Sub-grain structure in additive manufactured stainless steel 316L

Yuan Zhong

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Abstract

The thesis focuses on exploring the sub-grain structure in stainless steel 316L prepared by additive manufacturing (AM). Two powder-bed based AM methods are involved: selective laser melting (SLM) and electron beam melting (EBM). It is already known that AM 316L has heterogeneous property and hierarchy structure: micro-sized melt pools, micro-sized grains, nano-sized sub-grain structure and nano-sized inclusions. Yet, the relation among these structures and their influence on mechanical properties have not been clearly revealed so far. Melt pool boundaries having lower amount of sub-grain segregated network structures (Cellular structure) are weaker compared to the base material. Compared with cell boundaries, grain boundaries have less influence on strength but are still important for ductility. Cell boundaries strengthen the material without losing ductility as revealed by mechanical tests. Cellular structure can be continuous across the melt pool boundaries, low angle sub-grain boundaries, but not grain boundaries. Based on the above understanding, AM process parameters were adjusted to achieve customized mechanical properties. Comprehensive characterization were carried out to investigate the density, composition, microstructure, phase, magnetic permeability, tensile property, Charpy impact property, and fatigue property of both SLM and EBM SS316L at room temperature and at elevated temperatures (250°C and 400°C). In general, SLM SS316L has better strength while EBM SS316L has better ductility due to the different process conditions. Improved cell connection between melt pools were achieved by rotating 45° scanning direction between each layer compared to rotating 90°. Superior mechanical properties (yield strength 552 MPa and elongation 83%) were achieved in SLM SS316L fabricated with 20 µm layer thickness and tested in the building direction. Y2O3 added oxide dispersed strengthening steel (ODSS) were also prepared by SLM to further improve its performance at elevated temperatures. Slightly improved strength and ductility (yield strength 574 MPa and elongation 90%) were obtained on 0.3%Y2O3-ODSS with evenly dispersed nanoparticles (20 nm). The strength drops slightly but ductility drops dramatically at elevated temperatures. Fractographic analysis results revealed that the coalescence of nano-voids is hindered at room temperature but not at elevated temperatures. The achieved promising properties in large AM specimens assure its potential application in nuclear fusion. For the first time, ITER first wall panel parts with complex inner pipe structure were successfully fabricated by both SLM and EBM which gives great confidence to application of AM in nuclear industry.

Keywords: Additive manufacturing, Selective laser melting, Electron beam melting, stainless steel, Oxide dispersion strengthened steel, Cellular structure, Nano-inclusions.

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‘Snow in a PhD candidate’s life and work’ captured by the author

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IV. Manipulating the sub-grain cellular network structure during selective laser melting
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VIII. Fractography of self-glazed zirconia with improved reliability
*Journal of the European Ceramic Society, In Press*

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J. Zou, Y. Zhong, J.Z. Zhang, M. Ekelund and Z.J. Shen

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B. Qian, C.H. Xiao, J. Zou, Y. Zhong and Z.J. Shen
*RSC Advances* 4.58 (2014): 30754-30757

XI. Neck-formation between Ti6Al4V powder granules exposed to the electron beams
Submitted

XII. Comparison between microstructures, deformation mechanisms and micromechanical properties of 316L stainless steel consolidated by laser melting
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<tr>
<td>3D</td>
<td>Three Dimensional</td>
</tr>
<tr>
<td>AM</td>
<td>Additive Manufacturing</td>
</tr>
<tr>
<td>CAD</td>
<td>Computer Aided Design</td>
</tr>
<tr>
<td>CT</td>
<td>Computer Tomography</td>
</tr>
<tr>
<td>EBSD</td>
<td>Electron Back-Scattered Diffraction</td>
</tr>
<tr>
<td>EDS</td>
<td>Energy Dispersive X-ray Spectroscopy</td>
</tr>
<tr>
<td>FCC</td>
<td>Face-Centered Cubic</td>
</tr>
<tr>
<td>HIP</td>
<td>Hot Isostatic Pressing</td>
</tr>
<tr>
<td>ICP-OES</td>
<td>Inductively Coupled Plasma Optical Emission Spectrometry</td>
</tr>
<tr>
<td>MMC</td>
<td>Metal Matrix Composite</td>
</tr>
<tr>
<td>ODSS</td>
<td>Oxide Dispersion Strengthening Steel</td>
</tr>
<tr>
<td>OM</td>
<td>Optical Microscopy</td>
</tr>
<tr>
<td>SLM</td>
<td>Selective Laser Melting</td>
</tr>
<tr>
<td>SEBM</td>
<td>Selective Electron Beam Melting</td>
</tr>
<tr>
<td>SEM</td>
<td>Scanning Electron Microscopy</td>
</tr>
<tr>
<td>SS316L</td>
<td>Stainless Steel grade 316L</td>
</tr>
<tr>
<td>TEM</td>
<td>Transmission Electron Microscopy</td>
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<tr>
<td>XRD</td>
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<td>XRF</td>
<td>X-ray Fluorescence</td>
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1. Introduction

1.1 Metal additive manufacturing technology

Recently, metal additive manufacturing (AM) has drawn international attention due to its production advantages and the increased use of AM components in different applications. As the name suggests; metal AM fabricates a 3-dimensional (3D) component in a one-step process directly from metal precursor powders following a computer aided design (CAD) model. The metal AM is generally more difficult than AM of non-metallic materials. The energy absorption and the rapid heat distribution in metals away from the focussed energy beam spot area is different. A high melting point of any present refractory metal combined with an often very complexed metal solidification process makes it difficult to control the final result. Still, several materials with broad industry applications have already been successfully manufactured with satisfactory density from metal AM methods; which include stainless steels, nickel alloys, cobalt chrome, titanium alloys and aluminium alloys.

The metal AM family has three main branches depending on the precursor material and the used material feeding system: powder-bed system, powder-feed system and wire-feed system. The research in this project is conducted with a powder-bed system and two different energy beam methods: selective laser melting (SLM, Figure 1a) and selective electron beam melting (SEBM, Figure 1b). Both of SLM and SEBM follow a similar layer by layer manufacturing schedule: (1) the energy beam is focused upon the powder bed surface and melts the metal powder according to the pre-set CAD solid component contour; (2) the building plate lowers one-layer thickness and a new layer of powder is dispersed; (3) repeat step (1).

The melting process in SLM is trigged by a 200 W energy laser beam with a spot size of ~75 μm. It is normally conducted in inert N\textsubscript{2} or Ar atmospheres with external preheating to warm the building platform to 80°C. The layer thickness varies from 20-50 μm. Two commercial SLM machines are used in this study: EOS M270 from EOS Germany (Figure 1c) and AM250 from Renishaw UK (Figure 1d).

The SEBM process is conducted with an electron beam with up to 2 kW energy and within a vacummed chamber. Prior to the contour melting a pre-heating by a defocused electron beam is done to generate weak bonds between the powder particles that will prevent the repelling of particles due to
electrical charging. The layer thickness can vary from several tens of microns to hundreds of microns depending on the purposes. The SEBM facility used in this study is Arcam A2 from Arcam Sweden (see Figure 1e).

Figure 1-1. Schematic drawing of SLM (a) and SEBM facility (b); commercial SLM facilities: EOS M270 (c) Renishaw AM250 (d) and SEBM facility Arcam A2 (e). Reprinted from Ref. 24 with permission

1.2 Hierarchical microstructure features

The materials prepared by SLM and SEBM have different properties due to different parameters, processes and facilities. However, the materials prepared by either SLM or SEBM possess some common features: residual stresses\textsuperscript{55,27}, anisotropy, properties and hierarchical microstructures. The latter structures comprise melt pools, grains and sub-grain structures\textsuperscript{4}.

The energy beam scans upon the material surface and the high energy input will melt the target material leaving melt traces behind. Typical cross-section and side views of the formed melt pools can be found in Figure 1-2. Thus, the as-built material consists of stacking of a great number of melt pools. Investigation of the formation mechanism and behaviour of the melt pools aim to minimize the presence of any macro defects. The stability of a single laser melted track is crucial to fabricate complex parts with mechanical properties comparable to those of wrought material.\textsuperscript{28} Balling phenomenon is harmful to achieve a high density material and the mechanism has been explored and possible ways suggested to control it.\textsuperscript{29,31} Denudation of metal powders near the laser scanning track may result in a local lack of powder that develops into a pore.\textsuperscript{32} Many techniques have been used to control the laser melting process and to reduce defects; including synchrotron radiation micro-CT\textsuperscript{33}, simulation of the impact of laser intensity profile on the melt
track macrostructure and grain structure\textsuperscript{34}, high speed imaging and modelling of the formation of pores and a rough surface\textsuperscript{35}

\begin{figure}[h]
\centering
\includegraphics[width=0.5\textwidth]{figure1-2.png}
\caption{Melt traces in the cross-section plane (a) and melt pools in the side plane (b) revealed in etched SLM SS316L. Reprinted from Ref. 61 with permission.}
\end{figure}

The SLM/SEBM are featured by rapid melting and solidification processes in non-equilibrium state. The solid microstructure is ruled by the temperature gradient and grain growth rate, as shown in Figure 1-3a. In the SLM/SEBM process, the G/R values are high enough to facilitate planar growth and cellular/dendritic grain growth.\textsuperscript{35} There might also be some columnar dendritic growth and equiaxed growth near the centreline of the melt pool where the cooling rate is normally lower. However, the re-melting process of the next layer will diminish the dendrites and equiaxed grains at the melt pool centreline. Therefore, the AM material mainly consists of a planar and cellular structure, as observed in most studies.\textsuperscript{35}

Regarding a single melt pool, most grains grows into the liquid without changing the crystallographic orientation of the seed grain, i.e. epitaxial grain growth. However, the grains might have either epitaxial or non-epitaxial growth near the fusion line and a competitive grain growth away from the fusion line, as shown in Figure 1-3b. Thus, grains further away from the fusion line tends to grow towards either the maximum temperature gradient direction of the melt or the “easy” (low-energy) growth direction of the crystal.

