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Corrosion Testing of a Heat Treated 316 L Functional Part Produced by Selective Laser Melting

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Abstract

Selective Laser Melting (SLM) shows a big potential among metal additive manufacturing (AM) technologies. However, the large thermal gradients and the local melting and solidification processes of SLM result in the presence of a significant amount of residual stresses in the as built parts. These internal stresses will not only affect mechanical properties, but also increase the risk of Stress Corrosion Cracking (SCC). A twister used in an air extraction pump of a condenser to create a swirl in the water, was chosen as a candidate component to be produced by SLM in 316 L stainless steel. Since the main expected damage mechanism of this component in service is corrosion, corrosion tests were carried out on an as-built twister as well as on heat treated components. It was shown that a low temperature heat treatment at 450˚C had only a limited effect on the residual stress reduction and concomitant corrosion properties, while the internal stresses were significantly reduced when a high temperature heat treatment at 950˚C was applied. Furthermore, a specific stress corrosion sensitivity test proved to be a useful tool to evaluate the internal stress distribution in a specific component.

Keywords

316 L, Selective Laser Melting, Stress Corrosion Cracking, Residual Stresses, Heat Treatments

1. Introduction

Selective Laser Melting (SLM) is an Additive Manufacturing (AM) process which locally melts a metallic powder bed using a highly focussed laser beam. Complex functional metallic parts with competitive mechanical properties can be built using a layer by layer manner. The high flexibility in design, low material waste and fast production of near-net-shape parts are the main advantages compared to conventional processing routes.

The SLM process has been widely used for the production of 316 L parts and the optimization of its laser scan parameters has been widely reported [1] [2]. Referring to the microstructure after SLM, a cellular-dendritic microstructure is observed at the micrometer level and elongated grains across several layers at the macro level [3]. The mechanical properties of the SLM processed parts show an increased yield strength compared to wrought 316 L [4] [5]. The effect of heat treatments has been reported by Riemer *et al* [6] and Montero Sistiaga *et al* [7]. Both works show that stress relieving treatments show no significant change in grain size and mechanical properties. Hot Isostatic Pressing (HIP) and annealing treatments show a decrease in yield strength while maintaining the ultimate tensile strength due to complete dissolution of cellular dendrites and maintained grain size [3] [7].

316 L stainless steel is characterized by a high corrosion resistance thanks to the combination of chromium, nickel and molybdenum [8]. However, in SLM components high thermal gradients are obtained due to local melting and a fast solidification process, which can result in residual stresses in the as built condition [9] [10]. These internal stresses will not only affect mechanical properties, but also increase the risk of Stress Corrosion Cracking (SCC). Hence, heat treatments to relieve these stresses are normally applied after the SLM process.

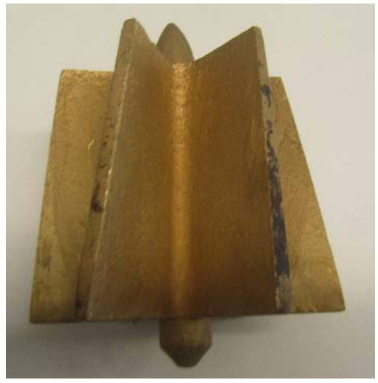
A so-called twister component, used in an air extraction pump of a combined cycle gas turbine plant condenser to create a swirl in the water (**Figure 1**), proved to be a good candidate to be produced by SLM. Producing a spare twister by conventional casting would require making a casting die and would prove to be slower and more expensive than creating the part by AM. Also, since the original ex-service part shows a lot of erosion and impact damage on the surface, surface roughness is not an issue for this part. In addition, the consequences of a component failure are only minor, since a second extractor is present to take over.

Since the twister is a static part and the extraction pump operates at room temperature, the main in-service damage mechanism for this component is corrosion and possibly SCC. Since the risk of SCC can be reduced by reducing the internal stresses, different heat treatments were carried out on the components manufactured by SLM. Subsequently, immersion corrosion tests and SCC tests were carried out on as-built and heat treated components. This paper describes the results of these tests.

