Microstructure and mechanical properties of Selective Laser Melted 18Ni-300 steel.

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Abstract

Selective Laser Melting (SLM) is an Additive Manufacturing process in which a part is built in a layer by layer manner. A laser source selectively scans the powder bed according to the CAD data of the part to be produced. The high intensity laser beam makes it possible to completely melt the metal powder particles to obtain almost fully dense parts. In this work, the influence of process parameters in SLM (e.g. scan speed and layer thickness) and various age hardening treatments on the microstructure and mechanical properties of 18Ni-300 steel is investigated. It is shown that almost fully dense parts with mechanical properties comparable to those of conventionally produced maraging steel 300 can be produced by SLM.

Keywords: Selective Laser Melting; Maraging steel 300; Additive Manufacturing; Microstructure

1. Introduction

Selective laser melting, SLM, is a powder-based additive manufacturing process that allows the production of functional three-dimensional parts directly from a CAD-model. During the process, successive layers of metal powder particles are molten and consolidated on top of each other by the energy of a high intensity laser beam. Consequently, almost fully dense parts without need for any post-processing other than surface finishing are produced. Customized medical parts, tooling inserts with conformal cooling channels and functional components with high geometrical complexity are good examples to reveal the scope of the application areas of this process [1, 2].
Laser processing of materials is generally accompanied with high cooling rates due to the short interaction time and high thermal gradients. The high cooling rates during SLM may result in the formation of non-equilibrium phases, quasi-crystalline phases and new crystal phases with extended composition ranges [2]. Finer structures may be observed in the microstructure at sufficiently high cooling rates compared to the conventional manufacturing methods [3]. Moreover, during the SLM process, gas bubbles and oxide inclusions can become entrapped in the material during solidification due to various causes such as decrease in the solubility of the dissolved elements in the melt pool during cooling and solidification, chemical reaction or trapped gas. Therefore, the mechanical and material properties obtained after SLM may be different than the properties of materials produced by conventional production techniques.

In this study, the influence of the SLM-process parameters on the density, the microstructure and the mechanical properties was investigated. Moreover, the effect of temperature and duration of the hardening (aging) heat treatment on the obtained hardness was studied.

2. Background

Maraging steels are well known for combining good material properties like high strength, high toughness, good weldability and dimensional stability during aging heat treatment. Mainly maraging steels are used for two application areas: the aircraft and aerospace industry in which superior mechanical properties and weldability of maraging steels are the most important features, and secondly in tooling applications which require superior machinability [4]. The nominal composition of maraging steel, grade 300, is given in Table 1.

Table 1. Chemical composition of 18Ni-300 steel [5,6]

<table>
<thead>
<tr>
<th>Alloying element</th>
<th>Fe</th>
<th>Ni</th>
<th>Co</th>
<th>Mo</th>
<th>Ti</th>
<th>Al</th>
<th>Cr</th>
<th>C</th>
<th>Mn,Si</th>
<th>P,S</th>
</tr>
</thead>
<tbody>
<tr>
<td>wt %</td>
<td>rest</td>
<td>17-19</td>
<td>8.5-9.5</td>
<td>4.5-5.2</td>
<td>0.6-0.8</td>
<td>0.05-0.15</td>
<td>&lt;0.5</td>
<td>&lt;0.03</td>
<td>&lt;0.1</td>
<td>&lt;0.01</td>
</tr>
</tbody>
</table>

Maraging steels are a special class of high-strength steels that differ from conventional steels in that they are hardened by a metallurgical reaction that does not involve carbon [4]. The relatively soft body centered cubic martensite, that is formed upon cooling, is hardened by the precipitation of intermetallic compounds at temperatures of about 480°C.

3. Experimental procedures

A Concept Laser M3 Linear machine was used to build the specimens. This machine employs a diode-pumped Nd:YAG laser with a wavelength of 1,064 nm and a maximum laser output power of approximately 100 W measured in continuous mode. The laser beam diameter d99% at the powder bed surface is about 180 μm. The powder materials were supplied by Concept Laser (CL 50WS) [5] and LPW (M300-1) [6].
The SLM samples used to perform the heat treatments and mechanical testing were built with a set of process parameters chosen in terms of maximal density: a layer thickness of 30 μm, a scan speed of 150 mm/s and a scan spacing of 112 μm (62% of the spot size). The samples were heat treated in a vertical tube furnace in N₂-atmosphere and cooled down in air.