Efforts have been made to control or modify the grain structure and texture, since the grains play such an important role in determining the material properties. Simulation showed that the grain texture will change by different scanning strategies and this was proved by subsequent experimental results.\textsuperscript{38} The columnar structure grow epitaxially from partially melted grains, but change growth direction from the seed crystal toward the direction of the largest temperature gradient found in the melt pools.\textsuperscript{35} The microstructure was even observed to change along the height of part and presented a gradient structure.\textsuperscript{36,37}
Inside the grains, a typical sub-grain cellular structure is observed in many metallic AM materials. This cellular structure is featured by an element segregated boundary. The segregated element varies in different materials. For example, Cr and Mo segregation is observed in SLM SS316L⁶ while Si segregation is found in SLM AISi10Mg². Dislocations tend to accumulated at the same sites as the segregations, as seen in Figure 1-4c. The dislocation pinning effect of cell boundaries, sub-cell boundaries and particles was proved by in-situ compression tests in TEM.⁴² Considering the cellular structure, a suitable heat treatment regime is also required for metallic AM materials⁴³,⁴⁴.

Some nanoinclusions are generated in the material matrix with the size around several tens of nanometres by in-situ reaction between active metals and oxygen. The oxygen may come from the precursor powders or from residual oxygen in the protecting chamber atmosphere.
Based on previous results, the hierarchical structure of AM materials can be summarized in Figure 1-6.

Figure 1-6. Summary of hierarchical structure: parts built by SLM (a), melt pools stacking (b), grains in a single melt pools (c), sub-grain cellular structure (d) and nano-inclusions (e). Reprinted from Ref. 23 with permission

1.3 Basics of sub-grain structure

As mentioned above, the sub-grain structure comprises the cellular structure and the nano-inclusions.

1.3.1 Cellular structure

The sub-grain cellular structure has attracted much attention and many studies have been carried out to investigate its formation, its relation to the mechanical properties and possible ways to control it. The cellular structure is a result of solute redistribution and segregation during cellular/dendritic grain growth. The formation mechanism is similar to the sub-grain structure
of a weld pool. Constitutional undercooling \(^{45}\) explains the solidification mode change in Figure 1-3a from thermodynamic aspects and cellular/dendritic grain growth is facilitated in the way as explained in section 1.2. Stainless steel 316L is used as a typical example to explain the segregation process. As seen from the phase diagram of the binary Fe-Mo system in Figure 1-6, the amount of solute Mo in the solid is less than Mo in the melt. Similar phenomenon also exists in the Fe-Cr binary system. \(^{46}\) Therefore, the Mo/Cr elements would be “squeezed out” at the growing solid cellular/dendrite front and be accumulated in the remaining melt. When two adjacent cellular/dendrite fronts meet a thin boundary rich in Mo/Cr is generated between them.

In other words, the cellular structure is formed by solute segregation similar to typical grain growth in a weld pool. The stacking of melt pools generates a network of cellular segregation structures in the whole material. The complex thermal history makes the control of these cellular structures difficult. The cell morphology, cell spacing and the cell orientation are determined by factors like the thermal gradient, the undercooling, the heat flux and the Marangoni flow. \(^{47}\) Recently, it was observed that the energy input of laser beam influences the cell spacing, the sample density and in turn strongly affected the mechanical properties. \(^{39}\)

![Fe-Mo Phase diagram](image)

*Figure 1-6 Fe-Mo Phase diagram \(^{47}\). Reprinted from Ref 47 with permission*

1.3.2 NanoInclusions

NanoInclusions are formed by *in-situ* chemical reactions in the hot melt pool. The elements that have high affinity to oxygen react with any oxygen present. Small oxygen amounts might be present either in the precursor powder or in the process chamber. The oxide nanoInclusions solidify prior to the base metal due to their high melting point and migrate along with the melt pool flow and grow larger by time. The Marangoni flow in the melt pool disperses the nanoInclusions evenly and the high solidification rate

..
prevents the nanoinclusions from agglomeration and growing. The nanoinclusions (rich in Cr, Si and O) present in SLM SS316L solids are normally randomly dispersed in the metal matrix with a size around several tens of nanometres. Well distributed nanoinclusions might have a positive effect on the mechanical properties of steel by pinning the dislocation flow during mechanical stress. Therefore, many methods have been tried in order to increase the amount of nanoinclusions to fabricate oxide dispersed strengthening steel (ODSS). The precursor powders for SLM can be pre-alloyed or ball milled mixtures. Laser melting of ball milled reinforcement powder and matrix powder has been used with the aim to fabricate in-situ formed nanoinclusions, i.e. steel ODSS nanocomposites.\(^48, 49\) However, segregation or agglomeration of nanoinclusions in the steel structure to form larger faults are negative. This behaviour was observed in all the SLM experiments using pre-alloyed ferritic powders and was found to deteriorate the mechanical properties.\(^50-53\) Surface laser melting of a mixture of steel powder and \(\text{Y}_2\text{O}_3\) powder (20-50 nm) also resulted in particle coarsening to 150 nm due to active agglomeration of Y-Ti-O nanoinclusions, carbides and Ar bubbles.\(^54\) Sintering SS316L powder (50-100 \(\mu\)m) and \(\text{Y}_2\text{O}_3\) powder (\(\leq 40\) nm) was also tried by SEBM, but a low tensile strength was obtained. However, ball milled mixture of ferritic powder, W (800 nm), Ti (50 nm) and \(\text{Y}_2\text{O}_3\) (40 nm) was successful consolidated by SEBM with a homogeneous dispersion of oxide precipitates.\(^55\) Recently, Springer et al. developed a new way of producing oxide- and nitride- dispersion strengthened materials through atmospheric reaction in melt metal deposition.\(^56\)

1.4 Stainless steel 316L

SS316L is a molybdenum-bearing austenitic stainless steel that is widely used in architecture, locomotion industry, medicine, etc. It has also been selected as one structural material in nuclear fission and fusion industry due to its combination of good mechanical properties at elevated temperatures, excellent corrosion resistance and good machinability.\(^57, 58\) The materials used in a fusion reactor has stricter control on composition and mechanical properties due to the critical working environment. The composition range and mechanical property requirement are listed in Table 1-1.

| Table 1-1. Requirements for SS316L-IG (ITER Grade)\(^59\) |
|-----------------|----------------|-----------------|----------------|----------------|----------------|----------------|----------------|
| 316L ITER       | C   | Si   | Mn   | P    | S    | Cr   | Ni   | Mo   | Cu   | N   | Fe   |
|                 | \(<0.03\) | \(<0.5\) | 1.6-2 | \(<0.025\) | \(<0.01\) | 17-18 | 12-12.5 | 2.3-2.7 | \(<0.3\) | 0.06-0.08 | Bal |
| Temperature     | Tensile strength | Yield strength | Elongation |
1.5 Aim of this study

This thesis aims to achieve a deeper understanding of formed sub-grain structures in stainless steel 316L (sub-grain cellular structure and nano-inclusions) manufactured by both SLM and EBM prepared SS316L. The relation between sub-grain cellular structure, the melt pools and the grains was investigated by comparing SEM images (on an etched surface) and EBSD images at the same spot. Efforts were made to manipulate the cell spacing and cell growth continuity by adjusting scanning parameters and scanning strategies in SLM in order to improve the strength and ductility of SLM SS316L. Mechanical properties were tested and their relation to the microstructure was discussed by carefully observing the fracture surfaces and the microstructure of tensile tested specimens.

The sub-grain structure in a large EBM SS316L specimen was revealed and compared with that from smaller sized specimens in order to investigate the microstructure homogeneity in the larger specimen. The mechanical properties were also tested. The aim is to provide useful advices for large scale EBM component production for industrial applications.

A new way of preparing ODSS by SLM was developed by laser melting mixed steel powder and ceramic powder to broaden the usage of SS316L in industry. Different small amounts of additional Y₂O₃ were mixed with steel powder prior to the SLM process with the aim to manipulate the amount of oxide nano-inclusions in the final product. The microstructure and mechanical properties were characterized and compared for the non-ODSS and different ODSS solids at room temperature and at two elevated temperatures of 250°C and 400°C, respectively.
2. Experimental details

2.1 Materials and AM facilities

The precursor materials used in this study was spherical gas-atomized SS316L powder from Carpenter Powder Products AB (Torshälla, Sweden). The size range is 10-45 μm for the SLM facilities and 53-150 μm for the EBM facilities. The morphology of the powder precursors is shown in Figure 2-1. The compositions of the precursor powders are listed in Table 2-1.

![Figure 2-1. The precursor powders for SLM (a) and EBM (b) process. Reprinted from Ref. 24 with permission](image)

<table>
<thead>
<tr>
<th></th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>Cu</th>
<th>N</th>
<th>Fe</th>
<th>O</th>
</tr>
</thead>
<tbody>
<tr>
<td>SLM</td>
<td>0.014</td>
<td>0.70</td>
<td>1.69</td>
<td>0.014</td>
<td>0.004</td>
<td>17.8</td>
<td>12.5</td>
<td>2.38</td>
<td>0.04</td>
<td>0.09</td>
<td>Bal</td>
<td>165 ppm</td>
</tr>
<tr>
<td>EBM</td>
<td>0.013</td>
<td>0.50</td>
<td>1.70</td>
<td>0.009</td>
<td>0.006</td>
<td>17.6</td>
<td>12.3</td>
<td>2.46</td>
<td>0.05</td>
<td>0.075</td>
<td>Bal</td>
<td>145 ppm</td>
</tr>
</tbody>
</table>

Three additive manufacturing facilities were used in this study. The SLM work in Chapter 3 and 4 were carried out in EOS M270, and the work presented in Chapter 6 was carried out in Renishaw AM250 and, finally, the EBM work in Chapter 5 was done in Arcam A2.

