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In-situ micro-tensile testing of additive manufactured maraging steels in the SEM:
Influence of build orientation, thickness and roughness on the resulting mechanical properties
*La Metallurgia Italiana*, (3): 27-33

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Selective laser melting (SLM), which is one of the most widely used metal additive manufacturing (AM) techniques is capable of producing various end-use parts with complex shapes and intricate designs which can be difficult to produce with conventional methods. The SLM process uses a laser beam to melt and fuse the metal powder through layer by layer approach to build 3-D parts directly from a CAD design [1]. The SLM method, generally characterized by high cooling rates and high thermal gradients, results in non-equilibrium or fine microstructures [2]. This can be a limiting factor in case of brittle materials which cannot resist high internal thermal stresses and lead to quench cracking. The rapid cooling makes SLM process to be used in inert or controlled atmosphere, such as nitrogen or argon, to avoid oxidation and to reduce the contamination from the atmosphere. Usage of inert or controlled atmospheres can also create problems such as entrapment of gas bubbles, formation and entrapment of inclusions which ultimately lead to porosity, unintended impurities and even delamination between layers [3, 4]. The surface roughness is another limiting factor in terms of surface quality especially in the production of thin sections [5]. The poor surface quality is due to surface porosity, sticking of the powder particles to the edge of the thin sections and waviness of solidified built-up layers during SLM. Previous studies on the surface quality and roughness show that a careful selection of the size distribution of the powder particles as well as laser parameters can improve the surface quality [5]. The presence of porosity and impurities, build orientation, layer thickness and the interface between the built-up layers as well as the surface quality are main contributing factors when considering the tensile strength and elongation at break for the
sections below 1 mm thickness. These factors can be controlled by careful and optimized selection of SLM process parameters, and a good design approach is necessary in order to produce good quality thin sections [6]. It is necessary to study the mechanical properties of thinner sections when strength is the main design parameter. The tensile properties of the sections with the thickness lower than 2 mm have to be carried out by micro-tensile testing. Majority of mechanical properties of maraging steel studies were focused on bulk samples. So the aim of the present work is to study the influence of surface roughness, built orientation and thickness (≤ 1 mm) on the tensile strength as well as to compare with the bulk tensile properties.

**Experimental method**

The material used for this study is maraging steel which is characterized by good tensile strength, high toughness and good weldability. Maraging steels are mainly used in different tooling applications. The material is well suited for additive manufacturing due to its good weldability and resistance to cracking when subjected to rapid cooling and heating [7, 8].

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<tbody>
<tr>
<td>Fe</td>
<td>Ni</td>
<td>Co</td>
<td>Mo</td>
<td>Ti</td>
<td>Al</td>
<td>Cr, Cu</td>
<td>C</td>
</tr>
<tr>
<td>Bal.</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>each ≤ 0.5</td>
<td>≤ 0.03</td>
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<tr>
<td>Mn, Si</td>
<td>each ≤ 0.1</td>
<td>P, S</td>
<td>each ≤ 0.01</td>
<td></td>
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Tab. 1 - The material composition in wt. % of MS1 [9]

The maraging steel powder with a commercial name EOS Maraging Steel MS1 (equivalent to 1.2709 alloy composition according to European standard) was used for this study and the nominal composition of MS1 is given in Table 1 [9]. All samples were build using a selective laser melting machine EOS M290 equipped with 400 W Ytterbium fiber laser source working under nitrogen atmosphere. A 40 µm build layer thickness was used for all samples. Bulk tensile test bars were prepared from the selective laser melted build blocks according to DIN EN ISO 68921. The bulk tensile tests and micro-tensile tests were carried out in as-built condition (without any heat treatment) at room temperature. Microtest 5000 N tensile stage from Gatan Inc., with the capability to record stress-strain curves from the Microtest software was used for the micro-tensile testing. This microtest module from Gatan offers in-situ tensile tests of the specimens inside the scanning electron microscope (SEM). The micro-tensile tests were performed on as-received rough samples outside of the SEM and also on polished test samples inside the SEM with the motor speed of 0.1 mm/min. The force and elongation data were collected during the micro-tensile testing and, SEM images were continuously captured from the surface of the polished sample by continuous scanning during the in-situ testing to study the plastic deformation.

