observed that, in certain regions, the micro-segregation during solidification leads to enrichment of the ferrite stabilising elements Cr and Mo, and thus to fine ferritic structures within the austenitic matrix [10,11].

This study focuses on the comparative properties of wrought 304 stainless steel and AM-produced 304 stainless steel. The materials are compared in their respective as-received or as-built condition. The methods used for the characterisation and comparison were micro-structural evaluation, hardness tests, charpy impact testing, tensile testing, and X-ray diffraction.

2 METHODOLOGY

2.1 Material preparation

The DED technique was used to fabricate a 60 mm x 60 mm x 10 mm block and a 110 mm long x 11 mm wide x 70 mm tall wall from pre-alloyed commercial 304 stainless steel powder.

The components were fabricated by melting an injected stream of powder layer-by-layer using an IRE-Polus Group (IPG) fibre laser with a 1073 nm wavelength, 150 mm collimator, 200 µm fibre core, and 300 mm lens. The powder was delivered using a Mark XV powder feeder to the three-way nozzle head (see Figure 1) mounted on a KUKA robot. The molten clads were protected from oxidation by injecting argon gas as a shield at a fixed flow rate of 15 litres a minute. The power was set at 1kW, with a robot translational speed of 25mm/s, a spot size of 2mm, and a 12mm stand-off distance. The powder flow rate was calibrated to 4.4g/min.

2.2 Material characterisation

The XRD phase analysis was carried out using a Philips X’Pert modular powder diffractometer equipped with a Co-Kα X-ray source (λ=1.78901A), and a graphite mono-chromator. Bragg-Brentano geometry was used with the following parameters: 40 kV, 40mA, a scan step size of 0.05 [°2θ], and a four-second exposure for each data point. The as-built AM sample was compared with the wrought material.

The charpy test was carried out on three specimens cut from the 60mm x 60mm x 10mm block to the standard sized V-notched bars of a 10mm x 10mm x 55mm bent bar with a 2mm-deep 45° notch according to standard ASTM E23 (Figure 1). The tensile specimens were extracted perpendicular to the build direction (x-axis) along their length. Three specimens were cut in accordance with ASTM E8, with a gauge length of 20 mm, a gauge width of 6mm, and a thickness of 3mm (Figure 1). Uniaxial tension tests were done using the Iston 4202 10 kN load cell, at a strain rate of 1.2 x 10⁻³/s.

The AM-produced samples underwent micro-structural evaluation and hardness testing to compare them with the wrought material’s properties. The micro-structural samples were polished using 0.05 µm colloidal silica and were electrolytically etched using 20 wt% NaOH in deionized water at 5V for 4 to 10 seconds. Hardness tests were done using an Innovatest Nexus 8000XL-Microvickers hardness tester with a 0.3kg load and a holding time of 10s. Five indenters were made in the bulk of each sample and averaged out.

The wrought material for comparison - a 304 stainless steel rod - was sectioned for micro-structural analysis, hardness testing, and XRD analysis, as described above, for comparison with the AM-produced 304 stainless
steel. For the mechanical test specimens, the comparisons with the wrought material were made using comparisons in the literature.

Figure 1: Charpy test specimens and specifications for samples extracted from the DED blocks

Results and Discussion

2.3 Micro-structure and XRD analysis

Figure 2: Micro-structure of the as-built AM sample: (a) the bulk of the sample; (b) at the substrate interface

The micro-structure illustrated in Figure 2 represents the results of the as-built AM produced samples. There is evidence of the columnar-type micro-structure that is inherent in the AM process as a result of the repeated rapid heating and cooling cycles. Columnar grains are generally regarded as undesirable, as their presence can imply solidification defects and mechanical property anisotropy; however, the thermal conditions experienced during additive manufacturing make the development of columnar grains characteristic in standard AM processes, and are unavoidable [12-13].
Towards the centre of the build (Figure 2a), the dendrites have a clear growth direction, showing columnar grains, and the growth direction along the temperature gradient becomes obvious. With an increasing number of layers, the rate of cooling decreases and the direction of heat dissipation becomes apparent along the build direction, and the columnar grains appear parallel to each other. The substrate consists of fine dendrites (Figure 2b) owing to the high cooling rate with the interface of the initial bead or built layers, as described by Ji et al. [14].

Figure 3 shows the micro-structure of the wrought sample, revealing finer uniformly distributed equiaxed grains, which is typical for traditional austenitic stainless steel.