2.2 Process parameters

The scanning strategies and parameters are summarized in Figure 2-2. Three scanning strategies are illustrated below which are named meander, strip and island pattern. Meander pattern is the basic strategy for parameter develop-
ment that generates the largest residual stress in the as-build specimen. Striae and island pattern are normally applied for manufacturing the larger components in which the parameters development should also regard the spacing between strips or islands. The scanning parameters are laser power (P), scanning speed (v), hatch spacing (d) and layer thickness (h). A relative energy density (W) is then calculated to guide the parameter development:

\[ W = \frac{P}{v \cdot d \cdot h} \]

The detailed parameters for different experiments will be introduced in each separate section.

![Schematic drawing of the scanning strategies and process parameters](image)

**Figure 2-2. Schematic drawing of the scanning strategies and process parameters**

### 2.3 Characterization methods

X-Ray Diffraction (XRD) patterns were collected using a PANalyticalX’Pert Pro diffractometer with a copper radiation source (40 mV voltage and 20 mA current) and with a Ge monochromator. It took 7.5 h to record the analyzed 2θ–angle range of 40-100°. The average densities of the as-build samples were measured by Archimedes law in deionized water and were also checked by image analysis from OM images. Scanning electron microscopy (SEM) images were taken by JEOL JSM-7000F field emission scanning electron microscopy (JEOL, Tokyo, Japan). The cutting, mounting and polishing were carried out by Secotom-50 cutting machine, CitoPress-15 mounting press and LaboPol-60 grinding and polishing system (Struers Aps, Denmark). The etching agent is diluted HF solution (HF/HNO₃/H₂O = 1:4:45). The chemical composition was analyzed according to ASTM E353 or by an Energy Dispersive X-ray Spectroscopy (EDS) detector (Oxford Instruments, Oxford, UK). Transmission electron microscope (TEM, LaB₆ 2100, JEOL, Tokyo, Japan) was used for detailed microstructure characterizations. The samples for TEM analysis were prepared by mechanical polish-
ing followed by electrochemical polishing using Struers TenuPol-5 Jet Polisher. The electrochemical polishing process is carried out at -10°C with a solution \((\text{H}_2\text{O} : \text{HClO}_4 : \text{C}_2\text{H}_6\text{O} : \text{C}_6\text{H}_14\text{O}_2 = 9:7.8:73.1:10)\).

2.4 Mechanical properties

Mechanical tests were performed according to international standards: ASTM E8 (tensile test at RT), ASTM E21 (tensile test at an elevated temperature), ASTM E23 (Impact test) and ASTM E606 (Fatigue test). Some general information is listed below while other details will be introduced in each separate section:

- In Chapter 4 and 6, the mechanical tests were performed at Tecnalia (San Sebastian, Spain). The sizes of the as-build specimens are \(\Phi17\times150\) mm for tensile tests, \(11\times11\times70\) mm for Charpy-V tests and \(\Phi13\times162\) mm for fatigue tests. The tensile test used a crosshead speed of 1 mm/min up to yield value, and afterward 3 mm/min up to tensile strength. The fatigue tests were conducted at 250°C in a strain controlled manner under symmetric tension/compression conditions \((R=-1)\) using a 0.5 Hz sine cycle and an axial strain amplitude of 1%.

- In Chapter 5, tensile tests were performed at Shanghai Aeronautical Materials & Structures Testing Co., LTD (Shanghai, China). The size of the as-build specimens for tensile tests is \(\Phi8\times52\) mm. The strain rate is 0.015 before yielding and 0.05 after yielding at RT, and is 0.005 before yielding and 0.05 after yielding at ET. The effective cross-section is \(\Phi3\) and gauge length is 12 mm.

- In Chapter 3, tensile tests were carried out in Institute of Materials Research of Slovak Academy of Sciences (Kosice, Slovakia). The size of as-build specimens is \(40\times4\times1\) mm for tensile tests. The cross-section is \(1\times1\) mm and the gauge length is 10 mm.

- Vickers hardness was determined by a Zwick/Roell ZHV indenter with a dual time of 10s.
3. Sub-grain cellular structure in SLM SS316L

Before entering to details, some definitions are clarified here:

- Normal grain boundaries are high angle boundaries with crystallographic misorientation >15°
- Sub-grain boundaries are low angle boundaries with crystallographic misorientation <15°
- Cellular structure is a sub-grain dislocation and segregation network structure
- Colony of cells is a group of sub-grain cellular structure with the same morphological orientation and cell spacing

3.1 A deeper understanding of the cellular structure

An EBSD mapping depicting crystallographic orientation difference in SLM SS316L is shown in Figure 3-1. Besides the grains boundaries (bold white line), some sub-grain boundaries (thin white line) and a large number of boundaries with misorientation larger than 0.5° are revealed. It proves the presence of low angle sub-grains and also indicates the adjacent cells may have small crystallographic misorientation. As discussed in the introduction part, the cellular structure is a result of the cellular grain growth and element microsegregation. The first question arises; are the colonies of cells all identical (with both same cell spacing and same cell orientation) within the grain? The positions of the cell boundaries and the grain boundaries were compared. The result is shown in Figure 3-1b where the grain boundaries are outlined in red and the sub-grain boundaries are outlined in green. The magnified images of the local sites are shown in Figure 3-1c, d and e. It is clear seen that there are different colonies of cells in the same grain. Then the second question arises; are the colonies of cells the same in the shared sub-grain? The colonies of cells in a sub-grain are always the same, but not if they cross melt pool boundaries.
It can now be concluded that although the cellular structure is a result of sub-grain growth it is also influenced by the temperature gradient inside the melt pool. The relationship between cellular structure, grains and melt pools can be summarized in Figure 3-2. There are five possible cases:

1. Colonies of cells are the same within one sub-grain across melt pool boundaries.
2. Colonies of cells are different within one sub-grain across melt pool boundaries.
3. Colonies of cells are different in different sub-grains.
4. Colonies of cells are the same in different sub-grains.
5. Colonies of cells are the same within one sub-grain and one melt pool.

A schematic drawing of the different cases is shown in Figure 3-2a and all these cases have been observed by comparison of BSE images and morphological images exposed at the same place of an etched surface, as shown in Figure 3-2 b-d. Case 5 is resulted from the same low-energy crystallographic growth direction and the locally identical direction of the temperature gradient. At the melt pool boundaries, the grains grow either in an epitaxial mode from a previous grain or in a non-epitaxial mode based on new nucleation. In the epitaxial grain growth, the colonies of cells continuously grow from the previous colonies and keep a similar growth direction if the change of temperature gradient direction is small, which results in case 1. If the temperature gradient changes more significantly, the cellular growth direction also changes which results in case 2. In the non-epitaxial grain growth, the rev
grain starts by planar grain growth and follows by cellular grain growth, which results in case 3. Finally, the colonies of cells in adjacent sub-grains with small misorientation merge and grow toward along the temperature gradient direction, which results in case 4.

The colonies of cells exhibit a continuous feature in case 1, 4 and 5 while there is a ‘gap’ between the adjacent colonies without any segregation network in case 2 and 3. It is known that the cellular segregations have strengthening effects due to the dislocation accumulation at cell boundaries forming a dislocation wall. It is reasonable to predict that a ‘gap’ without dislocations will weaken the strengthening effect to some extent. In addition, the melt pool boundaries are sinks for defects and are assumed as weak points. Therefore, minimizing the gaps without introducing defects at the melt pool boundaries is a possible way to improve the material behavior. That means case 1, 4 and 5 should be encouraged while case 2 and 3 should be minimized. Some possible ways are listed below:

1. Enlarge the melt pool profile to generate fewer melt pool boundaries, which benefits case 5 and lowers the possibility of case 2. It can be achieved by decreasing the scanning speed or by increasing the layer thickness.
2. Ensure the temperature gradient in adjacent melt layers is similar, which facilitates case 1 and 4. It can be achieved by changing the laser scanning direction in adjacent layers.
3.2 Cell spacing control

As discussed above, the scanning speed was changed with the aim to adjust cell spacing. The hatch spacing was tuned simultaneously to keep the same
energy input. Cubic specimens with 10 mm length were prepared. The parameters are summarized in Table 3-1.