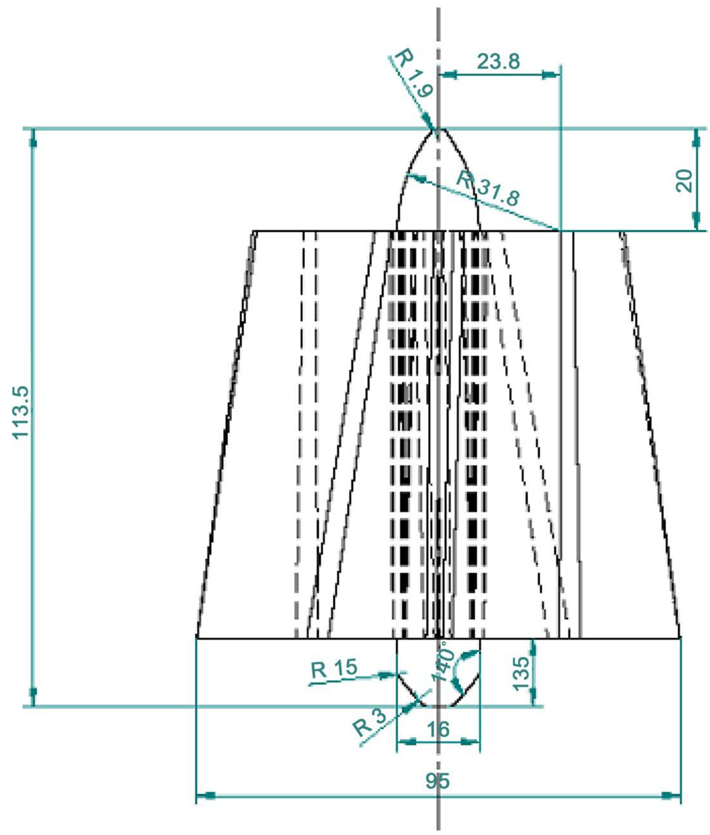
2. Experimental Set-Up

The material used in this study was 316 L stainless steel provided by SLM Solutions Group AG with powder particle sizes ranging from 10 to 45 μm and spherical in shape. The 316 L powder composition is shown in **Table 1** as defined in ASTM B243.

(a)



(b)



(c)

Figure 1. The original twister component which was selected for AM. (a) Twister inside the air extraction pump; (b) ex-service twister; (c) schematic drawing of the twister component.

Table 1. Chemical composition in weight % of 316 L powder provided by SLM solutions AG.

wt%	Fe	Cr	Ni	Mo	Mn	Si	P	S	C
316 L powder	Bal.	16.8	10.4	2.1	1.11	0.56	0.03	0.011	0.01

A SLM 500 machine from SLM Solutions Group AG, Germany, was used to build 4 twisters and two cylinders with roughly the same dimensions as the axis of the twister (11.5 cm long with a diameter of 1.6 cm). The SLM500 machine provides a build envelope of 500 × 280 × 320 mm^3 and is equipped with four 400 W fibre lasers.

Twister 1, cylinder 1 and twister 4 were kept in the as-built condition. Twister 2 and cylinder 2 were submitted to a high temperature heat treatment (HT-HT, 2 h at 950˚C, air cooling) and twister 3 to a low temperature heat treatment (LT-HT, 2 h at 450˚C, air cooling).

The twisters were slightly sandblasted before the corrosion tests.

For the immersion corrosion tests, samples were immersed for 33 days in demineralised water with 200 ppm chlorides and 450 ppm sulphates, which represents the most severe condition the actual component could experience in reality.

To further evaluate the internal stresses in the different components ASTM G36 [1] SCC tests were carried out in boiling MgCl2 solution at 155˚C ± 1˚C. A first visual inspection was carried out after 3 h, a second one after 5.5 h, followed by daily visual inspections. The test was stopped when cracks were found or after a maximum of 77.5 h.

Fluorescent penetrant testing was carried out to better identify the cracks.

Both cylinders were cut in half in the longitudinal direction. One half was used to study the microstructure after HT-HT and compare it with the as-built microstructure. The microstructure and crack propagation of the twisters was also analysed. All the metallographic samples were polished and etched using oxalic acid and examined using an Axiocam Leica optical microscope. Micro Vickers hardness tests were performed using a Future Tech FV-700 hardness tester with an indentation load of 0.5 kg during 15 s.

The geometry of the twisters has been captured before and after heat treatment using a HANDI SCAN 700 (with a resolution of 0.2 mm). The comparison of geometries did not reveal major deformations after heat treatment (**Figure 2**).