Both optical and Scanning Electron Microscopy (SEM) techniques were used to evaluate the microstructure and the fracture surfaces of the mechanical test specimens. X-ray diffraction patterns were measured by the Siemens D500 goniometer, whereas Rietveld Refinement analysis was done by X’pert software Plus.

To indentify the mechanical properties, tensile, hardness and Charpy impact tests were conducted. The tensile tests were conducted on an Instron 4505 testing machine. Macro-hardness is measured according to the Rockwell A scale (60kg load), while micro-hardness was measured on a Vickers scale (0.5 kg load). Most hardness results were transformed to the Rockwell C scale, according to [7].

4. Results and discussion

This study mainly consists of three parts. First the influence of the process parameters (scan speed and layer thickness) on the hardness and microstructure is investigated. Secondly, the age hardening heat treatment was optimized for maraging steel 300 parts produced by SLM. Lastly, the mechanical properties of the as-built SLM parts and heat treated parts are determined and compared to each other and to the values obtained for conventional wrought maraging steel.

4.1. Influence of process parameters on hardness and microstructure

The influence of the layer thickness (30–60 μm) and scan speed (120–600 mm/s) on the macro-hardness is shown in Figure 2a. These process parameters both have an important influence on macro-hardness through their influence on the part density. Relative density of the parts is depicted in Figure 2b.

![Figure 2](image.png)

Figure 2: (a) Macro-hardness for samples with different scan speeds and layer thicknesses, within a confidence level of 95% for 8 measurements. (b) Relative density for samples with different scan speeds and layer thicknesses.
The indentations of the micro-hardness measurements are much smaller, making it possible to only indent the consolidated material. As a result, the hardness results are no longer influenced by the porosity, which is shown in Figure 3. The layer thickness and scan speed have almost no significant influence on the micro-hardness. For high scan speeds and/or layer thicknesses there is a small decrease in hardness and increase in scatter due to the difficulties in performing the measurement for very porous samples.

The top and side cross-sections were observed under a SEM as depicted in Figure 4. The micrograph on the left shows bi-directional scan tracks while the one in the center depicts the cellular/dendritic solidification morphology and epitaxial growth of the grains on a section perpendicular to the layer build sequence. In SLM, the cooling rate is very high and rapid solidification prevents formation of a lath martensite. The intercellular spacing is less than 1 μm for the cellular structure and this contributes to the excellent strength and hardness. These statements are also validated by other researchers working on direct metal laser sintering of maraging steel 300 processed on an EOS machine [8]. In Figure 4b, one can observe a large inclusion with a size of about 10-20 μm, visible as a dark spot in the cross-section. The EDX analysis carried out on this inclusion confirmed that this was a titanium and aluminum combined oxide (TiO₂ : Al₂O₃). Also other oxides containing a combination of Ti, Mo, Al and Si in other ratios were present in the sample [8].

In order to perform mechanical testing, porosity must be eliminated as much as possible. Laser re-melting every SLM layer before adding a new layer can contribute to a higher density, up to 99.4%. Re-melting every layer results in an increase of both density and micro-hardness, at the cost of longer production times.
4.2. Optimization of heat treatment

The superior properties of the maraging steels, i.e. good strength and toughness, are achieved by the age hardening of a ductile, low-carbon body-centered cubic (bcc) martensite structure with relatively good strength. Therefore, the aging heat treatment is standard for maraging steels. It is aimed to form a uniform distribution of fine nickel-rich intermetallic precipitates during the aging of the martensite. These precipitates serve to strengthen the martensitic matrix. A detrimental side effect is the reversion reaction of metastable martensite into austenite and ferrite [9]. Fortunately, the kinetics of the precipitation reactions of iron-nickel maraging steels are such that a significant age hardening of about 20 HRC points already occurs before the start of this reversion reaction.

The aging heat treatment for maraging steels can be performed for different durations at various temperatures providing that the temperature is lower than the austenite start temperature. For maraging steel 300, the values recommended by the ASM Handbook are 3 to 8 hours at a temperature between 460 and 510°C [9]. In this study, a duration between 1-8 hours and a maximum temperature between 460°C and 500°C are tested.

