# Selective laser melting manufacturing of stainless steels: heat treatment effect on microstructure and hardness of maraging steels

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Laser Powder Bed Fusion (L-PBF) Selective Laser Melting (SLM) is a widespread additive manufacturing technology in industrial applications, for metal components manufacturing. Maraging steel is a special class of Fe-Ni alloys, typically used in the aerospace and tooling sectors due to their good combination of mechanical strength and toughness. This work reports about the heat treatment effect on the microstructure and hardness value of 300-grade maraging steel manufactured by the L-PBF SLM process. The considered heat treatment consists of a solution annealing treatment followed by quenching and ageing hardening treatment. The effect of ageing temperature is reported, in a wide temperature range. Results show that solution annealing treatment fully dissolves the solidification structure caused by the SLM process. Moreover, the ageing hardening treatment has a significant impact on the hardness, hence on strength, of SLM maraging steel. The optimal ageing conditions for the SLM maraging steel are identified and reported: in particular, results show that the hardness of 583 HV is achieved following ageing treatment at 490 °C for 6 hours. A higher treatment temperature leads to over-ageing resulting in a decrease of hardness. Conversely, an excessive ageing time does not seem to affect the hardness value, for the ageing temperature of 490 °C.

**KEYWORDS:** MARAGING STEEL, 300-GRADE, SELECTIVE LASER MELTING, LASER POWER, SCANNING SPEED, RELATIVE DENSITY, CARBIDES PRECIPITATION;

## INTRODUCTION

Additive Manufacturing (AM), also known as 3D-Printing, is an emerging technology, in the spotlight for its unique capability to produce near-net-shape components, even geometrically complex, without part-specific tooling needed. Moreover, it is particularly suited for small batches production and part-customization: this is why it first emerged as a rapid prototyping technology. The adoption of AM technologies resulted in new production paradigm [1], where the designer can project a new component, or optimize the geometry of an alreadyexisting one, according to its service condition, free from production related constraints (e.g. undercuts, straight cuts, internal ducts with sharp edges). At the same time, AM made possible to simplify components assembly, merging different parts in one single monolith: most emblematic example being the fuel nozzle showed in [2], that passed from being an assembly of 20 parts to a single unit, allowing for a 25% weight reduction [3,4].

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Today the term Additive Manufacturing refers to a range of technologies, making possible to process several classes of materials – polymers, metals, ceramics, composites and sand – in different delivery condition – i.e. liquid, powder, wire or sheets. In particular, a wider AM adoption happened thanks to the gained possibility to process metal alloys with mechanical properties comparable to equivalent wrought alloys. Since 2000, the AM technology is assisting to a fast acceleration [5] due to the degree of development gained in several sectors, the most relevant being lasers, computers, CAD technologies, Programmable Logic Controllers (PLCs) and data storage systems [6]. The first commercially available AM system, back to 1987, is SLA-1 by 3D Systems and it is based on the stereolithography technique: the desired piece is obtained through the superimposition of thin layers of ultraviolet light-sensitive liquid polymer solidified by a ultraviolet (UV) laser source. Growing interest in the field led several companies and researchers working contemporarily to develop systems capable to handle metal alloys. EOS Gmbh presented its first prototype - EOS M160 - for metal processing in 1994 and, the following year, EOS M250 system was launched on the market. In the meanwhile, Deckard filed a patent [7] concerning an apparatus capable to sinter powders thanks to a laser source. The cited manufacturing devices are the precursors of modern Laser Powder Bed Fusion (LPBF) technology (also known as Selective Laser Melting SLM, Direct Metal Laser Sintering or Laser Cusing) which, among Additive Manufacturing, is the most relevant for mechanical components production. The recent spreading of International Standards regarding metal alloys LPBF-manufactured reflects this condition.

LPBF is a layer-wise production technology accomplishing material consolidation through a highly focused laser source: the spot of the laser, impinging on the powder bed, releases a quantity of energy necessary to melt metal particles.

The success of LPBF lies in its:

- capability to obtain the best geometrical and dimensional tolerances among AM
- capability to obtain near-net-shape components
- capability to produce components characterized by relative densities, with respect to wrought or forged metals, up to 99.9%
- possibility to process a wide range of materials, and, in particular, various metal alloys.