Table 3-1 Processing parameters for cell spacing control and the calculated relative density (theoretical density (TD) of SS316L is 8000 kg/m³). Reprinted from Ref. 60 with permission

<table>
<thead>
<tr>
<th>No.</th>
<th>P, w</th>
<th>v, mm/s</th>
<th>d, mm</th>
<th>h, mm</th>
<th>W, J/mm³</th>
<th>angle, °</th>
<th>ρ, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>C45-1</td>
<td>195</td>
<td>7000</td>
<td>0.01</td>
<td>0.02</td>
<td>139</td>
<td>45</td>
<td>95.3</td>
</tr>
<tr>
<td>C45-2</td>
<td>195</td>
<td>4250</td>
<td>0.02</td>
<td>0.02</td>
<td>114.5</td>
<td>45</td>
<td>98.6</td>
</tr>
<tr>
<td>C45-3</td>
<td>195</td>
<td>1700</td>
<td>0.05</td>
<td>0.02</td>
<td>114.5</td>
<td>45</td>
<td>99</td>
</tr>
<tr>
<td>C45-4</td>
<td>195</td>
<td>850</td>
<td>0.1</td>
<td>0.02</td>
<td>114.5</td>
<td>45</td>
<td>99.2</td>
</tr>
<tr>
<td>C45-5</td>
<td>195</td>
<td>566</td>
<td>0.15</td>
<td>0.02</td>
<td>114.5</td>
<td>45</td>
<td>99.5</td>
</tr>
</tbody>
</table>

The average cell spacing, density and Vickers hardness of different specimens are shown in Figure 3-3. The average cell spacing in each specimen is calculated by counting more than 1000 cells at 10 randomly chosen sites. As expected, lower cooling rate (lower v) facilitates the growth of cells of larger size. The cell spacing is estimated to be 300 nm with v=7000 mm/s, while it becomes 660 nm with v=566 mm/s. The coarsening of cells is more clearly seen in Figure 3-3b. However, although the input energy density is similar, the bulk densities are still influenced. The highest relative density of 99.5% is obtained in specimen C45-5 with the lowest scanning speed. Hardness tests over a larger volume were carried out by applying a high load of 10 kgf. Five tests were performed on each specimen. It is found that the relative density takes the leading role in increasing hardness until it reaches 99.2%TD. Then, increase of density result in reduced. A possible explanation is that the cell spacing starts to influence the hardness when the tested area is dense enough.
Therefore, to understand the influence of the cell spacing on hardness, the indentation hardness was measured on a limited specimen area having different cell spacings with a low load (100 gf). The small indentation size (≤30 μm) makes it possible to hit a single colony of cells. Hardness values were recorded and then the specimen was etched to reveal the cellular structure for cell spacing calculation at each indentation mark. The average cell spacing is calculated by counting more than 50 cells around the indentation. More than 100 hardness values were recorded. The results revealed that the hardness increases with decreasing cell spacing, as shown in Figure 3-4b. The hardness is 240(HV100) at sites where cell spacing is 0.554 μm and it recedes to 210(HV100) where cell spacing is 0.889 μm. A linear relation between the local hardness and the square root of cell spacing is extracted from Figure 3-4b:

\[ HV = 449.3 - 251.9 \times d^{-\frac{1}{2}} \]

where HV is hardness and d is the average cell spacing. This relation is similar to the Hall-Petch law and thus reflects the boundary strengthening effect of the sub-grain cellular structure at a much smaller level than grains.
Efforts have also been made to verify if the colonies of cells have anisotropic hardness in different directions, as shown in Figure 3-4c. The average hardness in z direction (Figure 3-4d) is 234 (HV100) while that in xy direction (Figure 3-4e) is 227 (HV100). Thus, the insignificant difference indicates no or very small hardness anisotropy. However, the calculated indentation depth is only 5 μm, which is not enough for a quantitative comparison since the cell spacing is several hundred nanometers. A higher indentation load might solve the problem, but an enlarged indentation area will include more than a single colony of cells.

**Figure 3-4.** Indentation hardness with respect to melt pools (a), Hardness versus cell spacing (b), schematic drawing of cells (c), indentation taken in direction z (d), indentation taken in direction xy. Reprinted from Ref. 60 with permission

According to the Hall-Petch strengthening theory, the dislocation pinning at the grain boundaries has an effect on the yield strength. Dislocations were also observed at the sub-grain cellular structure boundaries and a strengthening effect was expected due to the much smaller cell spacing. The relation between cell spacing and the yield strength fits well with the Hall-Petch law according to our recent research and the above results.61, 62 A drastic drop of the yield strength is observed for heat-treated SLM specimens where the cellular structures disappear. In addition, the relation between crystallographic texture and strength was vague.6, 43, 61, 64 It is reasonable to conclude that the great amount of dislocations at the sub-grain cellular boundaries are essential in influencing the yield strength. This fit with the observations from SLM prepared materials with lower amount of residual dislocations at the grain boundaries.

In summary, the average cell spacing is successfully adjusted by changing the scanning speed. The hardness changes by an inverse relation with the cell
spacing at high enough density. However, it is impossible to keep the same density even when a similar input energy was applied. Within the narrow process window of SLM SS316L, this method of changing scanning speed and hatch spacing has a limited application potential to achieve customized mechanical properties.

3.3 Cell continuity control

The cell continuity may be changed by changing the scanning directions in adjacent layers, as explained in section 3.1. Therefore, experiments are designed as shown in Figure 3-5 and the process parameters are listed in Table 3-2.

![Figure 3-5: Illustration of different test specimens (a), PH45 on the building plate (b), tensile specimens (c), tensile tests facility (d). Reprinted from Ref. 60 with permission](image)

| Table 3-2: Processing parameters for cell continuity control and the calculated relative density (theoretical density (TD) of SS316L is 8000 kg/m³) |
|---|---|---|---|---|---|---|
| No | P, w | v, mm/s | d, mm | h, mm | W, J/mm³ | angle, ° | p. % |
| PH45 | 195 | 850 | 0.1 | 0.02 | 114.5 | 45 | 99.2 |
| PH90 | 195 | 850 | 0.1 | 0.02 | 114.5 | 90 | 99.3 |
| PV45 | 195 | 850 | 0.1 | 0.02 | 114.5 | 45 | 99 |

The relative densities of all specimens reach 99% TD as measured by Archimedes method. No macrocracks or pores as a result of insufficient melting are observed in the optical microscopy images in Figure 3-6. The scanning direction changes are clearly revealed by the melt pool tracks in Figure 3-6a and c. Different colonies of cells can be outlined by the contrast difference.
The colonies of cells are more easily to cross the melt pool boundaries in PH45 than those in PH90.

![Images of cross-sections](image1)

*Figure 3-6. OM image on the cross-section plane (a) and (c), on the side plane (b) and (d). Reprinted from Ref. 60 with permission.*

The discontinuity of cells is more clearly illustrated by SEM in Figure 3-7. Colonies of cells in PH45 cross melt boundaries, as indicated by the arrows in Figure 3-7c, while those in PH90 stop at most of the appearing melt pool boundaries. As stated before, besides factors influencing the sub-grain growth (temperature gradient, "easy" growth direction), colonies of segregation network are also inclined to epitaxial grain growth. If epitaxial grain growth mode is activated when the laser rotates 45°, the colony of cells can connect at the melt pool boundaries. When the laser rotates 90°, the sharp change in temperature gradient direction makes it difficult for epitaxial grain growth. Instead new nucleation occurs, planar grain growth starts and new cellular grain growth follows with different colonies of cells. Similar change of grain growth at melt pool boundaries were simulated and proved in a recent study. A model is thus developed and schematically drawn in Figure 3-7e and f. In PH45, long colonies of cells connect the melt pool boundaries and there will be no gap or very narrow gap at melt pool boundaries, as seen in Figure 3-7g. The strengthening effect of the cells at melt pool boundaries is indicated by the zigzag shaped melt pool boundaries directly observed in SLM SS316L after tensile test, as shown in Figure 3-8. On the other hand, in PH90, short colonies of cells are present in adjacent melt pools, as shown in Figure 3-7f. New grains start to grow with planar growth mode without any segregation network, which results in a gap with width more than several micrometers at melt pool boundaries, as shown in Figure 3-7h. It is reasona-
ble to predict that the melt pool boundaries without segregation network strengthening are more easily to ‘glide’ and be pulled out during tensile tests or to be teared apart during impact tests.

Figure 3-7. Overview of PH45 and PH90 (a) and (b), colonies of cells crossing melt pool boundaries in PH45 and PH90 (c), Schematic drawing of the feature of cells in PH45 and PH90 (e) and (f), gaps between adjacent melt pools (g) and (h). Reprinted from Ref. 60 with permission.
The EBSD mapping in Figure 3-9 proves that most grains in PH90 experience a non-epitaxial growth mode at the melt pool boundaries. The grains are constrained by the melt track boundaries, as seen in Figure 3-9b and d. The grains have more possibility to grow epitaxial in PH45, as seen from Figure 3-9a and c. Thus, a stronger texture in the (101) building direction is also expected in PH45. The texture difference is also observed in another study.  

The fracture surfaces are also examined to further prove the model proposed in Figure 3-7. The models in the fracture tips with respect to the tensile direction are illustrated in Figure 3-10a and e. The etched side view of PH45 in Figure 3-10b clearly reveals the long colonies of cells cross two melt pool boundaries and deform in the building direction. The colonies of cells are teared apart from the longitude side at the site pointed by an arrow in Figure 3-10c and leaving strips on the fracture surface shown in Figure 3-10d. On the other hand, the side view of PH90 shows different colonies of cells in adjacent melt pools. No feature of tearing apart colonies of cells is observed. Instead gliding between colonies of cells with different orientations is observed in Figure 3-10h and leaves some craters on the fracture surface in Figure 3-10g. In other words; the proposed model agrees well with the fracture surface characterization.
Figure 3-9. EBSD of PH45: cross-section plane (a), side plane (c) and colorkey (e); EBSD of PH90: cross-section plane (b), side plane (d) and colorkey (f). Reprinted from Ref. 60 with permission.
PH45 has yield strength of 470 MPa and tensile strength of 608 MPa. PH90 has slightly lower yield strength of 447 MPa and tensile strength of 558 MPa. The elongations at rupture of the two specimens are similar with a value around 30%. The higher strength of PH45 could be the combination
effect of the grain morphology, texture and the sub-grain structure. The larger grains and the weak texture of PH90, identified in Figure 3-9, could be one reason. Another possible reason is the different arrangement of the colonies of cells. The long colonies of cells make the gap across the melt pool boundaries smaller and act as a “rope”. The cells together with the grain boundaries generate a higher initial strain hardening rate in PH45. A larger stress is needed to shear the strengthened boundaries with long colonies in PH45. In PH90, the short colonies are easier to be pulled out since less strengthening occurs. The appearance of the fracture surface of PH45 compared to the presence of more craters on the fracture surface of PH90 indirectly confirms this mechanism.