In order to study the mechanical properties of thin plates and to compare them with the bulk material properties, square sections (20 x 20 mm²) in parallel and perpendicular directions to the built orientation, and plates with 0.5 mm, 0.8 mm and 1 mm thickness were built. The micro-tensile test samples as shown in figure 1 were prepared using water jet cutting in horizontal and vertical directions to the build orientation. The polishing of micro-tensile test samples was carried out by standard grinding and polishing procedures. The surface topography of the as-built and polished samples was characterized using Wyko NT9100 optical profilometer. The 3-D surface roughness parameters were measured at four different areas (3 x 6 mm²) on as-built and polished samples. These parameters provide roughness, spatial and hybrid information for 3-D surfaces. The microstructure of the samples was characterized by optical, stereo and scanning electron microscope. SEM-EDS (energy dispersive spectroscopy) was used to analyze the composition of the material and inclusions. Light optical microscopy was carried out using Leica DMRME, stereo microscopy was carried out using Leica MZ16 and SEM was carried out on rough, polished and fractured samples using Zeiss Ultra 55 FEG-SEM equipped with an Oxford Instruments INCA energy dispersive X-ray spectroscopy (EDS) system.
Results and discussion

Figure 1 shows the schematic diagram of the build plate showing the build orientation and the direction of micro-tensile test samples, and optical and stereo images of side, top and front views in order to understand the build orientation. Table 2 shows the average 3-D surface roughness parameters (S parameters) for as-built samples where Sa is the average surface roughness, Sq is the root mean square roughness and Sz is the Ten Point Height over the complete 3-D surface. The S parameters show a slight increasing trend with increasing thickness. Figure 2 shows the surface roughness contour plots of as-received and polished samples of 0.5 mm thick micro-tensile samples in horizontal direction to build orientation. The roughness map of as-received sample clearly reveals the horizontal build orientation and the polished sample shows uniform and smooth surface with 3-D surface roughness, Sa approximately 1 µm. However, it can be seen that there are bright elongated regions along the build orientation representing the porosity and surface defects (figure 1 – front view).

<table>
<thead>
<tr>
<th>Thickness</th>
<th>Sa (µm)</th>
<th>Sq (µm)</th>
<th>Sz (µm)</th>
</tr>
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<tbody>
<tr>
<td>0.5 mm</td>
<td>5.2 ± 0.99</td>
<td>6.98 ± 1.45</td>
<td>63.43 ± 18.8</td>
</tr>
<tr>
<td>0.8 mm</td>
<td>5.78 ± 0.72</td>
<td>7.69 ± 0.85</td>
<td>65.35 ± 4.97</td>
</tr>
<tr>
<td>1 mm</td>
<td>5.89 ± 0.94</td>
<td>7.84 ± 1.29</td>
<td>77.66 ± 8.55</td>
</tr>
</tbody>
</table>

Figures 3(a) and 3(b) show the SEM secondary electron and back scattered images of the unpolished sample respectively. These images show clearly that the top surface of the as-built sample is characterized by built layers (blue arrow), partially molten powder particles adhered to the surface (red arrow) and dark regions in between the built layers (black arrow). The dark regions were characterized with SEM-EDS and was found to be rich in Ti, Al and O, preferably titanium oxide and alumina or combination of both as shown by previous studies [4]. The oxides were deposited generally in between the layers as they float to the top of the molten pool during the SLM process due to lower density [5]. The entrapment of slag or gas within the layer may sometimes lead to weak metallurgical bonding between the built-up layers (blue arrow). Large pores or inclusions on the top of the sample as shown in the figure 1 (front view).
can be observed on the polished surface. The roughness of the unpolished samples is mainly due to the surface porosity, partially adhered powder particles and the relatively rough solidified built-up layers. Polishing, removing the top 50-60 μm, will remove the adhered particles can obtain 3-D surface roughness Sa ~1 μm but reveal the surface porosity. Previous studies on the surface roughness of SLM parts reveal that the surface quality can be increased by selecting smaller layer thickness [5, 10].

Table 3 presents the in-situ SEM micro-tensile test results for the polished samples of 0.5, 0.8 and 1 mm thickness prepared in horizontal and vertical directions to the build orientation. Figure 4(a) shows the force-elongation plots and figure 4(b) shows the engineering stress-strain diagrams for polished 0.5, 0.8 and 1 mm thickness samples. It is difficult to accurately obtain the stress strain data for the as-built rough samples due to pronounced surface roughness. As can be seen, the ultimate tensile strength (Rm) and the plastic strain at fracture of 0.8 and 1 mm samples are higher as compared to the 0.5 mm thick samples. The horizontal samples exhibit slightly higher elongation at break as compared to vertical samples. Previous studies also revealed the tensile strength of maraging steel in the range of 1100 to 1200 MPa with the ductility of about 8 to 12 % [7, 11, 12]. However micro-tensile test results reveal higher engineering strain values. This is due to the calculation of engineering strain is based on the gauge length of about 5 mm and the elongation during the test was obtained from the distance between the holding pins which are about 26 mm.