Focusing on metal alloys, it is in principle possible to manufacture every weldable metal alloy, the proper setting of working parameters is required material by material (e.g. laser power, layer thickness, gas fluxing). Working parameters identification and validation on manufactured components is the real know-how of LPBF systems producers and technology developers. Today, the most established and LPBF-verified alloys comprehend: Aluminum alloys (AlSi10Mg, AlSi7Mg0.6), Cobalt alloys (CoCrMo), Nickel alloys (Haynes HX, Inconel 625, Inconel 718), Iron alloys (Maraging steels, AISI 304, AISI 3016L, Tool steels), Titanium alloys (Ti6Al4V, Ti6Al4V ELI, CP-Titanium Grade 2).

Stainless Steels are nowadays diffused in almost every application field, thanks to their peculiar combination of properties namely, strength, corrosion resistance and relative low cost that made its fortune since its discovery in the early 19th century [8,9]. In particular, following their good strength/ductility combination coupled with their excellent corrosion resistance stainless steels are adopted in many applications including automotive [10], construction and building [11], energy [12,13], aeronautical [14], medical [15] and food [16]. The implementation of stainless steel grades in LPBF systems, together with a deeper understanding of the technology, could definitely result in a wide adoption of the technology itself. Among stainless steels, maraging ones are quite promising following their high mechanical characteristics combined to ductility and toughness. Such combination allows to maraging steels to be widely used in aerospace and aircraft sectors and in application in which is required high precision, as gears and molds [17-20]. Maraging steels represents a category of steels [21], with a high content of elements as Ni, Mo, Co, Ti and Al [22,23]; the low carbon and the high Ni content, combined with a specific cooling process (from a solid solution of γ-Fe), promotes a soft martensite microstructure, highly dislocated. The combination of strengthening and toughness is allowed by the aging treatment [24], an heat process occurring in a range temperatures between 400 °C - 700 °C which allows the precipitation of nano sized intermetallic compounds [25, 26] (such as $Fe_2Mo$, NiAl, $Ni_3$(Ti, Al, Mo), Ni(Al, Fe), etc.). It is precisely the formation of precipitates, following this heat treatment, which guarantees the inhibition of the dislocation motion [27] and, consequently, the improvement of the mechanical characteristics. The aging treatment, referring to maraging steels, represents the last step of a production process characterized by casting, plastic deformation, solution annealing, quenching and machining [28]. Thanks to the low carbon content and to the characteristic of good ductility, maraging steels can be produced through additive manufacturing (AM) technologies [29] the most widely used method is SLM [30-32]. The low carbon content in maraging steel, allow to this class of material to be very usefull, as this characteristic prevent the formation of cracks during rapid cooling (this allows to avoid carbides or carbon segregation phenomena). Many efforts are focused to optimize the parameters of solution annealing and aging heat treatment [33], with the aim to obtain the best conditions of mechanical properties, required by aerospace or tool-manufacturing industries to produce complex geometries by SLM technology [34]. The maraging steels structure deeply depends on scanning strategy and on laser source settings, which affect the relative density and the performance of compounds (e.g., [35-39]). Yongqiang Yang et al. [40] investigated the effect of laser powder, scanning speed and scanning space on the relative density of maraging steel 300; they studied the best configuration in terms of laser power and scanning speed, to obtain a relative density higher than 99%. Igor Yadroitsev et al. [41] studied the influence of laser power, scanning speed and layer thickness on residual stresses, distortions and achievable density for maraging 300-steel, in order to achieve the optimum input parameter combinations. Hanzl et al. [42] evaluated no differences between laser power and scanning speed on the impact on the mechanical and physical properties of SLM manufactured parts. The aim of this work is analyzed the effect of aging heat treatment, in terms of temperature-time combination, on microstructure and mechanical features of 18Ni 300-grade maraging steel based on the SLM technology. The as-built and the solution annealed sample were also compared with the material after aging treatment, in order to show the differences in terms of mechanical behavior.

