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# Advanced techniques applied to analyse the influence of the heat treatment on the mechanical properties, microstructure and texture of additively produced components from maraging steel

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**Abstract.** The experimental program Metallic materials in the process chain of additive manufacturing is focused on the characterization of laser powder bed fused materials (L-PBF). Metallographic analysis, electron backscatter diffraction supported by micro tensile testing were used to analyse the material properties of the maraging steel MS1. The main goal of the research was to use advanced techniques to investigate the influence of the additive manufacturing with very fine powders, and subsequent heat treatment on the microstructure, texture and mechanical properties of the components from maraging steel.

## 1 Introduction

In the case of additive manufacturing (AM) production, the component is formed by the gradual application of thin layers of metal powder, which are locally melted by a laser beam. Due to the gradual formation of the component layer by layer and high solidification rates, different microstructures, local residual stresses, or the formation of pores and many other phenomena may occur. Due to the large number of influencing factors and relative novelty of AM technologies based on powder bed fusion, there are numerous areas where the relationships between the parameters of AM processes, materials, and created microstructures are not completely explored.

Maraging steel MS1 (1.2709), officially named LaserForm Maraging Steel (B) by 3D SYSTEMS, is a genuine tool steel fine-tuned for the use with ProX® DMP 200 metal printers. It is easily heat-treatable in a simple age-hardening process resulting in excellent hardness and high - strength, with good weldability and machinability [1]. Martensite in alloys of low-carbon Fe-Ni-C system with high nickel content is plastic and tough. Martensite precipitation hardening is promoted by a fine dispersion of intermetallic phases, which can be simple or complex compounds (e.g. Ni<sub>3</sub>(Ti, Al, Mo), Fe<sub>2</sub>Mo, Ni(Al, Fe)). Precipitation hardening takes place at a temperature of 450 to 500 °C for 3 to 6 hours. A strength limit of 1400 to 2140 MPa with high toughness and contraction (50 to 70 %) can be achieved with such processing [2], [3].



## 2 Materials and Methods

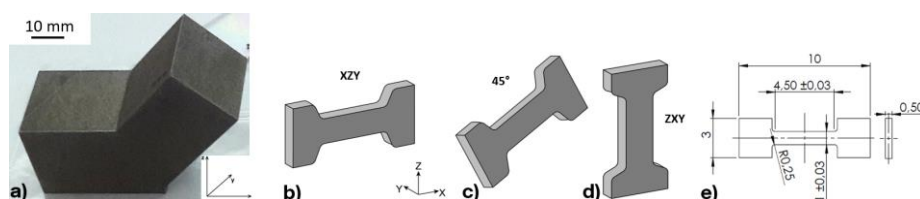
Maraging steel 1.2709 supplied by 3D SYSTEMS as a gas-atomized powder was used as the experimental material. Chemical composition supplied by the powder manufacturer is summarized in Table 1. Powder analysis obtained by laser diffraction on a HELOS/BR instrument (Sympatec GmbH) proved probability density function of the particle size distribution  $x_{90} = 20.6 \mu\text{m}$ . The parts were manufactured with standard parameters on a ProX® DMP 200 3D SYSTEMS metal printer with laser speed 1200 mm/s at a laser power 135 W and layer thickness 30  $\mu\text{m}$ . This range of 3D metal printers works with very fine powder fractions.

Experimental material was analysed by metallographic analysis and mechanical testing. The experiment was performed in as-built, solution annealed and precipitation hardened states. The heat treatment conditions are summarized in Table 2. Metallographic specimens were prepared by a standard procedure of grinding and subsequent polishing. The microstructures were revealed by etching in Nital solution and photographed using the light microscope NIKON ECLIPSE MA200 equipped with a NIS Elements 5.2 digital image processing and analysis software. Detailed microstructural observation and electron backscatter diffraction (EBSD) analysis were performed on a scanning electron microscope JEOL IT 500 HR. Electron backscatter diffraction, a unique tool for assessing the microstructure and texture of a material, provided detailed information about the additively manufactured samples. Observation was performed under the backscattered electrons with EDAX Hikari Super camera. EBSD analysis was performed at a step size of 0.1  $\mu\text{m}$ , size of the analysed area 128 x 100  $\mu\text{m}$ , acceleration voltage 20 kV, and scanning speed was 90 - 100 diffractograms per second and 4 x 4 binning. EBSD maps were processed in 1000x magnification. Data post-processing (neighbour Confidence Index correlation and grain dilation algorithm) was performed using the OIM software (Version 8.0). Individual data points were grouped into grains if the critical disorientation angle between two adjacent pixels within the grain did not exceed 5° and the minimum number of pixels forming the grain was 2. The evaluation of retained austenite fraction were performed using EBSD and X-ray diffraction (BRUKER D8 DISCOVER device with Cu K $\alpha$  radiation). Diffraction patterns were collected in 2 $\theta$  range from 30° to 120° with a step size of 0.25° and exposure time 0.5 s/step. The measurement was conducted on the polished surface of samples. The peaks of austenite (200), (202), (311), (222) were used for quantification analysis.

Mechanical properties were analysed by means of the Micro - Tensile Testing procedure (M-TT), which was developed in COMTES FHT based on the demand to measure reliably material characteristics by tensile tests from the minimum amount of the experimental material. The M-TT has proved to be effective in earlier studies [4] - [8]. M-TT samples were produced by means of electro-discharge machining in XZY, 45° and ZXY orientations (Figure 1). The ultimate tensile strength (UTS), yield strength (YS), elongation (El), and cross section reduction (Z) of the specimens were measured from 3 samples in each direction. Hardness HV10 was measured using laboratory hardness tester Struers DuraScan 50 with 3 indents in YZ plane.