3. Results and Discussion

3.1. Microstructural Investigation

The microstructures of cross-sectioned samples are shown in **Figure 3** for Twister 1, Twister 2 and Twister 3. For Twister 1 and Twister 3 no significant difference in the microstructure can be observed (**Figure 3(a)**, **Figure 3(c)**). The melt pool's boundaries can be clearly seen as depicted in yellow and the sub-grain cellular dendritic microstructure is visible. The cell size is around 1 µm in size and the grains grow across several layers, with an average of 100 µm in length and parallel to the building direction. This type of microstructure has also been observed in earlier works [3] [7] [11]. Twister 2, which was heat treated at high temperature (950˚C) shows no cellular dendritic structure and also no melt pool boundaries.

Micro hardness tests were performed in order to analyse the effect of the ap-

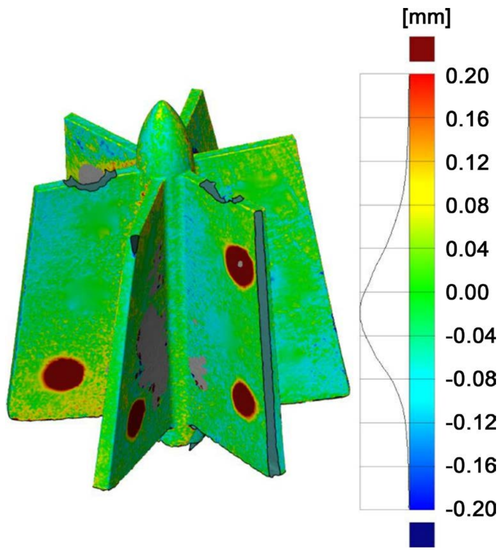


Figure 2. Surface comparison of 3D scan before and after heat treatment.

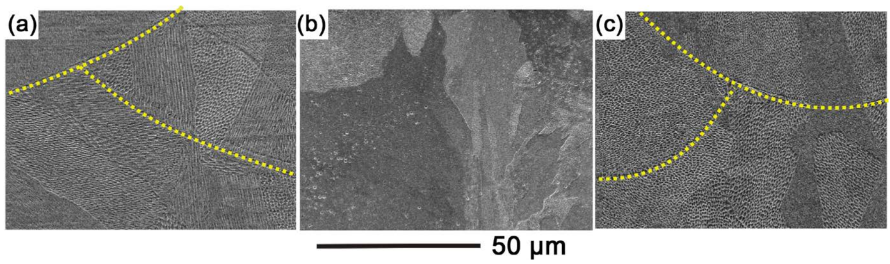


Figure 3. Secondary electron micrograph parallel to the building direction of SLM produced 316 L. (a) Twister 1 in as built condition, (b) HT-HT twister 2 and (c) LT-HT twister 3.

Table 2. Micro vickers hardness of SLM 316 L in as built condition and after low temperature (LT-HT) and high temperature (HT-HT) heat treatment.

Vickers Hardness (HV0.5)	Twister 1 As built	Twister 2 HT-HT	Twister 3 LT-HT
	240 ± 6	195 ± 8	239 ± 5

plied heat treatments on the hardness. The results are depicted in **Table 2**. For the as built condition 240 ± 6 HV is obtained which is comparable to the values seen in literature [3] [4] [7]. The LT-HT has the same hardness as the as built condition, thanks to the similar microstructure. These values are in accordance to the 235 HV found in other works [3] [4]. On the other hand, for HT-HT a decrease in hardness is observed. This can be attributed to the dissolution of the cellular dendritic substructure and hence the loss of strengthening sites compared to the as built and LT-HT condition, as observed in **Figure 3**.

3.2. Immersion Corrosion Tests and Field Test

Twister 1 and 1 half of cylinder 1, both in as-built condition, were subjected to the immersion test for 33 days. No cracks were observed on any of the components. Only a mild discoloration on the rougher edges of twister 1 (former location of the support structure) was observed (**Figure 4**). Since the as-built components, containing the largest amount of internal stresses, did not show any cracking in the representative immersion test, it was not necessary to subject the heat treated components to an immersion corrosion test. It was also decided that the as-built twister 4 could be used for an operational test. Twister 4 was put into service in October 2016 and removed after 2000h in operation. No cracking was observed visually (Figure 5). This confirms the validity of the immersion corrosion test and also the effective performance of the SLM processed twister in service.