![Figure 3-11. Engineering stress-strain curve of PV45, PH45 and PH90. Reprinted from Ref. 60 with permission](image)

The strength of SLM SS316L is much higher than that of the traditional SS316L, but the ductility of PH45 and PH90 are still not adequate. The cellular structure is confirmed to influence the strength, but its effect on ductility is still under consideration. Meanwhile, it is well known that the ductility is affected by the grain morphology and the texture. Due to the directional solidification process, the columnar grains tend to grow in the component building direction in SLM SS316L. Previous studies have shown that when more boundaries are encountered, higher strength and lower elongation are expected. Thus, if the tensile stress is applied perpendicular to the longer axis of the columnar grains, higher elongation is expected. Specimens PV45 were fabricated with the laser rotating 45° and tested along building direction. Yield strength of 455 MPa and tensile strength of 551 MPa were obtained, which is a bit lower than that of PH45. However, a significant rise
in elongation reaching 84% was achieved compared to 30% elongation in PH45. The SS316L with such high yield strength and high ductility has never been fabricated by any other method before.69, 70

3.4 Influence of the hierarchical boundaries on mechanical properties

In the section above, the effects of sub-grain boundaries on mechanical properties were discussed. It should be noted that other structure boundaries also affect the properties and the mechanical properties are based on the whole structure features. Here we proposed a summary of the relation between hierarchical boundaries and mechanical properties.

Melt pool boundaries exist in all materials prepared by AM due to its typical layer-by-layer melting fabrication process. Defects are more likely to accumulate at melt pool boundaries and they are normally regarded as weak points of AM materials. To minimize the number of melt pool boundaries are considered good for both strength and ductility. The possible ways of reducing the number of melt pool boundaries could be to increase the melt pool size and to decrease the melt pool overlapping.

Grain boundaries are believed to have great impact on both strength and ductility. However, a great amount of sub-grain cellular boundaries weaken the effect of grain boundaries. The almost doubled yield strength in AM materials proves that Hall-Petch law doesn't agree with grain size but agrees with sub-grain cell spacing. Nevertheless, the fact that PH45 has higher strength than PH90 proves that the grain morphology still has some influence to the observed strength. On the other hand, the influence of grains on ductility is obvious.

Sub-grain cell boundaries can impede the dislocation movement thus increase dislocation storage, resulting in a strengthening effect. The segregated element can keep the shape of the dislocation walls which further strengthen the material via dynamic Hall-Petch strengthening. The achieved outstanding elongation at room temperature can be explained partly by the strong (101) texture along the building direction. Moreover, the ductility may have also been influenced by the cell boundaries. The tremendous dislocations together with the deformation twinning brings recovery of strain hardening rate in SLM SS316L tested in the building direction at room temperature, which delays the necking and increases the uniform elongation. Material failure occurs by coalescence of micro-voids formed during tensile tests, as seen in Figure 3-12a. Cell boundaries can also act as “rope” between melt pool boundaries and strengthen the weak bonding of melt pools. Instead of generating large voids at the melt pool boundaries that trigger immediate necking,
many small voids were observed in the whole gauge length region of the tensile specimen. The voids can be formed at the sites of nano-inclusions or the cell boundary intersections. The micro-voids are observed to be constrained by the cell boundaries, as seen in Figure 3-12b. The cell boundaries prevent the coalescence of micro-voids and thus delay the necking and cracking. Based on the above evidences, the author believes the cell boundaries can also influence the ductility although more experiments are needed.

Figure 3-12. Crack propagation in ODSS-316L at 400°C (a) and pores pinned by cell boundaries (b). Reprinted from Ref. 82 with permission

Table 3-3. Influence of different boundaries on strength and ductility in SLM SS316L

<table>
<thead>
<tr>
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<th>Melt pool boundary</th>
<th>Grain boundary</th>
<th>Cell boundary</th>
</tr>
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<tbody>
<tr>
<td>Strength</td>
<td>-</td>
<td>+</td>
<td>++</td>
</tr>
<tr>
<td>Ductility</td>
<td>-</td>
<td>+</td>
<td>?</td>
</tr>
</tbody>
</table>
4. Sub-grain structure in EBM SS316L

The sub-grain cellular structure in SLM SS316L was discussed in the last chapter. A similar sub-grain segregation network structure has been observed in previously prepared EBM SS316L. However, it should be noted that there is a special preheating regime in the EBM process. Prior to the real melting process, the powder bed is scanned by a defocused electron beam to generate some necking and thus increase the conductivity between powder granules. Otherwise the electrons will accumulate in the powder granules and they will repel each other. In a severe case, small particles will elevate and form a smoke in the way of the electron beam and force the build to stop. The powder bed temperature for consolidating SS316L is normally kept between 700-830°C during the whole building process. Therefore, it is reasonable to predict that the microstructure tends to change if exposed to this temperature for a long time. Small specimens, which can be finished within hours, might not be affected but larger component that takes days to build will be influenced. Therefore, it is important to explore the microstructure evolution of large components during the building process.

In this study, large tensile bars were prepared by EBM for 36 h. The microstructure is compared with that of the previously prepared small specimens. The evolution of the sub-grain structure is discussed and summarized. Severe precipitation occurs due to long time ‘annealing’ process that deteriorates the mechanical properties.

The Arcam A2 facility was used in the work reported in this chapter. Layer thickness is 100 μm. The powder bed was preheated to 820°C and was kept between 700-830°C during the whole component build. It took around 85 sec for building each layer. The whole component took around 36 hours to finish, followed by 13 hours of slow cooling to 100°C.

4.1 Microstructure

XRD result in Figure 4-1 reveals that EBM SS316L is mostly austenitic. The broader peak may be attributed to the residual stress or the lattice distortion resulted from micro-segregation. The composition after consolidation shows slight variation in Mn and Si contents while most elements keep unchanged.
Figure 4-1. XRD pattern of the precursor powder and consolidated EBM SS316L. Reprinted from Ref. 62 with permission

Table 4-1. Composition of EBM SS316L

<table>
<thead>
<tr>
<th></th>
<th>Bulk</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>Cu</th>
<th>N</th>
<th>Fe</th>
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</thead>
<tbody>
<tr>
<td>EBM</td>
<td>&lt;0.02</td>
<td>0.53</td>
<td>1.5</td>
<td>&lt;0.01</td>
<td>0.007</td>
<td>17.4</td>
<td>12.4</td>
<td>2.5</td>
<td>0.053</td>
<td>0.06</td>
<td>Bal.</td>
<td></td>
</tr>
</tbody>
</table>

Some defects in EBM SS316L are revealed by OM images in Figure 4-2. Large crescent-like pores in Figure 4-2a are believed to be formed by insufficient bonding at melt pool boundaries. This is further proven by the traces of some un-melted powder granules observed inside those pores. The large layer thickness applied to accelerate building is responsible for this insufficient melting. Despite the defects, the overall density is still 99.8%TD as calculated from images. The edge of the specimen in Figure 4-2d shows more porosity than the inner part of the specimen due to the complex thermal situation at powder-solid boundaries.
An etched surface of EBM SS316L is shown in Figure 4-3. Hierarchical structures including melt pools, grains and sub-grain structures are also expected due to the similar manufacturing process between SLM and EBM. Although some defects are seen at melt pool boundaries, an overall well overlapping between adjacent melt pools is achieved. Columnar grains growing in the building direction have a length reaching 1 mm and a width reaching 300 μm, which is much larger than those of SLM SS316L. The larger grains are attributed to the much faster scanning speed and the higher energy of the beam source.
The grain morphology and orientation are revealed more clearly in EBSD mapping, as seen in Figure 4-4. The results of columnar grain size agree well with the etched surface. Some slight orientation change inside the grains is evidenced, which indicates the presence of sub-grain structures. In agreement with the XRD results, less than 0.1% amount of ferrite phase is indexed.

![EBSD IPF mapping on the side plane (a) and the cross-section plane (b). Reprinted from Ref. 62 with permission](image)

**4.2 Sub-grain cell evolution**

A sub-grain structure is also revealed inside the grains by deep etching, as shown in Figure 4-5a. The sub-grains structure is irregular shaped and has a size range between 1-9 μm. The distinct TEM diffraction spots in Figure 4-5b was obtained from the whole area and proves that all sub-grains have the same crystallographic orientation. TEM images can reveal the details of the sub-grains more clearly. Dislocations are observed both at the sub-grain boundaries and inside the sub-grains, as seen in Figure 4-5b, c. The sub-grains kept their shape after tensile testing as shown in Figure 4-5d. However, a great amount of additional dislocations were observed induced by tensile stress.
Figure 4-5. Sub-grain structure by SEM (a) and TEM bright field imaging (b), (c) and TEM after tensile test (d). Reprinted from Ref. 62 with permission.

