Microstructure examination of additive manufactured 316L steel

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Abstract. The additive manufacturing (AM) is a new field in research. In our research, the samples were made by direct metal laser sintering (DMLS). During the production of 316L material, the piece was formed layer by layer, so the existing layer was re-heated treated when the new layer was formed. Due to the multiple heat treatment, the post-heat treatment of the product is necessary. Microstructure changes were investigated after two different heat treatments. The first was at 450 °C for 4.5 hours, and the second was at 1100 °C for one hour, followed by annealing in each case. The heat-treated sample at 450 °C showed no significant difference. In contrast, the sample heat-treated at 1100 °C the particles were better separated, and segregation in grain boundary was observed, which showed high phosphorus concentration.

1. Introduction

There are many ways to design a product. Which option was chosen depends among other things, for example, the available technology, the required quality, and the cost of production. Recently, additive manufacturing (AM) is gaining ground, and more and more technology is available. AM technology can also provide a geometry that is not or only very difficult to produce with traditional manufacturing technologies [1,2]. With AM techniques are easy to produce complex shaped functional metallic components such as tubes, gear wheels, or toolmaking, e.g. blow moulding, extrusion, die casting and sheet metal forming.

1.1. AM technology

During the research, direct metal laser sintering (DMLS) was used to produce the test specimens. The DMLS does not melt the powder of the base material but heats it to a temperature close to the melting point, which results in a chemical bond between the powder particles, i.e. the sintering process [3]. With this technology cannot use pure metal powder, only a mixture of multiple powders [4], but DMLS is still the most common additive technology.

The equipment consists of 4 main components: a metal powder feeder, a continuous carbon dioxide (CO₂) laser, a powder spreader (Figure 1), and a computer system that controls and monitors the process [5]. For the manufacturing of the product, a 3D model is required. It can be converted to an STL file format for the AM device [6].
The STL file has a layer thickness of approximately 50 µm, which depends on the technology [5,7]. During layer by layer formation [8], a new layer reheats the existing layer(s). As a result, a re-heat treatment was made layer by layer, which can result in a characteristic microstructure. Special care must be taken to the microstructure of the additively manufactured piece because it can influence the mechanical properties. If the application area only allows a slight change in properties, it is recommended a post-heat treatment after the product is finished [7].

1.2. Heat treatment
The purpose of heat treatment is to achieve structural changes in the products or to eliminate the residual stress due to technology [9].

According to Katherine et al. [10], controlling residual stresses and microstructure was investigated for 3D printed 316L material. Two different heat treatments were carried out, one at 490 °C for 5 hours, and the second product was heat-treated at 1200 °C for 1 hour, followed by annealing in each case. The heat treatment has been prepared according to Chen et al. [11] whose stated that mechanical and microstructural properties were improved in a positive direction when the heat treatment was made at 490 °C. The aim was a regenerative, restorative heat treatment at 1200 °C, which was sharpened to the sigma phase, but neither recrystallisation nor grain growth was observed. During mechanical tests, heat treatment at 490 °C resulted in a reduction in the hardness of the specimens, while at 1200 °C, the hardness of the pieces increased. After the tensile test, the heat treatment at 490 °C gave substantially different tensile curves similar to the reference (untreated) sample, which broke after nearly the same elongation. After the heat treatment at 1200 °C, the value of tensile strength decreased, but the elongation increased [10].

The aim of this research is to compare the effects of heat treatment on two different test specimens relative to the initial untreated state.

2. Materials and methods
The specimens were made from 316L stainless steel with EOS M100 equipment. The composition, mechanical and physical properties of 316L material are presented in Tables 1-2 [12]. The first test specimen was heat-treated at 450 °C for 4.5 hours (later marking: HT1 – heat treated 1) and annealed. The second test specimen was heat-treated at 1100 °C for 1 hour (later marking: HT2 – heat treated 2) and annealed. The heat treatment was carried out in the air for both samples. After the heat treatment, the samples were mechanically ground with 60 to 2500-grit SiC and polished with 1 µm diamond suspension. After the polishing, the samples were etched in Kalling’s No. 2 reagent. The cleaning fluid was acetone, and it was dried with hot air. The images were taken with Olympus SZX16 stereomicroscope, Olympus PMG 3 optical microscopic, and Zeiss EVOma 10 scanning electron microscope (SEM). The composition analyses were performed with energy-dispersive spectrometry (EDS). The hardness was measured with Vickers microhardness tester (Buehler IndentaMet 110/220).

Table 1. The 316L material chemical composition in wt% [12]

| C   | Mn | Si | P   | S   | Cr | Mo | Ni | N  | Fe    |
|-----|----|----|-----|-----|----|----|----|----|-------|
| 0.03| 2.0| 0.75| 0.045| max | 0.03| 16-18| 2-3| 10-14| max 0.1 | balance |
### Table 2. 316L material mechanical properties [12]

|                        | Ultimate Tensile Strength (MPa) | Yield Strength 0.2% Offset (MPa) | Modulus of Elasticity in Tension (GPa) | Hardness (HV1) |
|------------------------|---------------------------------|----------------------------------|---------------------------------------|----------------|
|                        | min 485                         | min 170                          | 200                                   | 223            |

3. Results and discussion

The microstructure of 3D products was different from traditionally manufactured products. Figure 2 shows the microstructure of the 316L specimen produced by additive and traditional manufacturing technology. In Figure 2 (a) can be observed that the grain boundary cannot be distinguishable clearly. Figure 2 (b) shows that the grain boundary can be distinguishable, and the twins plane of the austenitic structure can be seen.