**Table 3 - In-situ SEM micro-tensile test results of polished samples**

<table>
<thead>
<tr>
<th>Direction</th>
<th>Horizontal</th>
<th>Vertical</th>
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</thead>
<tbody>
<tr>
<td>thickness</td>
<td>Rm, MPa</td>
<td>Eng. strain, %</td>
</tr>
<tr>
<td>0.5 mm</td>
<td>1088</td>
<td>20</td>
</tr>
<tr>
<td>0.8 mm</td>
<td>1148</td>
<td>27</td>
</tr>
<tr>
<td>1 mm</td>
<td>1185</td>
<td>29</td>
</tr>
</tbody>
</table>

**Fig. 3 - SEM images ((a) secondary electron mode and (b) backscattered electron mode) of 1 mm thick sample**
Additive manufacturing

Figure 4 shows the force-elongation curves of 0.5, 0.8 and 1 mm thick polished samples (a) and engineering stress-strain curves of 0.5 and 1 mm thick polished samples (b) obtained during in-situ SEM micro-tensile testing.

Figure 5 shows the SEM micrographs of 0.5 mm thick polished samples taken during the in-situ micro-tensile testing at various plastic strains after necking. As can be seen, there is a clear necking in both samples resulting in a final fracture at about 60° angle to the tensile direction. The bottom images in figure 5 reveal the presence of large pores and cracks at the edges at 18 % of strain just before the fracture. Pores or impurities in between the built-up layers can open up and extend as large pores when the microstructure is exposed to high uniaxial deformation. The lower elongation values for the vertical samples can be connected to the presence of pores and impurities along the build lines which are perpendicular to the tensile direction.

Figure 6 shows SEM images of fracture surfaces after micro-tensile testing of 0.5 mm polished samples. The fracture surfaces of horizontal and vertical build samples show large voids including pores which were resulted from the entrapped gas and the impurities due to slag formation. In addition to the defects, large solid area reveals ductile type fracture. The presence of a high number of pores can be detrimental for the mechanical properties such as fatigue resistance and the tensile strength. Thus the lower tensile strength for 0.5 mm samples as compared to 1 mm samples (figure 4(b)) is due to low amount of solid contact area and the presence of large and high number of pores in the cross section. When the thickness is reduced below 0.8 mm, the amount of porosity and inclusions play major role in strength. The strength and plastic strain at failure sharply decrease with the reduction of thickness below 0.8 mm [13]. Thus, the density and the porosity is the main contributing factors in the strength of thin sections produced in SLM process [12].
Additive manufacturing

The main advantage of micro-tensile testing is the possibility to study the deformation mechanisms of the as-built microstructures and the influence of porosity. Figure 7 shows a surface defect before and after micro-tensile testing observed on a horizontal sample in the SEM. It shows large oxide inclusion on the surface and can be observed that several cracks were initiated and extended after the micro-tensile testing. This type of surface defects can be detrimental for fatigue strength.

Fig. 6 - SEM fracture micrographs of 0.5 mm polished horizontal direction samples (a) and (b), and vertical direction samples (c) and (d)

Fig. 7 - Influence of surface defect and inclusions on the deformation behavior during the micro-tensile test
Additive manufacturing

Bulk tensile test results presented in table 4 reveal the ultimate tensile strength is about 1200 MPa for samples built in parallel and perpendicular direction. However, parallel built samples exhibits higher elongation values as compared to perpendicular built samples similar to the micro-tensile test results. The yield strength (Y.S) is higher in perpendicular built samples as compared to the parallel built samples. The ultimate tensile strength of 1 mm thick samples is closer to the bulk tensile strength. The tensile strength is lower when the thickness is less than 1 mm which can be connected to the amount and size of the porosity and impurities.

Conclusions
In this study, plates of various thicknesses (0.5, 0.8 and 1 mm) were prepared from EOS maraging steel MS1 using SLM method to study the influence of thickness, built orientation and surface quality on tensile properties. The results were compared with bulk tensile properties obtained from bulk tensile bars. The 3-D surface analysis results show a slight increasing trend with increasing thickness of the samples. The as-built surface is characterized by rough surface due to surface porosity, built-up layers, partial sticking of powder particles and high quantity of slag in between the built-up layers. Polishing 50 to 60 µm can remove the surface defects and can obtain polished surface with approximately 1 µm average 3-D surface roughness (Sa). The in-situ micro-tensile testing can be used to obtain detailed information of the deformation mechanism of AM samples as well as to study the mechanical properties of micro-tensile test samples (< 2 mm). The micro-tensile test results show that the ultimate tensile strength of thin samples depends on the porosity and the inclusions. The tensile strength values are lower for 0.5 mm thick samples as compared to 1 mm samples due to presence of large and high amount of porosity. Bulk tensile strength are comparable to 1 mm thick samples. Higher elongation was observed in the parallel build samples in both bulk and micro-tensile test samples.

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REFERENCE