It is noticed that the sub-grain structure of the prepared EBM SS316L is different from previously obtained ones. The sub-grains in SLM SS316L have pentagon/hexagon cells with element segregated boundaries and cell spacing less than 1 μm. The previously prepared small EBM SS316L specimens also have the features seen in Figure 4-6a, b. The differences are attributed to the long building time (36h) in this study while normally small specimens take less than 2 h to build. The preheating regime in EBM kept the powder bed and the consolidated bulk at around 800°C for more than 36h. Therefore, the consolidated EBM SS316L, especially the volume fabricated at the beginning of the build, will experience a process similar to annealing and the diffusion of the initially segregated elements is facilitated. The dislocations will also move and at the same time the residual stress is relieved. Consequently, the previously smaller sub-grains are rearranged and merged into the larger sub-grains seen in this study. Besides the building time involved here, the cell spacing can also be controlled by the temperature gradient and the solidification rate as discussed in Chapter 2. Small sub-grain cells with thin boundaries are easily formed if the solidification rate is high. High solidification rate can be achieved by higher scanning speed. If the solidification rate decreases, the severe segregated cells with small cell spacing have time to diffuse and larger cells with thick cell boundaries are formed as seen in Figure 4-6b. The solidification rate can be slowed down by generating larger melt pool or decreasing scanning speed. Furthermore, if the preheating time (building time) is long, the already weakened element segregation will continue to diffuse until there is no element segregation at
the sub-grain boundaries, as observed in this study. Similarly, the sub-grain and grains will grow larger. The schematic drawing of the sub-grain structure evolution is summarized in Figure 4-6.

Figure 4-6. SEM and schematic drawing of the evolution of sub-grains. Reprinted from Ref. 62 with permission

4.3 Precipitation

The longer annealing time result not only in diffusion of the cell segregation, but also in precipitation. Since precipitates have never been observed in previous prepared specimens, the longer annealing time should be responsible for their occurrence. The formation mechanism should be similar to that found in traditional annealing, which is well described in literature.\textsuperscript{72} Precipitates are observed on etched surfaces, as shown in Figure 4-7a. They are present both at the grain boundaries and inside the grain. EDS mapping revealed that the precipitates are rich Cr and Mo and EDS on some precipitation sites showed 27.6 wt% Cr and 8.6 wt% Mo. Besides the random distributed precipitations, at some locations there are a concentration of lots of precipitates. Similar agglomerations of precipitates are also observed on the surface of some brittle fracture sites, as revealed by Figure 4-7c, d. This proves that these agglomerations of precipitates are detrimental at tensile stress. The same area represents also an un-sufficiently bonded material as proved by the presence of un-melted steel particles. It has also been discussed in literature that the precipitates tend to be accumulated at the defect site, \textit{e.g.} around the pores.\textsuperscript{73} An overview of the precipitates versus grain boundaries is shown in Figure 4-7. It is observed that precipitates are mostly present at the grain boundaries, which are definitely weak points under tensile stress. Some cracks are formed at the place where the grain boundaries are detached when the grains are deformed, loosened and teared apart during tensile tests. The cracks in Figure 3-8c and 3-8f have similar size and shape, which indicates they probably represent the same structure. In figure 4-7g, precipitation is directly evidenced at the grain detaching sites. In summary,
these observations proved the drawback of precipitation on the tensile properties.

Figure 4-7. EDS mapping on precipitations (a), lots of precipitates at a local site (b), fracture surface (c), brittle fracture with concentration of precipitates and un-melted powder (d), cracks at grain boundaries and detaching of grain boundaries (f), precipitation at grain boundaries before tensile test (e), after tensile test (g). Reprinted from Ref. 62 with permission.
5. Sub-grain nanoinclusions in SLM SS316L

The sub-grain cellular structure in SLM 316L increased the yield strength to 455 MPa in a small sized specimen (gauge length 10 mm, effective cross-section 1×1 mm), see Chapter 2. The sub-grain nanoinclusions are also widely known as a way to improve strength and oxide dispersion strengthening steel has been developed (ODSS).\textsuperscript{74-78} Previously, ODSS parts were fabricated by complex processes that included mechanical alloying (MA), degassing, hot extrusion, rolling and heat treatment.\textsuperscript{79, 80} The joining and machining of ODSS are also difficult due to the possible agglomeration of the fine nanoinclusions, not to mention fabricating components with complex structure.\textsuperscript{81} Therefore, it is a demanding task to develop new fabrication and joining techniques.

SLM may be a suitable preparation technique from many aspects; \textit{in-situ} formed oxide nanoinclusions and, most important, a simple fabrication process regardless of structure complexity. There are also some problems that must be solved comprising the amount, size and dispersion of nanoinclusions. The amount can be adjusted via controlling the oxygen partial pressure in the process chamber, but there exists a limit for oxygen uptake. External addition of small quantities of oxides to the precursor is another way to increase the amount of oxides. The dispersion can be achieved by the recoil effect and Marangoni convection in the melt pools. These facts explore a possibility to prepare ODSS with good mechanical behavior by SLM. In this chapter, attempts of fabricating ODSS-316L have been made by laser melting of ball-milled mixtures of 316L powder doped with Y\textsubscript{2}O\textsubscript{3} powder.

5.1 Fabrication of ODSS-316L

A schematic drawing of the process is shown in Figure 5-1a. The precursor powders are SS316L powder (Figure 5-1b) and Y\textsubscript{2}O\textsubscript{3} powder with an average size of 800 nm (Figure 5-1c). The two powders were mixed and milled by ball milling in PM 100 (Retsch, Haan, Germany) at a speed of 250 rpm for 2 h. Two batches of powder with different nominal weight ratios were prepared: 1% Y\textsubscript{2}O\textsubscript{3} (ODSS-1) and 2% Y\textsubscript{2}O\textsubscript{3} (ODSS-2). 99.5% Ethanol was used as milling agent and WC balls were used as grinding media with a ball-to-powder ratio of 5:1. The powder after ball milling is shown in Figure 5-1d, e. The steel powder is covered by a layer of Y\textsubscript{2}O\textsubscript{3} powder and micro-
sized agglomerations are also evidenced. The powder mixtures were then consolidated by SLM and the process parameters are summarized in Table 5-1. Pure steel powder is also consolidated for comparison, referred as ODSS-0. All the building processes were conducted with a 1% partial oxygen pressure in the chamber.

![Image](image.png)

**Figure 5-1. Process of preparing ODSS by SLM (a), precursor 316L powder (b) and Y2O3 powder (c), ball-milled powder (d) and (e), Printing process (f), as-build specimens (g) and size of tensile bar (h) 82. Reprinted from Ref. 82 with permission**

<table>
<thead>
<tr>
<th>Specimen</th>
<th>P, W</th>
<th>V, mm/s</th>
<th>d, mm</th>
<th>h, mm</th>
<th>W, J/mm³</th>
<th>wt% Y2O3</th>
</tr>
</thead>
<tbody>
<tr>
<td>ODSS-0</td>
<td>195</td>
<td>900</td>
<td>0.15</td>
<td>0.02</td>
<td>72.2</td>
<td>0</td>
</tr>
<tr>
<td>ODSS-1-1</td>
<td>195</td>
<td>800</td>
<td>0.08</td>
<td>0.02</td>
<td>152</td>
<td>1</td>
</tr>
<tr>
<td>ODSS-1-2</td>
<td>195</td>
<td>1200</td>
<td>0.1</td>
<td>0.02</td>
<td>81</td>
<td>1</td>
</tr>
<tr>
<td>ODSS-1-3</td>
<td>195</td>
<td>850</td>
<td>0.1</td>
<td>0.02</td>
<td>115</td>
<td>1</td>
</tr>
<tr>
<td>ODSS-1-4</td>
<td>195</td>
<td>700</td>
<td>0.1</td>
<td>0.02</td>
<td>140</td>
<td>1</td>
</tr>
<tr>
<td>ODSS-2</td>
<td>195</td>
<td>700</td>
<td>0.06</td>
<td>0.02</td>
<td>232</td>
<td>2</td>
</tr>
</tbody>
</table>

5.2 Microstructure

The density and hardness were checked first where a similar trend was observed; the hardness increases with density to a certain value and then starts to drop while the density is still increasing. The specimens of ODSS-1 are
discussed as an example here. The highest hardness 240(HV10) is achieved in ODSS-1-3 with a density of 7.93 g/mm³ while the highest density is obtained in ODSS-1-1, cf. Figure 5-2. The reason for this has been discussed previously in Chapter 3. The parameters need to be adjusted properly to achieve a combination of high density and also high mechanical properties. Pores are easily generated if insufficient energy is applied, as seen from Figure 5-3.

XRF and ICP-OES were applied to check the Y content in as-build specimens and the results are listed in Table 5-2. XRF detect signals mainly from the outmost specimen surface layer and there is a risk of losing nanoinclusions during the polishing process. Then it is reasonable that the Y quantified by XRF is lower than that quantified by ICP-OES. Anyway, it is clear some Y₂O₃ is lost when the powder mixtures were consolidated by SLM. The residual Y₂O₃ can be increased by adding more Y₂O₃ into the powder mixture.
Table 5-2. Chemical composition of ODSS-1-1 and ODSS-2 measured by XRF and ICP-OES. Reprinted from Ref. 82 with permission

<table>
<thead>
<tr>
<th></th>
<th>XRF: Y (Y₂O₃), wt%</th>
<th>ICP-OES: Y(Y₂O₃), wt%</th>
</tr>
</thead>
<tbody>
<tr>
<td>ODSS-1-1</td>
<td>0.08 (0.129)</td>
<td>0.233 (0.296)</td>
</tr>
<tr>
<td>ODSS-2</td>
<td>-</td>
<td>0.295 (0.374)</td>
</tr>
</tbody>
</table>

The absorption of the Nd:YAG laser beam is different depending on precursor materials. For example, Fe-alloy powder can absorb above 60% while ceramic powder can normally take up less than 6%. This is the reason why higher energy input is needed to consolidate Y₂O₃ doped steel powder. Since we already know the actual Y content in the consolidated bulk specimens a formula can be used to calculate the theoretical density:

\[
\rho = \frac{\rho_a \rho_b}{\rho_a \cdot b\% + \rho_b \cdot a\%}
\]

where \( \rho_a \) and \( \rho_b \) are the density of SS316L (8 g/cm³) and Y₂O₃ (5.01 g/cm³) and \( a\% \) and \( b\% \) are the weight percentage of SS316L and Y₂O₃. Based on Table 5-2, the calculated densities should be 7.986 g/cm³ and 7.982 g/cm³ for ODSS-1-1 and ODSS-2, respectively. The relative densities are thus calculated to be 99.3, 99.4 and 99.4%TD for ODSS-0, ODSS-1-1 and ODSS-2, respectively, which are almost the same value.