![Figure 2](image)

As previously mentioned, the product was re-heat-treated layer-by-layer, which can produce unpredictable properties. The finished product was polished before heat treatment to provide a basis for comparison with the heat-treated samples.

3.1. The original state

Stereomicroscope images were made from the unpolished surface of the samples, which are seen in Figure 3. At lower magnification, the surface was like drum-pressed, and at higher magnification, the surface was more like caterpillar seam.

![Figure 3](image)

Before the heat treatment, the surface of the product was prepared and etched with Kalling’s No. 2 reagent. Figure 4. shows the microstructures. At higher magnification, the forming lines were visible, which was also comparable to a caterpillar seam. Due to the technology, it resulted inhomogeneous appearance of austenitic grains with precipitations and/or inclusions.
3.2. Heat-treated sample at 450 °C temperature – HT1
The microstructure of HT1 is seen in Figure 5. After etching for less time in lower magnification, the surface showed caterpillar structure and the reflecting of the laser beam movement [13], but after 2 minutes etching [14], it can be seen in higher magnification that there were enrichments, precipitation or inclusions along the grain boundary and no twin plane was observed.

3.3. The heat-treated sample at 1100 °C temperature – HT2
The microstructure images of HT2 are seen in Figure 6. In this case, a much more spectacular change has occurred, since the grain can be distinguishable, and they were more closely resembled austenite microstructure in lower magnification. In higher magnification, Figure 6 shows the twin plane of austenite and a large number of tiny inclusions.

3.4. Hardness test
Table 3 shows the results of the hardness tests. The hardness was measured with a load of 1 kilogram for 11 seconds. As we can see, the hardness of the HT2 was higher, but in these two procedures, the hardness was higher than the base material according to the metals catalogue [12,15].
### Table 3. The hardness of the heat-treated products

| Number of measurements | Hardness of HT1 (HV1) | Hardness of HT2 (HV1) |
|------------------------|-----------------------|-----------------------|
| 1                      | 161                   | 220                   |
| 2                      | 154                   | 221                   |
| 3                      | 154                   | 217                   |
| 4                      | 153                   | 222                   |
| 5                      | 155                   | 210                   |
| **Average**            | **155**               | **218**               |

#### 3.5. SEM and EDS results

After the stereo and optical microscope measurements, the samples were examined with SEM. The main aim was to specify precipitations origin. The surface of the samples can be seen in Figure 7. The twin planes of HT2 can be observed, which were not visible at the HT1.

![Image a) and b)](image)

**Figure 7.** The microstructure images of the 3D samples by SEM after a) at 450 °C b) at 1100 °C heat treatment

The identification of the precipitations can be seen in Figure 8. In the two images show the microstructure and the measured area was marked with the red circle. The EDS examination results of Figure 8 (a) was: Si: 0.29%; P: 0%; Mo: 2.52%; Cr: 20.21%; Mn: 1.36%; Fe: 62.78%; Ni: 12.84% in wt%. The EDS examination result of Figure 8 (b) was: Si: 0.39%; P: 0.22%; Mo: 3.01%; Cr: 19.98%; Mn: 1.35%; Fe: 62.13%; Ni: 12.91% in wt%. The EDS results did not show differences in the composition of the base material.

![Image a) and b)](image)

**Figure 8.** The EDS analysis results and the measured area of the samples by SEM after a) at 450 °C b) at 1100 °C heat treatment
According to the EDS results, it can be stated that these formations at grain boundary are porosity, not precipitation. By the HT2, the EDS showed five times more phosphorus in the material like the allowed value. Similar values were measured for several different measurement points as well.

4. Conclusion
It can be stated that the microstructure of the 3D product was showed austenitic characteristic. Homogenisation was observed after the post-heat treatment, which resulted in a more ordered and larger grain size. The austenitic structure was observed by the heat-treated sample at 1100 °C, because of the twin plane. After heat treatment, porosity appeared at 450 °C along the grain boundary, and it can be visible at 1100 °C both in the grain and also their boundary. The hardness increased compared to the base material in both cases, but the hardness of HT2 was more significant approximately 60 HV. One possible reason for the difference in hardness value is the grain coarsening. The higher temperature of the heat treatment has a greater chance to coarse of the grains, which remains coarse-grained even after cooling. This can even affect hardness.

The HT2 indicated the microstructure similar to that traditionally manufactured microstructure. The high phosphorus concentration was measured and was not favourable as it can negatively affect the mechanical properties. Phosphorus segregation was formed due to the heat treatment, but further investigations are needed to determine this phenomenon. The effect of this transformation on mechanical properties requires further investigations. More examinations are required to minimise the number of porosity in the procedure and/or in heat treatment as well.

Acknowledgement
Our research was supported by the ÚNKP-19-3 New National Excellence Program of the Ministry for Innovation and Technology. The authors are grateful for Budapest University of Technology and Economics to providing our research.

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