## MATERIAL AND METHODS

Test samples were produced through SLM technology (Model EosM290), with a laser power of 400 W and an high scanning speed up to 7.0 m/s (23 ft./sec). The layer thickness was 0.05 mm and the plate form temperature was kept at 40 °C, moreover the machinery was equipped whit a precision optics F-theta lens Yb fibre laser with a nominal diameter of 100 μm (0.004 in). For the manufacturing process was used a 300-grade maraging steel powder, produced by gas-atomization and with a nominal chemical composition (wt.%) showed in Tab. 1.

**Tab.1** - 300-grade maraging steel powder chemical composition (wt.%).

| Element | Content, wt.% |
|---|---|
| Fe | To balance |
| Ni | 17.00 – 19.00 |
| Co | 8.50 – 9.50 |
| Mo | 4.50 – 5.20 |
| Ti | 0.60 – 0.80 |
| Al | 0.05 – 0.15 |
| Cr | 0.50 |
| Cu | 0.50 |
| C | 0.03 |
| Mn | 0.10 |
| Si | 0.10 |
| P | 0.01 |
| S | 0.01 |

The test samples were machined along a plane parallel to the build direction (BD), polished and etched with a solution of 2 % Nital. An optical microscope (OM) (Eclipse LV150NL, Nikon) and a high-resolution scanning electron microscope (SEM) (FE-SEM Zeiss, Gemini Supra 25) were used to analyze the microstructure of 300-grade maraging steel. The solution annealing and quenching treatment (SAT + Q), follows the manufacturing process of maraging steel samples and, the time-temperature conditions have been chosen to obtain the highest homogenization of the composition and to dissolve the columnar microstructure and the micro-segregation, typically induced by AM process. The SAT + Q was performed according to the heating profile showed in Fig. 1; then the effect of aging hardening treatment (AHT) was investigated for temperature between 450 °C and 550 °C in a time range of 6 - 24 h (heat treatment parameters in Tab. 2).

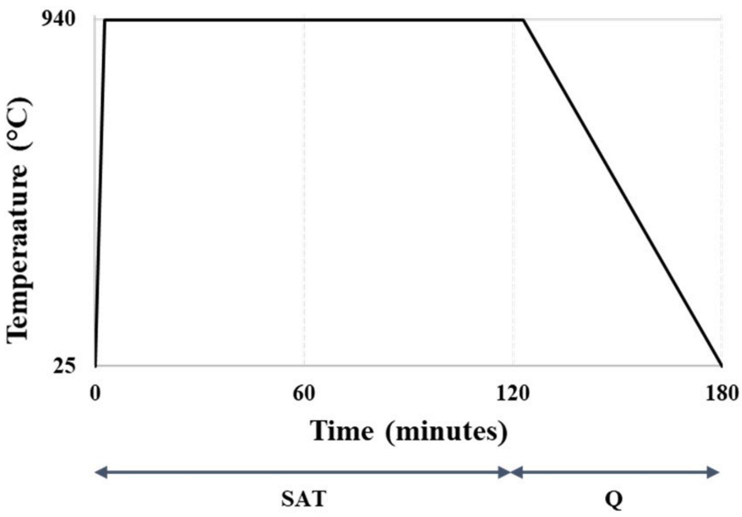


**Fig.1** - Heat profile (SAT + Q).

**Tab.2** - Heat treatment scheme carried out on 300-grade maraging steel based on L-PBF process.

| Experiment No. | SAT + Q | | | AHT | |
|---|---|---|---|---|---|
| | Temperature (°C) | SAT holding time (hours) | Cooling rate (°C/minutes) | Temperature (°C) | Aging time (hours) |
| 1 | 940 | 2 | 16 | 450 | 6 |
| 2 | 940 | 2 | 16 | 470 | 6 |
| 3 | 940 | 2 | 16 | 490 | 6 |
| 4 | 940 | 2 | 16 | 490 | 10 |
| 5 | 940 | 2 | 16 | 490 | 24 |
| 6 | 940 | 2 | 16 | 510 | 6 |
| 7 | 940 | 2 | 16 | 530 | 6 |
| 8 | 940 | 2 | 16 | 550 | 6 |

The morphology of particle (Fig. 2) was analyzed by the high-resolution scanning electron microscope. The morphology of the powders was generally spherical and some particles have satellites; moreover a fraction of elliptic shape particles can be observed. The average size of maraging steel powders is > 63 µm.