The micro-hardness of the samples was measured and plotted in Figure 5. When the maximum aging temperature is 460°C, the hardness shows a linear relationship with aging time. As you keep the part longer at this temperature, the hardness continues to increase without any sign of overaging. However for other temperatures tested in the scope of this study, at prolonged durations, the hardness starts to slightly drop. This is an indication of overaging meaning that the reversion of metastable martensite and coarsening of the intermetallic precipitates takes place. These two phenomena together decrease the hardness as the part is kept at elevated temperatures for a prolonged time.

Optimal heat treatment in terms of hardness and aging time is selected as aging 5 hours at 480°C. After this heat treatment a hardness of 58 HRC is achieved, which means an increase of 18HRC compared to the as-built part using remelting (this is the re-scanning of a recently/just scanned and solidified layer). Higher hardness can be achieved for lower aging temperatures, but the increase in hardness is not significant in terms of the additional aging time.

In order to determine the volumetric percentage of austenite (fcc, face cubic centered) and martensite (bcc, body cubic centered), that are present before and after aging, a XRD measurement was performed. The results are shown in Figure 6. The applied heat treatment causes an increase of the austenite phase. The austenite reversion is inevitable for long aging times, because the martensite is metastable and transforms to the stable austenite. The release of Ni into the Fe matrix which accompanies the transformation from Ni3(Mo,Ti) to the more stable Fe2Mo precipitates promotes the austenite reversion [10].
Table 2. Results of the Rietveld Refinement analysis

<table>
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<tr>
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<th>Austenite γ</th>
<th>Martensite α</th>
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<tbody>
<tr>
<td>As-produced</td>
<td>5.8%</td>
<td>94.2%</td>
</tr>
<tr>
<td>Age hardened 480°C, 5h</td>
<td>9.4%</td>
<td>90.6%</td>
</tr>
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</table>

As-built parts are not fully martensitic: also an austenitic phase is present (about 5.8%). The aging causes an increase of the austenite phase to about 9.4%, indicating that austenite reversion has taken place. Since the presence of austenite was found under all aging conditions (not only overaged samples), it may be assumed that reversion is taking place before the re-dissolution of the Ni₃(Ti, Mo) precipitates. In SLM maraging steel parts, Ni-rich zones (caused by the segregation that is coupled with the cellular-dendritic growth) make an early austenite reversion possible [11]. Although some austenite is already present, the hardness could still be increased over the hardness values normally found for wrought maraging steel.

4.3. Mechanical properties

Inclusions deteriorate the mechanical properties of the maraging steel, especially in the aged conditions where this steel grade is more brittle than in the as-built condition. Therefore, the specimens for mechanical testing were produced in order to obtain maximum density. Therefore, every layer was re-melted according to the island scanning strategy with the same SLM parameters.

4.3.1. Tensile tests

The mechanical properties that result from the tensile tests are given in Table 3, for as-produced and age hardened test specimens. For comparison, Table 3 also summarizes these properties for wrought maraging steel 300 [12, 13]. The values show the mean value for 4 specimens with 95% confidence intervals.
Table 3. Mechanical properties of maraging steel 300, SLM-produced.

<table>
<thead>
<tr>
<th></th>
<th>x ± s</th>
<th>E</th>
<th>Rp₀₂</th>
<th>Rm</th>
<th>ε break</th>
<th>HRC</th>
</tr>
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<tbody>
<tr>
<td></td>
<td>GPa</td>
<td>MPa</td>
<td>MPa</td>
<td>%</td>
<td></td>
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<tr>
<td>SLM-produced</td>
<td>163 ± 4.5</td>
<td>1214 ± 99</td>
<td>1290 ± 114</td>
<td>13.3 ± 1.9</td>
<td>39.9 ± 0.1</td>
<td></td>
</tr>
<tr>
<td>SLM + aging (5h, 480°C)</td>
<td>189 ± 2.9</td>
<td>1998 ± 32</td>
<td>2217 ± 73</td>
<td>1.6 ± 0.26</td>
<td>58 ± 0.1</td>
<td></td>
</tr>
</tbody>
</table>

E = Young’s modulus  
Rp₀₂ = 0.2% proof stress  
ε break = Elongation at break  
HRC = Hardness Rockwell C

Aging for 5 hours at 480°C leads to an increase in hardness and strength through the precipitation of Ni₃(Mo, Ti) and Fe₂Mo intermetallics. The ultimate tensile strength increases from 1290 MPa to 2216 MPa, which is an increase of 72%. The Rockwell C hardness increases with 45% to reach a value of 58 HRC, however the elongation was reduced from 13% to 1.6% by age hardening the maraging steel.