The hierarchical microstructure of ODSS-316L is similar to that of non-ODSS-316L. It is difficult to tell the differences of melt pool morphology and size or cell spacing between different specimen structures. It is noticed that there are many ‘black spots’ which might be nano-inclusions in Figure 5-4j-l. The amount of those black spots increases with increasing Y₂O₃ amount.
Figure 5-4. The microstructures of ODSS-0, ODSS-1-1 and ODSS-2 presented by three image columns from left to right, respectively: OM images of melt traces (a), (b), (c) on the cross-section plane and melt pools on the side plane (d), (e), (f); SEM image of etched surface revealing cells of SGCN (g), (h), (i) and finely polished surface indicating nanoinclusions (j), (k), (l). Reprinted from Ref. 82 with permission

5.3 Nanoinclusions in ODSS-316L

With the aim to identify the nature of the observed ‘black spots’, TEM images were taken from ODSS-1-1. The presence of nanoinclusions is evidenced by both bright-field and dark-field TEM images, as seen in Figure 5-5a, b. These nanoinclusions are found to be distributing randomly in the steel matrix. It is noticed that the black spots in Figure 5-5c are similar to the nanoinclusions in Figure 5-5b, which indicates the black spots in SEM image are also nanoinclusions.

The composition of these nanoinclusions was analyzed by EDS. Thus, EDS mapping in Figure 5-5e proves that the nanoinclusions are rich in Y. Also
EDS in TEM verified the nanoinclusions have different compositions, as seen in Figure 5-5f. The nanoinclusion with a darker contrast has 2.25% Y and 4.41% Si while the one with brighter contrast has 9.65% Y and 4.89% Si. It proves that the Si in steel powder and Y₂O₃ powder have reacted and they together formed nanoinclusions. Previously, Si, Cr, O rich oxides are reported to form in SLM SS316L₈₄ The size of observed nanoinclusions is summarized in Figure 5-6. Most of the nanoinclusions are smaller than 100 nm and the dominant size range is 10-70 nm. The large precursor Y₂O₃ powder was crushed into small particles in the ball milling process. The small irregular shaped particles were then melted and dispersed by the melt pool flow. It indicates that the precursor powder size is not so crucial in this process because similar particle size was obtained in previous studies using much finer ceramic powder.₅₄,₅₅
Some nanoinclusions are observed agglomerated in ODSS-1-1, as seen in Figure 5-5d. In severe cases, micro-sized agglomerations rich in Y, Si and O may occur as seen in Figure 5-7. The melt pool flow and the solidification rate can influence dispersion of nanoinclusions in the melt pools, which has been proven in preparing metal matrix composites (MMC) by SLM.\textsuperscript{85} The shape of the nanoinclusions is mostly spherical, which indicates Y\textsubscript{2}O\textsubscript{3} powder is melted in the process. The melt pool flow stirs and disperses the melted Y\textsubscript{2}O\textsubscript{3} droplets. Using suitable parameters, the large agglomeration of Y\textsubscript{2}O\textsubscript{3} can be ruptured into small pieces by the liquid capillary force, the Marangoni force and the recoil pressure. In addition, the rapid solidification rate, reaching 10\textsuperscript{6} K/s, will freeze the nanoinclusions fast enough prohibiting that they start to regroup or agglomerate.\textsuperscript{86} In summary, Y\textsubscript{2}O\textsubscript{3} can be dispersed evenly during SLM process due to the convective melt flow and rapid solidification.
A better dispersion of nanoinclusions needs strong convection and fast solidification. It has already been discussed that the faster scanning speed resulted in faster solidification due to smaller generated melt pools. But the influence of process parameters on melt pool flow is still in debate. A lower energy density weakened Marangoni convection and made the powder particles sink down and agglomerate but can also refine the TiC from coarse dendritic to nano-sized lamellar. A higher scanning speed and thicker layer (lower energy density) can increase the velocity of melt flow according to a simulation. Controversial results have been claimed in previous studies. Therefore, the effects of scanning speed on the dispersion of nanoinclusions are explored here by applying different scanning speeds (600, 800 and 1200 mm/s) to consolidate ODSS-1-1. The nanoinclusions floated and aggregated on the single melt trace at 600 mm/s, which was relieved at higher scanning speed as pointed out by arrows in Figure 5-8a-c. Insufficient convection or slower solidification at low scanning speed could both be the reasons for the particle floating. Although the re-melting of previously solidified melt pools can diminish the aggregation, it is sure that the agglomeration will be present at the uppermost layer. Two consequences are expected: loss of Y$_2$O$_3$ in the consolidated bulk and increased difficulty for even dispersion of nanoinclusions. The loss of lighter Y$_2$O$_3$ on the upper most layer is already evidenced in Figure 5-8a and Table 5-2. Aggregation in the solid bulk is demonstrated in Figure 5-8d. Higher scanning speeds at 800 or 1200 mm/s can solve this problem, but the insufficient energy input achieved with too high scanning speed may result in porosity in the bulk. The second phase nanoinclusions tend to accumulate around the pores and form particle-porosity clusters. It is also evidenced that although the floating of nanoinclusions is relieved in Figure 5-8c, a lot of particle-porosity clusters are observed in the bulk material shown in Figure 5-8e. In this study, the suitable scanning speed was found to be 800 mm/s, which avoids porosity in the bulk, minimizes the loss Y$_2$O$_3$ and achieves enough convection for even dispersion of nanoinclusions. As seen from the schematic drawing in Figure 5-9, the already aggregated Y$_2$O$_3$ in the previous layer increases the difficulty for dispersion of nanoinclusions, especially at site 1. The aggregation in a single melt pool at site 1 is observed under all scanning speeds, as seen in Figure 5-8a-c. Firstly, simulation has proved the Marangoni convection near the melt pool boundary (site 1) is weaker compared to that in the middle of the melt pool (site 2). The induced capillary force is not enough to scatter the already concentrated Y$_2$O$_3$. Secondly, the melt close to the single melt pool boundaries solidifies first and it leaves no time for the aggregation to disperse. This problem can be solved by decreasing hatch spacing to ensure enough overlapping between adjacent melt pools. The hatch spacing should be less than $l_1-2l_1$ to make sure the aggregation at site 1 disappears. This is also the reason for the smaller hatch spacing value used in ODSS-1-1 than that in ODSS-0, as listed in Table 5-1.
5.4 Mechanical properties

The mechanical properties of different ODSS are listed in Figure 5-10. The results of SLM SS316L fabricated at lower oxygen partial pressure ($\leq 100$ ppm) presented in a previous study$^{61}$ and RCC-MR criterial are also listed for comparison. It is noticed that all the ODSS has much higher yield strength than the RCC-MR criterial, which can be attributed to the sub-grain cellular structure and nanoinclusions. Several conclusions can be made:

- The amount of nanoinclusions can be increased by increasing the oxygen partial pressure in the processing chamber. Both the strength
and ductility of ODSS-0 are higher than non-ODSS. The reason has been widely discussed in previous literature. The amount of nanoinclusions can be increased by additional doping with Y$_2$O$_3$ powder. The strength and ductility are both improved by introducing more nanoinclusions. Both the strength and ductility of ODSS-1-1 are higher than those of ODSS-0. Excess amounts of added Y$_2$O$_3$ powder increase the difficulty of even dispersion of nanoinclusions. There is upper limit for the capacity of Marangoni convection and recoil force. Agglomerations in ODSS-2 (Figure 5-7) are inevitable and deteriorate the strength. However, an increase in ductility reaching 96% is observed in ODSS-2 compared to other specimens. The strengthening effect on yield strength is still active at elevated temperature until 400°C. However, the ductility of ODSS drops to the level of non-ODSS at 250°C.

A recent study indicated that TiN particles with 20-70 nm size have limited contribution to the precipitation strengthening. Further refining of the nanoinclusions are important although the yield strength obtained in this study is 100 MPa higher than that from the TiN particle study.
The fracture surfaces of ODSS-1-1 at different temperatures were examined. It is fairly clear that the ODSS-1-1 has much higher elongation before rupture at RT. Discrete small pores are observed on the fracture surface at RT while larger craters formed by coalescence of small pores are identified at elevated temperatures. It is also found that no large pore exists on the polished side surface at RT. Instead small pores, smaller than cell spacing, were observed in the whole deformation region. On the other hand, large pores were found on the necking part at elevated temperatures. The above results indicate the effect of multi-level structures in ODSS in preventing microporosity from their coalescence lost function at elevated temperatures and resulted in low ductility.

Figure 5-11. Fracture surfaces of ODSS-1-1 tested at different temperatures. Reprinted from Ref. 82 with permission
6. Application in nuclear fusion

The components in a nuclear fusion reactor have a complex design due to thermal mechanical and radiation reasons. 
Meanwhile, the criteria for nuclear fusion materials (RCC-MR code) are stricter than that of other industry. Conventional fabrication methods of complex components are complicated with several steps of assembly, pre-joining, joining and are costly, time consuming. Therefore, a new method which can simplify the manufacturing processes and also guarantee properties are highly demanded. Some agencies considered using AM due to its advantage in manufacturing parts with complex structure. The author also believes AM has broad application in nuclear fusion and apply SLM and EBM to fabricate ITER first wall panel part to demonstrate its feasibility.