The microstructural analysis was carried out for different states of material, as built, after solution annealing and quenching and after the various time-temperature combination of the aging treatment. During the analysis, were also measured the values of hardness ($HV_{10}$) to estimate the best condition of AHT.

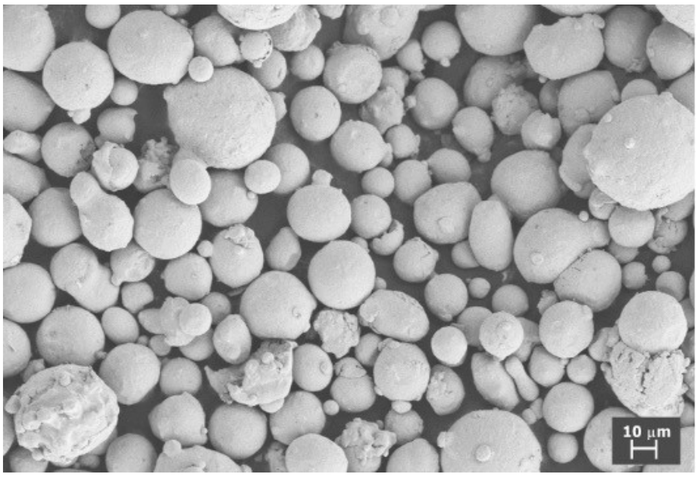


**Fig.2** - SEM image of morphology of 300-grade maraging steel powder.

**RESULTS AND DISCUSSION**

**The result of SAT + Q on SLM maraging steel**

The images in Fig. 3 and Fig. 4 show, respectively, the microstructure at low and high magnification (OM and SEM analysis), of the as-built sample along the vertical plane. The measured hardness value of the as-built sample is 384 HV. The solidified structure is composed by columnar and cellular morphology (red and black arrows respectively), and the melt pools are located by red dotted lines. Moreover through red dotted line, the laser track on etched samples are clearly visible. The cellular structure is dominant in morphology, mainly for the fast cooling rate within the melt pool during the SLM process.

The OM and SEM images after SAT + Q, showed a structure seriously changed (Fig. 5 and Fig. 6) if compared with the as-built material: the laser traces disappear completely and the cellular and columnar structure have been replaced by large lath of martensite. After the solution annealing treatment and quenching, the hardness of sample results 312 HV.


**Fig.3** - OM image of 300-grade maraging steel as-built sample. The red dotted lines delimitate the of the melt pools.

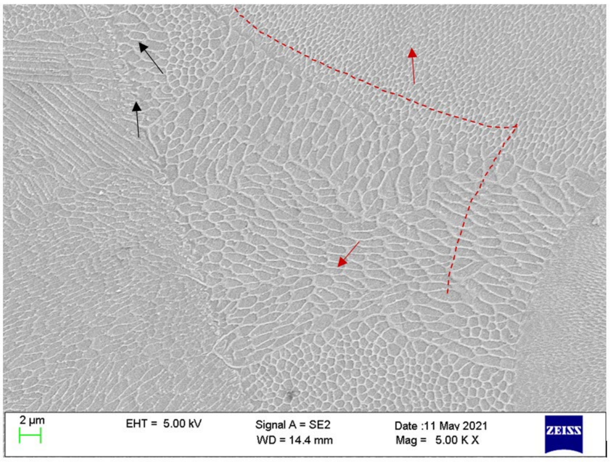


**Fig.4** - SEM image of 300-grade maraging steel as-built sample. The columnar and cellular solidification structures are clearly visible.