Tensile tests also show an increase in Young’s modulus to 189 GPa after applying the heat treatment. The formation of the Ni₃(Mo, Ti) precipitates lower the share of Nickel in the martensite matrix, leading to an increase in Young’s modulus, which is consistent with the study presented in [14].

A comparison of the mechanical properties of the SLM-specimens and wrought maraging steel, indicates the occurrence of aging during the SLM process. When melting a track, the neighboring material and layers beneath are heated. This allows precipitation reactions to occur in the heat affected zones. The microsegregation accompanied with the cellular growth may facilitate this. In SLM with laser re-melting after each layer, this phenomenon is more pronounced due to the additional heating in the surrounding material.

The embrittlement that is caused by the heat treatment is also shown in the fractography. The specimen without heat treatment broke after substantial plastic deformation. Typical for a ductile fracture is the formation of dimples (Figure 7a). In the part of the test specimen that undergoes a large deformation, micro-cavities arise where there are precipitates or imperfections. These cavities cause large stresses which lead to more micro-cavities. Eventually these cavities conjoin and the tear grows fast. A ductile fracture is always trans-granular [15].

For the age hardened specimen, the fracture is very brittle. There was hardly any plastic deformation before rupture. Both trans-granular and inter-granular fractures appear as shown in Figure 7b.

Figure 7 a): Formation of dimples at ductile fracture for as-produced specimens. b): Inter- and trans-granular fracture for brittle age hardened specimen.
4.3.2. Charpy impact tests

The Charpy impact test is a common method used to determine material toughness by hitting a test specimen with a hammer, mounted at the end of a pendulum. The specimen is broken by a single blow from the pendulum that strikes the middle of the specimen on the un-notched side. A V-shaped notch is generally used in the impact specimen in order to control the fracture process by concentrating stress in the area of minimum cross-section. In this study, Charpy tests are done according to ASTM E23 standard [16]. The size of the standard specimen is 10x10x55 mm with a notch as defined in the same standard.

![Figure 8: Results for Charpy impact tests on maraging steel 300.](image)

After the parts were produced by SLM, they were sand-blasted in order to remove the loosely sticking powder to the specimens’ walls. Some of the samples were then heat treated while some were kept as-built for a comparison. The impact energies are plotted in Figure 8 for the samples produced on the Concept Laser M3 linear machine.

Charpy tests show a significant decrease in toughness when an aging heat treatment is applied. The reduction in the toughness depends on the aging conditions. These conditions lead to higher hardness and result in lower toughness. The intermetallic precipitates promote the brittle fracture, so less energy will be needed to cause a complete rupture for a higher hardness. The comparison of the fracture surfaces shown in Figure 9 also indicates the brittleness of the heat treated specimens.

When compared to conventionally produced maraging steel 300, SLM processed specimens show a reduced toughness (30-40%) in the heat treated condition. As explained before, this may be due to the formation of Ti-Al combined oxides in the martensite matrix which deteriorate the mechanical properties significantly in the heat-treated condition when the material is much more brittle.

![Figure 9: Fracture surfaces of Charpy samples a) as-built b) after heat treatment](image)
5. Conclusions

Higher layer thickness and/or scan speed causes a decrease in density, which leads to a decrease in macro-hardness. On the other hand, there is no significant influence of layer thickness and scan speed on the micro-hardness of maraging steel 300 in the tested ranges.

Solidification takes place by cellular-dendritic growth mechanisms in SLM of maraging steel grade 300. Intercellular spacing is less than 1μm which contributes to the excellent strength and hardness achieved in both as-produced and age hardened conditions.

Re-melting of every layer causes higher density and an additional natural aging.

Age hardening the SLM-produced maraging steel for 5 hours at 480°C leads to a hardness of 58 HRC and an increase of ultimate tensile strength from 1290 MPa to 2216MPa. Elongation was reduced from 13% to 2%. The heat treatment causes an increase in Young’s Modulus to 189GPa due to the changes in the matrix for the formation of precipitates.

Comparison of the mechanical properties for wrought maraging steel and SLM-produced maraging steel shows the natural aging due to the SLM-process and due to re-melting every layer. The ductility and toughness values obtained by SLM were found to be lower than the ones obtained by conventional techniques.

References