6.1 Mechanical properties

The manufacturing of ITER first wall panel part was performed on Renishaw AM250 and Arcam A2. The tensile and impact properties are shown in Figure 6-1.

All the tensile tests were performed in the building direction, which normally gives the worse tensile properties of SLM materials. As explained before, SLM SS316L has much higher yield strength than conventional fabricated ones, the reason has been explained above in Chapter 3. Compared to the SLM SS316L, the strength of EBM SS316L is much lower. The elongation at rupture and the reduction of area are a bit higher. Both strength and ductility obtained from tensile tests properties degrade at ET, as expected. On the other hand, the absorbed energy increases 22% which indicates good impact toughness at ET.

The higher yield strength of EBM SS316L is again due to the strengthening effect of the sub-grain boundaries. The measured grain size of EBM SS316L in the building direction is several hundred micrometers and the calculated yield strength according to Hall-Petch law should be around 190 MPa while the result here is 253 MPa. Moreover, the observed crescent-like defects will further deteriorate the strength, especially in the building direction since the crescent is perpendicular to the building direction. The lower tensile strength of EBM SS316L is also attributed to the same reason and yield strength even lower than 190 MPa should be expected. However, the great
number of sub-grain boundaries compensates these draw backs and increase the yield strength to 253 MPa. The yield strength is better than that of HIP SS316L with the grain size in the range of 65-125μm².

![Graph showing tensile and impact properties of SLM and EBM SS316L at RT and 250°C](image)

*Figure 6-1. Tensile and impact properties of SLM and EBM SS316L at RT and 250°C. Reprinted from Ref. 24 with permission*

Typical stress strain curves of SLM and EBM SS316L are shown in Figure 6-2. The SLM SS316L shows little strain hardening while EBM SS316L shows obvious strain hardening after yielding. It has been proved from various SEM and TEM images that SLM SS316L has higher dislocation density than EBM SS316L which results from the faster solidification and no pre-heating. Therefore, there is not much space left in SLM SS316L for the increase of dislocations during plastic deformation (that causes strain hardening). This unexpectedly makes SLM SS316L highly resistant to defects. If defects are present, the experimental elongation drops much but the strength keeps almost the same value as that without defects.
It has been stated in literature that there might be no fatigue limit for FCC material and this is also observed here.\(^9\) The results in Figure 6-3 show that SLM SS316L had higher stress when the same strain amplitude was applied while EBM SS316L had prolonger fatigue life. The average elastic modulus obtained from the results are 107 GPa and 63 GPa for SLM and EBM SS316L.

The fractography images in Figure 6-4b and d indicates that the defect sites due to insufficient melting are crack initiation origins. Multiple origins sites are observed in SLM SS316L which indicates a high stress concentration, while a single origin is found in EBM SS316L.
6.2 Part printing

The CAD model is shown in Figure 6-5d, with maximum dimensional size $279 \times 163 \times 161$ mm. Complex cooling pipe systems are imbedded inside. This demo part is successfully fabricated both by SLM (Figure 6-5a) and EBM (Figure 6-5c) and summarized as follows:

1. EBM is more than 3 times faster than SLM in building this component. The building speed is decided by the layer thickness, scanning speed and hatch spacing. The inherent features makes the build efficiency of EBM much better than that of SLM.

2. The drawbacks of the EBM component are the size accuracy and surface roughness due to the larger beam spot. The measured 3-dimensional sizes of SLM part are exactly $279 \times 163 \times 161$ mm and no obvious distortion of loops are observed.

3. The residual stress in the SLM component is larger than that of the EBM component. The used preheating regime in EBM preparation can relieve the stress during printing.

4. The removing of support structures as seen in Figure 10 is the major problem for both methods. The supports are usually applied at the overhang area where the angle to the horizontal line is less than 45°, as seen in Figure 6-6. There is a risk of collapsing at the overhang if there is no support. The support structure has a frame construction and is normally easy to remove. However, if supports are built inside the complex internal pipe system, it is difficult or impossible to
remove them. For example, there are two places in this build where the support structure cannot be removed, as seen in Figure 6-2. One solution is a redesign of the pipe system to make all the overhang angles larger than 45° in one direction. Another solution is design of some necessary supports that are qualified for fluid dynamic simulation (cooling effectiveness and pressure drop) and leave them inside.

Figure 6-5. ITER first wall panel part fabricated by SLM (a), (b), by EBM (c) and CAD model (d). Reprinted from Ref. 82 with permission
Figure 6-6. Internal structure of ITER first wall panel part. Reprinted from Ref. 24 with permission.
7. Conclusion

This thesis moves one step further to understand the sub-grain structures and their influence on mechanical properties in AM SS316L. The conclusion can be drawn as follows:

1. The 3D cellular network structure is formed by micro-segregation in sub-grain growth and is influenced by the low-energy growth direction and temperature gradient direction. The cell spacing can be refined by increasing scanning speed but results in lower density. Colonies of cells crossing melt pool boundaries can strengthen the bonding between melt pools. The continuity of cells can be changed by altering scanning direction in adjacent layers.

2. Cell boundaries can improve the strength, but their influence on ductility is still unknown. At least they can constrain the size of micro-voids and prevent the coalescence of micro-voids under plastic deformation, which delays cracking. Grain boundaries have limited influence on strength but are still important for ductility. High strength and ductility SLM SS316L ($\sigma_{0.2}=552$ MPa, $\varepsilon=83\%$) can be prepared by carefully control of the grains and cells.

3. The distribution of nanoinclusions is determined by melt pool convection and solidification. Faster scanning speed can achieve more even dispersion, but may also sacrifice density. Additional Y$_2$O$_3$ can increase the amount of nanoinclusions. The capacity of melt pool convection results in inevitable loss of Y$_2$O$_3$ during SLM and limits the maximum number of external particles added. Superior properties ($\sigma_{0.2}=574$ MPa, $\varepsilon=91\%$) can be obtained in ODSS 0.3\%Y$_2$O$_3$-316L by adding 1\% Y$_2$O$_3$. The strength drops but elongation increases ($\sigma_{0.2}=553$ MPa, $\varepsilon=96\%$) in 0.37\%Y$_2$O$_3$-316L by adding 2\% Y$_2$O$_3$ due to the unavoidable agglomeration. The main challenge is the refinement of nanoinclusions into $<10$ nm level.

4. The already formed element segregated sub-grain cellular structure can be removed by the preheating regime in EBM, which results in a dramatic loss in strength. Excessive precipitations in a limited volume deteriorate ductility.

5. The strengthening effect in SLM ODSS-316L is active both at room temperature and elevated temperature up to 400 °C. However, the elongation drops dramatically from 91\% at room temperature to 29\% at an elevated temperature of 250 °C. It proves that the cellular
structure and the nanoinclusions lose their function on ductility at elevated temperatures.
Sammanfattning

Denna avhandling fokuserar på att utforska sub-korn strukturen i rostfritt stål 316L som tillverkats med additiv tillverkning (AM). Två olika pulverbäddsmetoder är inblandade: Selektiv lasersmältning (SLM) och Elektronstråle smältning (EBM). Det är känt sedan tidigare att AM 316L har heterogena egenskaper och en hierarkisk struktur: småtpoler av mikrostorlek, korn av mikrostorlek, sub-korns struktur av nanostorlek och inneslutningar av nanostorlek. Relationen mellan de här olika strukturerna och deras influenser på mekaniska egenskaper är inte helt klarlagda. Smältpols gränser har färre sub-korn segregerade nätverks strukturer (cellstrukturer) och är svagare jämfört med bulk materialet. Sub-korn gränser har mindre inflytande på styrka men är fortfarande viktiga för duktilitet. Sub-kornätverksstrukturgeränser (cellgränser) har styrkt materialet utan att det har förlorat duktilitet. Cellstrukturen kan växa igenom småtpolsgränser och lägvinkel sub-korno gränser, men inte igenom korngränser. Baserat på ovan nämnda förståelse kunde AM-processparametrar justeras för att åstadkomma anpassade mekaniska egenskaper. Omfattande karakterisering utfördes för att undersöka densitet, sammansättning, mikrostruktur, fas, magnetisk permeabilitet, dragegenskaper, Charpy slagprovs egenskaper, och utmattningsegenskaper, för båda SLM och EBM SS216L vid rumstemperatur och förhöjt temperatur (250°C och 400°C). Generelt så har SLM SS316L bättre styrka medan EBM SS316L har bättre duktilitet tack vare de olika tillverkningsmetoderna. Med 45° rotering av skanriktningen mellan varje lager, istället för 90°, kunde en bättre cell förbindelse åstadkommas. Överlägsna mekaniska egenskaper (draghållfasthet på 552 Mpa och förlägning på 83%) uppnåddes i SS316L tillverkat med 20 μm lager tjocklek och som testades i byggriktningen. "Oxide dispersed strengthening steel" (ODSS) med tillsatt Y2O3 tillverkades också med SLM för att förbättra prestandan vid förhöjd temperatur. Något förbättrad styrka och duktilitet (draghållfasthet på 574 Mpa och förlägning på 90%) kunde uppnås med 0.3%Y2O3-ODS med jämnt fördelade grova nanopartiklar (20nm). Stryrkans sjunker något medan duktiliteten sjunker dramatiskt vid förhöjd temperaturer. Fraktografiska resultat visar att koalescens av nanoskopiska tomrum förhindras vid rumstemperatur med inte vid förhöjda temperaturer vilket resulterar i låg duktilitet. De uppnådda, lovande egenskaperna hos stora AM tillverkade prover försäkrar möjlig tillämpning inom kärnfusion. För första gången var en panel för ITERs
första vägg, med komplex inre rörstruktur, tillverkad med både SLM och EBM, vilket ger ett stort förtroende till användning inom kärnfusion.
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