The XRD pattern of the as-built AM sample and of the wrought sample shown in Figure 4 confirm the diffraction planes for FCC-austenite and BCC-ferrite for the AM sample ($\gamma$ and $\alpha$ respectively). Wrought stainless steel, when annealed, can be expected to contain a low proportion of the ferrite phase. According to Astafurov et al. [15], it is common for DED-produced stainless steel samples to have an approximately 2 vol.% of the ferrite phase present, as shown in Figure 4b. The authors reported that the volume content of ferrite depends on the cooling rate and the Cr-eq/Ni-eq ratio value (chromium and nickel equivalent values). The cooling rate gradually decreases during sample growth, and an increase in ferrite content can be associated with the depletion of nickel in the melt and an increase in the Cr-eq/Ni-eq ratio.

2.4 Mechanical tests

Table 1 summarises the charpy impact tests completed at room temperature. According to Karnati et al. [4], as-built AM-produced samples that are built in the same direction as the samples in this study showed toughness values that exceeded 150J. However, when the samples were machined, the toughness values
ranged from approximately 80J to 130J, which corresponded with the values obtained in Table II. The discrepancy between the as-built and the machined specimens’ toughness values can be attributed to the oversized as-built specimens, as allowances are made for machining. The results in Table II fit well with those produced by Karnati et al. [4], even though their samples were produced by the selective laser melting (SLM) method. The wrought stainless-steel toughness values obtained by Karnati et al. [4] were significantly higher at 190J to 220J, indicating that the AM-produced samples in this study and those in Karnati et al.’s [4] study resulted in a significant reduction in toughness compared with wrought 304 stainless steel.

### Table 1: Mechanical properties of the as-built AM samples

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Charpy impact results taken at 20°C</th>
<th>Tensile properties</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Yield strength (MPa)</td>
</tr>
<tr>
<td>1</td>
<td>109</td>
<td>522</td>
</tr>
<tr>
<td>2</td>
<td>92</td>
<td>419</td>
</tr>
<tr>
<td>3</td>
<td>102</td>
<td>475</td>
</tr>
<tr>
<td>Average</td>
<td>101</td>
<td>472 ± 42</td>
</tr>
<tr>
<td>Forged</td>
<td>193</td>
<td>193</td>
</tr>
</tbody>
</table>

Table 2: Hardness properties of the as-built AM samples

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Average micro-hardness results (HV0.3)</th>
</tr>
</thead>
<tbody>
<tr>
<td>AM specimen</td>
<td>145</td>
</tr>
<tr>
<td>Wrought specimen</td>
<td>193</td>
</tr>
</tbody>
</table>

The tensile properties of the AM samples are also summarised in Table 1, with the yield strength, tensile strength, and elongation summarised (and averaged) to 472 MPa, 581 MPa, and 35.6% respectively. According to Astafurov et al. [15], who summarise the tensile mechanical properties of commercial wrought and AM-produced stainless steel 304, conventionally produced or wrought 304 stainless steel gives a yield of 170MPa, a tensile strength of 485 MPa, and an elongation of 40%. The results in Table 1 compare favourably with the properties of the wrought samples in terms of both strength and elongation (within the standard deviation of the average value calculated in Table I). In AM-produced samples from commercial systems, the yield strength ranges from 250MPa to 520MPa and the tensile strength from 500MPa to 890MPa; elongations range from 30 to 50%. However, these values were from different systems and AM processes, and also underwent various post-manufacture heat treatment cycles. The properties obtained in this study fell within the above-mentioned values.

Table 2 summarises the average micro-hardness values of the AM sample and the wrought material. There is a significant reduction in the micro-hardness of the AM sample when compared with the wrought material’s micro-hardness. These results can be attributed to the finer grain structure of the wrought material (compared in Figure 2 and Figure 3).

### 3 CONCLUSION

A comparative study of the properties of AM-produced and of wrought stainless steel 304 was made. These were micro-structure, hardness, XRD phase analysis, tensile testing, and charpy impact testing. The micro-structure results revealed differences in grain structure, with the AM sample producing elongated columnar grains that are typical of the AM process compared with the more conventional uniform equiaxed structure of the wrought material. This affected the subsequent micro-hardness results: the wrought sample showed a greater hardness because of its finer grain structure. The XRD analysis showed the presence of the ferrite phase in the AM sample, whereas the wrought sample showed no indication that any ferrite was present (fully austenitic within the detection limits of the equipment). This can be attributed to the staggered manufacturing process of DED AM, which can lead to the segregation of certain elements and thus to ferrite stabilisation. The tensile properties achieved in this study for the as-built AM sample exceeded the strength of wrought 304, and fell within the range of the ductility of the wrought stainless steel. It also fell within the range of AM 304 stainless steel samples that are produced by commercial systems [15]. The impact testing results showed that the samples produced in this study were comparable to AM-produced samples from commercial systems. However, there was a significant reduction in the impact toughness compared with that of wrought stainless steel when compared to the literature, which could be attributed to the micro-structural differences [4]. Research work continues, and will include the effects of heat treatment on the properties of the AM-produced samples.
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REFERENCES