3.3. SCC Tests

Twister 1, half of Cylinder 1, twister 2 and twister 3 were subjected to the ASTMG36SCC test. The results are summarised in Table 3.

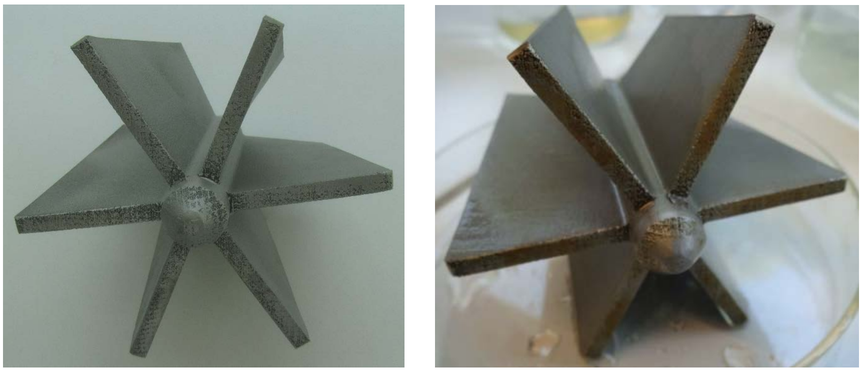


Figure 4. Twister 1 before (left) and after (right) the immersion corrosion test.

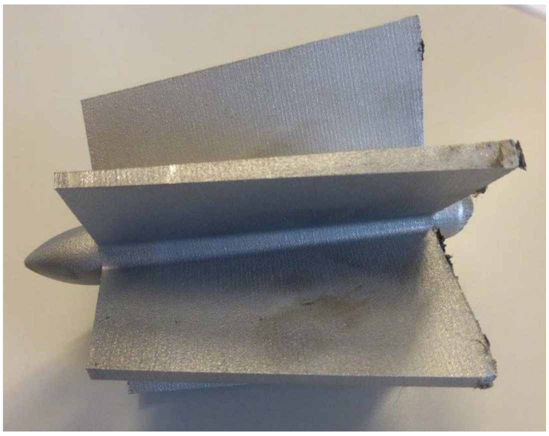


Figure 5. Twister 4 after service operation.

Table 3. Results of the ASTMG36 SCC tests on the twisters.

Component	Heat treatment	Test duration (h)	Visual inspection result
Twister 1	None	3	Multiple cracks
Cylinder 1	None	3	Multiple cracks
Twister 2	2 h @ 950˚C	77.5	No cracks
Twister 3	2 h @ 450˚C	5.5	Multiple cracks (but less than for Twister 1)

During the first visual inspection after 3 h only the as-built twister 1 and cylinder 1 showed multiple cracks. Cracks oriented both parallel and perpendicular to the build direction were observed on twister 1 (Figure 6(a)). The cracks parallel to the build direction were situated at the top and bottom edges of the blades, while the cracks perpendicular to the build direction and hence parallel to the build layers were not only present at the edges of the blades, but also on the blades' surfaces and on the tip of the twister. For cylinder 1, only cracks perpendicular to the build direction were observed.

During the second visual inspection after 5.5 h, multiple cracks were observed on the LT-HT twister 3, although the total amount was still less than those observed on twister 1 after 3 h. The orientation of the cracks was again both parallel and perpendicular to the build direction, as described for twister 1 (Figure 6(b)).

The HT-HT twister 2 was kept immersed in the ASTMG36 test for an additional 72 h (3 days), with daily visual inspections. It did not show any sign of cracking (Figure 6(c)).

The results of these SCC tests allow to have a rough idea of the magnitude of the internal stresses inside the components, based on the graph in the ASTMG36 standard (Figure 7). The internal stresses in the as built condition are probably higher than 250 MPa. After the low temperature heat treatment they are slightly decreased and will be somewhere in between 150 and 350 MPa. After the high temperature heat treatment, internal stresses have dropped significantly and will be below 110 MPa, significantly reducing the SCC risk.