**The effect of AHT on SLM maraging steel**

The aging hardness treatment effect have been studied for six different temperatures (450 °C – 470 °C – 490 °C – 510 °C – 530 °C – 550 °C) and an aging time of 6 hours; the treatment followed the solution annealing and quenching treatment. The Fig. 7 shows clearly the hardness decreasing trend in relation with the increasing of temperature, above 490 °C. For high temperature the over aging phenomena occurs, and leads to the coarsening of intermetallic compounds and the reconversion of the metastable martensite into austenite being [43]. In addition to this, an excessive long treatment time leads to the same trend with a decrease of mechanical characteristics, in terms of strength, on maraging steel. The best solution corresponds to the temperature of 490 °C and the aging time of 6 hours. For higher temperatures as 550 °C and same aging time (6 h), the hardness decreases from 583 HV to 532 HV, compared with 490 °C. The best configuration of AHT, corresponding to the temperature of 490 °C and the aging time of 6 hours. This condition has been deeply investigated and the Fig. 8 shows that at 490 °C the treatment time is not relevant as the heat treatment temperature. The aging time seems to not be affective for the characteristic of hardness, in particular after 24 h of treatment the hardness is 590 HV, against 583 HV for 6 h. Fig. 9 and Fig. 10 show, respectively, the SEM images relating to the best (490 °C, 6 hours) and the worst (550°C, 6 hours) aging treatment conditions, in terms of hardness.