These tests highlights the positive effect of heat treating SLM processed 316L parts at a temperature of 950˚C compared to the as-built and low temperature heat treatment condition as it results in a significant reduction of the internal stresses. These results confirm the results obtained in a previous work [7] where heat treatment at 950˚C resulted in high energy absorption values and higher tensile values than the minimum required from the EM10216-5; 2013 standard. On the other hand, in order to fully understand the effect of heat treatments on SCC performance, more heat treatment temperatures should be tested.

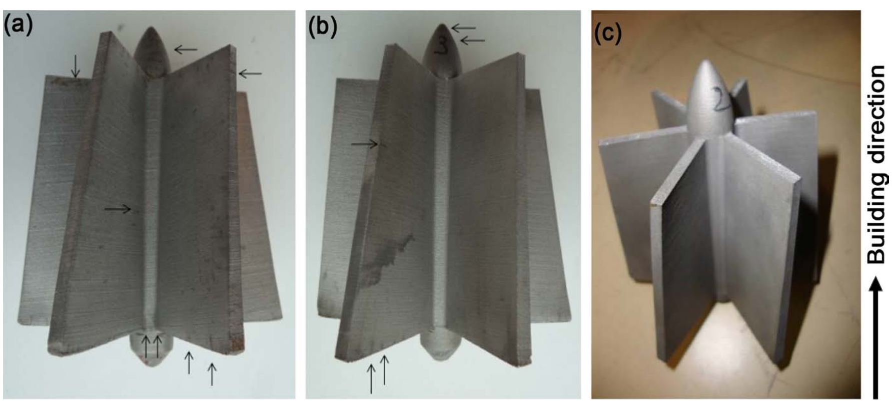


Figure 6. ASTMG36 SCC test. (a) As built twister 1 after 3 h showing multiple cracks. (b) LT-HT twister 3 after showing multiple cracks. (c) HT-HT twister 2 after 77.5 h showing no cracks.

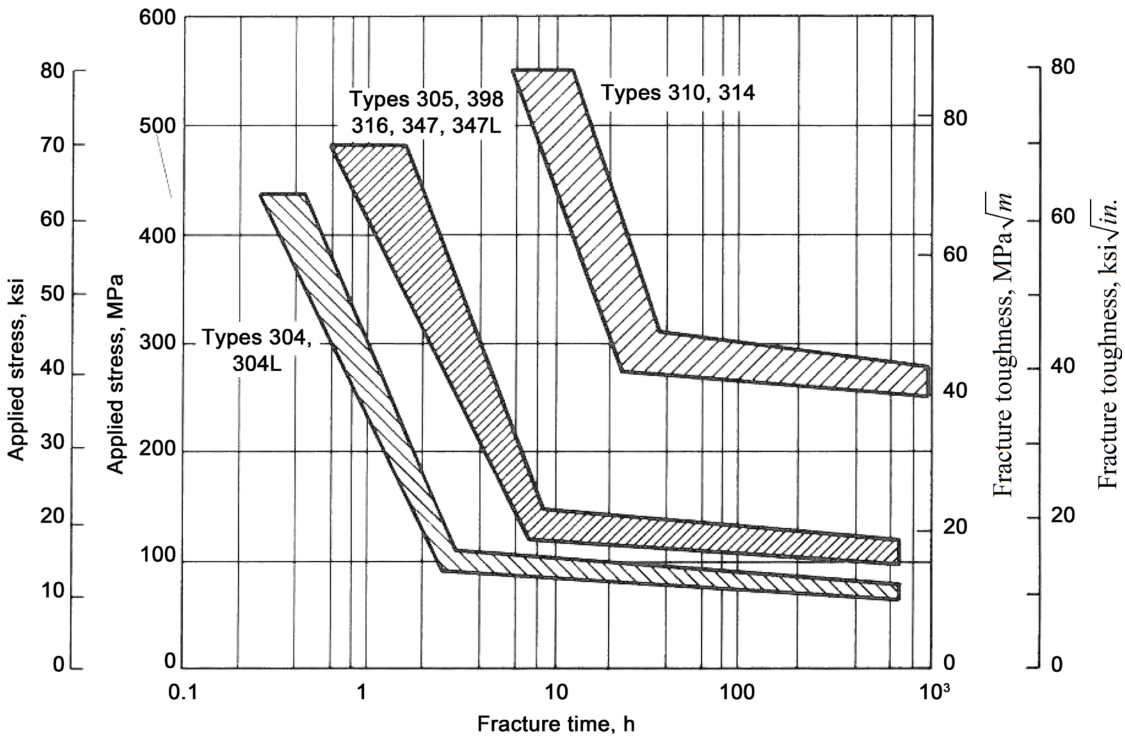


Figure 7. Effect of applied stress on the times to failure of various alloys tested in a magnesium chloride solution boiling at 154˚C [12].

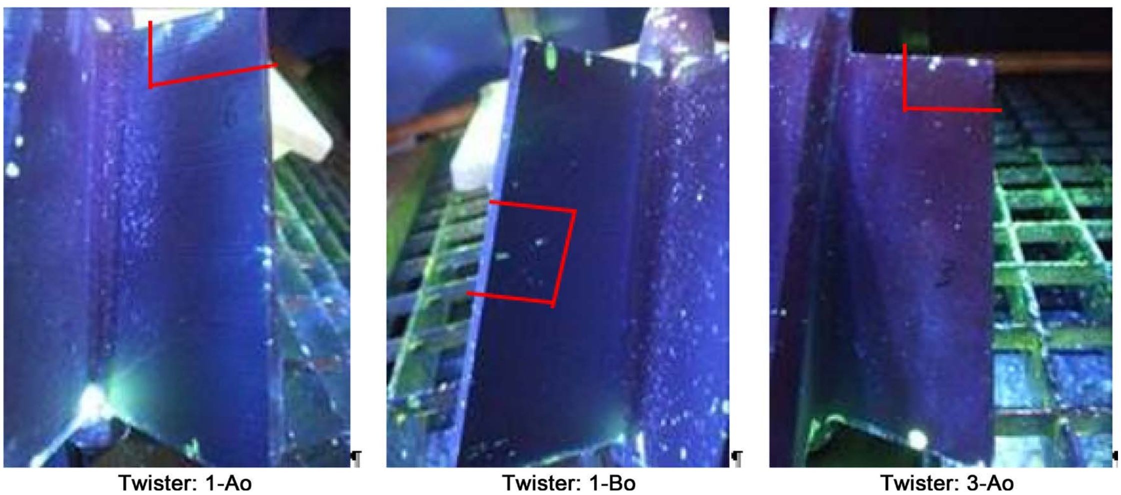


Figure 8. Fluorescent Penetrant Inspection on twister 1 and twister 3 to highlight the cracks. Red lines on the pictures indicate where the component was cut in order to examine some of the cracks in more detail.

3.4. Characterization of the Cracks

Fluorescent penetrant testing was used to highlight the cracks on twister 1 and twister 3 and some typical cracks in both directions were selected for more detailed microscopic inspection (Figure 8).

SCC is usually characterized by extensive branching and crack growth perpendicular to the stress direction. All observed cracks on the 3 samples had the typical branched aspect of stress corrosion cracks (Figure 9). Most of the cracks initiate at the edge of the blades where the stresses are the highest. For the cracks at the edge of the blades, parallel to the build direction (samples 1-A and 3-A), apart from the cracks that were visible with the naked eye, some smaller cracks were present (Figure 9(a)). The crack lengths were measured for all sections mentioned in Figure 8 and are depicted in Table 4. The largest observed crack on sample 1-A was about 3.75 mm and on sample 3-A about 2.5 mm. For the cracks perpendicular to the build direction, several cracks starting from the blade surface and not necessarily from the blade edge, could be observed. The largest one on sample 1-B was about 2.5 mm.

The crack density found for all cases corresponds with the results obtained from the fluorescent penetrant inspection. The samples from Twister 1 have a higher crack density than the sample from Twister 3. In addition, it has to be kept in mind that Twister 3 was subjected to the SCC test for 5.5 h and Twister 1 only for 3 h.