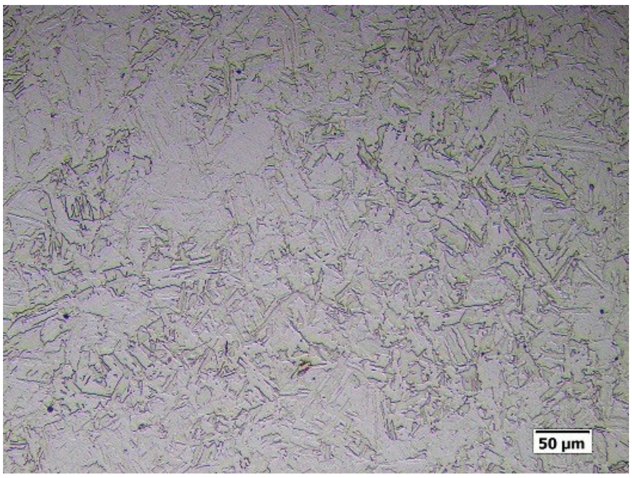


**Fig.5** - OM image of 300-grade maraging steel after SAT + Q heat treatment.

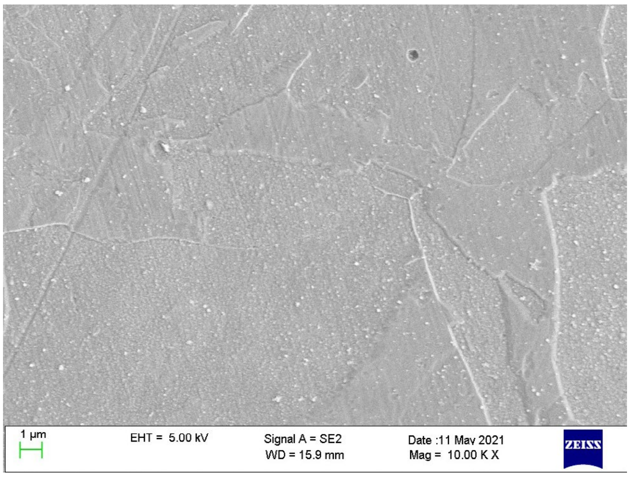


**Fig.6** - SEM image of 300-grade maraging steel after SAT + Q heat treatment.

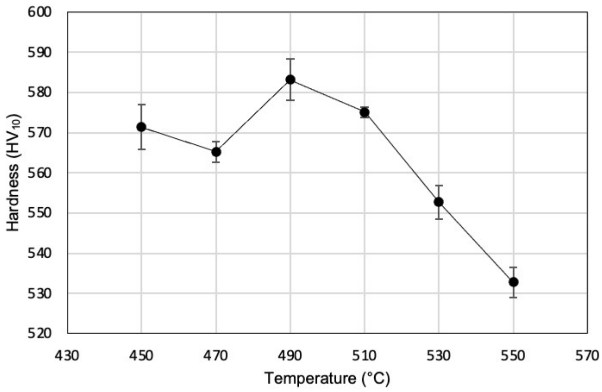


**Fig.7** - Values of hardness ($HV_{10}$) in relation with temperature (aging time: 6 hours).

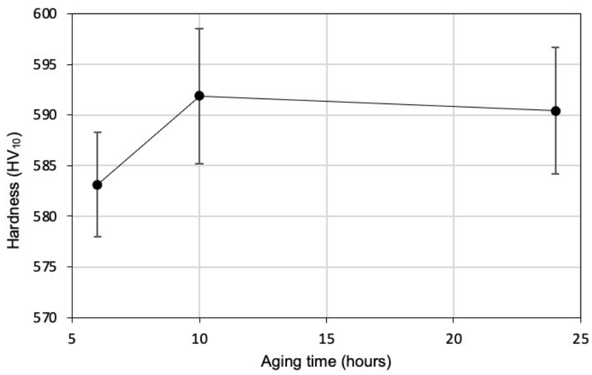


**Fig.8** - Effect of aging time on hardness (treatment temperature: 490 °C).

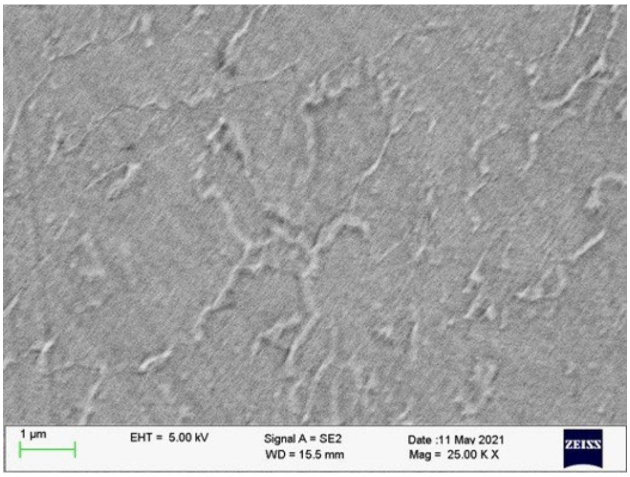


**Fig.9** - SEM image referred to the best condition of AHT (490°C, 6 h).

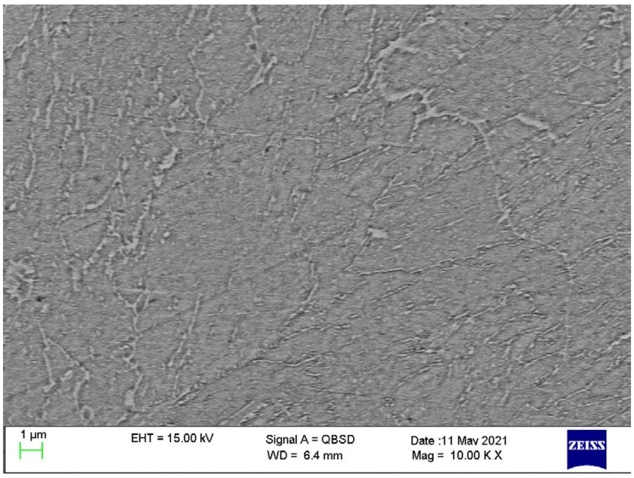


**Fig.10** - SEM image referred to the worst condition of AHT (550 °C, 6 h).

## CONCLUSIONS

In this work, the solution annealing treatment at 940 °C for 2 hours and quenching effect on 300-grade maraging steel, manufactured by SLM process, was investigated. Moreover, the effect of a following aging hardness treatment was reported for different times and temperatures conditions, in the time range of 6 h- 24 h and temperature range of 490 °C - 550 °C.

The main conclusions can be listed:

1. The original microstructure of 300-grade maraging steel, manufactured by SLM process, is characterize mainly by cellular and some areas of columnar solidification structure;
2. the solution annealing at 940 °C for 6 h and quenching, replace completely the solidification structure and the laser trace boundaries, with lath martensite microstructure. The hardness decreased from 384 HV to 312 HV after solution annealing treatment;
3. The best aging hardness treatment condition was in correspondence of temperature of 490 °C and treatment time of 6 h; the hardness measured was 583 HV. Higher temperatures led to a decrease of hardness, due to physical phenomena such as coarsened of intermetallic compounds and reversed austenite. Treatment temperature of 550 °C led to a hardness value of 530 HV;
4. The aging time seems to be not affective on 300-grade maraging steel produced with SLM technology. The difference between 6 hours and 24 hours of treatment was about 10 HV.

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