In Figure 9, the crack morphology of sample 1-A and sample 3-A can be observed. The cracks initiate at the surface of the part and propagate perpendicular to the surface and in the case of Figures 9(a)-(c) parallel to the building direction. In Figure 9(c) a better view of the crack propagation is shown. The crack

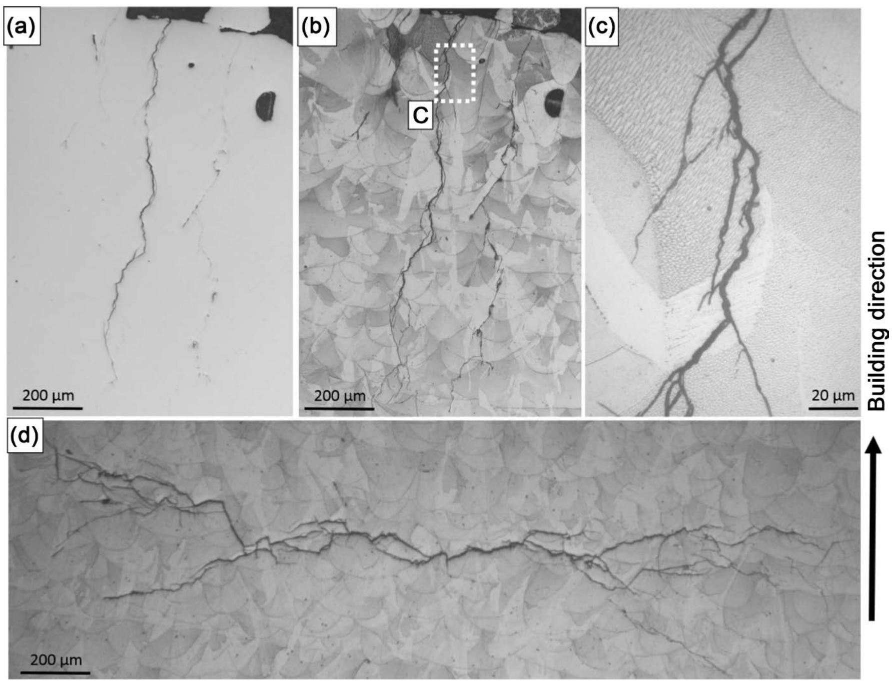


Figure 9. Light Optical Micrographs of the samples showing the typical SCC aspect of the observed cracks (a) sample 3-A without etching; (b) sample 3-A etched and (c) zoomed area of the intergranular/transgranular crack. (d) sample 1-A etched, crack perpendicular to building direction.

Table 4. Cracks lengths of the twisters after performing SCC test measured on the polished cross sections.

	Sample 1-A	Sample 1-B	Sample 3-A
Maximum crack length (mm)	3.75	2.5	2.5
Crack density (mm/mm^2)	0.040	0.044	0.033

path shows a mixed intergranular and transgranular crack propagation with a predominant transgranular crack pattern. In Figure 9(d), a crack perpendicular to the building direction of sample 1-A is shown.

4. Conclusions

Within this paper, the corrosion behaviour of SLM316L stainless steel was investigated. The influence of different heat treatments on the microstructural evolution and corrosion behaviour of 316L stainless steel was explored. Based on the results of the corrosion tests and the microstructural characterization of the SLM316L stainless steel, both after SLM, after heat treatment and after corrosion testing, the following conclusions could be drawn:

- As built and low temperature heat treated SLM316L components have a cellular dendritic microstructure and micro hardness values of 240 HV. A heat treatment of 950˚C converts this cellular dendritic structure into a coarser grained microstructure, significantly lowering the hardness.
- As-built SLM316L components contain a significant amount of internal stresses, which makes them sensitive to SCC. A low temperature heat treatment at 450˚C slightly lowers the internal stresses, but the SCC sensitivity remains high. A heat treatment at 950˚C significantly reduces the internal stresses and hence drastically lowers the SCC sensitivity.
- An ASTMG36 corrosion test in boiling magnesium chloride can be used to check which locations on an as-built SLM component are the most prone to SCC.
- For the twister component, the blade edges and the blade surface area are the most prone to stress corrosion cracking.

The SLM technology allows producing tailor-made complex components in a competitive way regarding lead time and cost. Nevertheless, these AM parts currently still need to be tested and qualified for ensuring fitness for service. The current paper shows that, provided a correct heat treatment is applied to SLM316L parts, their corrosion properties can be significantly enhanced, rendering them competitive with more traditionally manufactured 316 L components.

Although the ASTMG36 corrosion test indirectly allowed getting a qualitative idea of the internal stresses in the component it would be useful to have a more direct way to measure internal stresses. Deep-hole drilling and X-ray Diffraction are possible techniques for that, which are being investigated.

More detailed analyses of the microstructure before and after the 950˚C heat treatment at different locations, could be useful in helping to refine and optimise the heat treatment procedure.

Acknowledgements

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