**Table 1.** Chemical composition of the maraging steel supplied by 3D SYSTEMS [1].

| Element    | Fe   | Ni            | Co           | Mo          | Ti          | Si     | Mn     | C      |
|------------|------|---------------|--------------|-------------|-------------|--------|--------|--------|
| Weight [%] | Bal. | 17.00 - 19.00 | 9.00 - 11.00 | 4.00 - 6.00 | 0.90 - 1.10 | ≤ 1.00 | ≤ 1.00 | ≤ 0.03 |



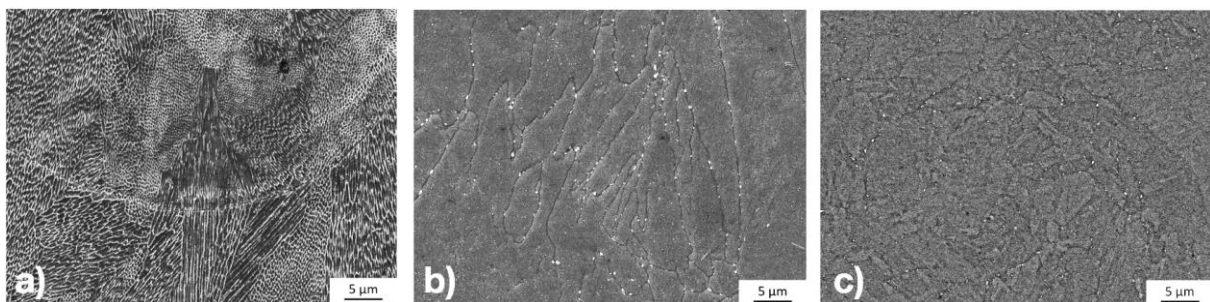
**Figure 1.** Manufactured part (a), M-TT sample XZY orientation (b), M-TT sample 45° orientation (c), M-TT sample ZXY orientation (d), M-TT sample dimensions (e).

**Table 2.** Heat treatment modes for the researched material.

| Sample | Heat treatment condition                                                                            |
|--------|-----------------------------------------------------------------------------------------------------|
| 0      | As-built                                                                                            |
| HT1    | Solution annealing 840 °C / 1 h, water quenching                                                    |
| HT3    | Solution annealing 840 °C / 1 h, water quenching, precipitation hardening 480 °C / 6 h, air cooling |

### 3 Results and Discussion

Microstructural analysis performed by means of light (LM) and electron microscopy (SEM) was performed parallel to the building direction. The use of Nital reagent principally revealed the microstructures (Figure 2, Figure 3) of the lath martensite in a low carbon steel.

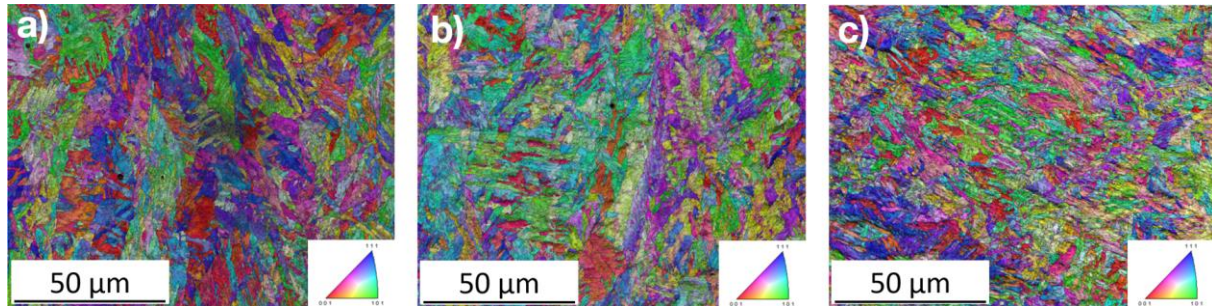
**Figure 2.** LM: a) as-built; b) solution annealing; c) precipitation hardening. Nital.**Figure 3.** SEM: a) as-built; b) solution annealing; c) precipitation hardening. Nital.

The solidification structure of the as-built state (Figure 2a, 3a) with epitaxial grain growth across the individual melt pool boundaries and fine cellular structure was observed by detailed electron microscopy analysis. Solution annealing eliminated cellular microstructure and melts pool boundaries. Undissolved phases (Figure 2b, 3b) in the lath martensitic matrix mainly at the grain boundaries, but also in the matrix of the solid solution state were observed. Aging of the material promoted the formation of intermetallic precipitates within fine martensitic laths (Figure 2c, 3c).

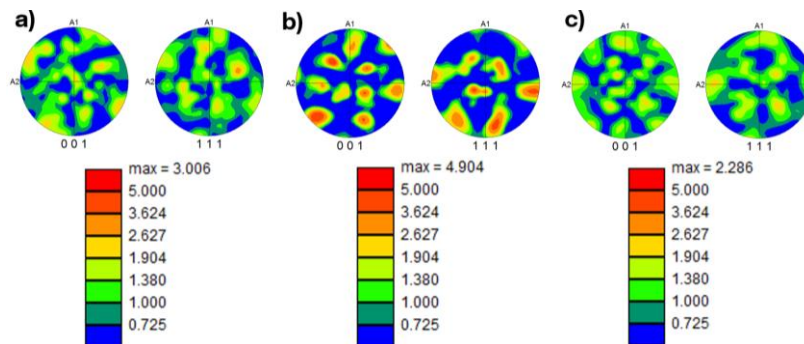
#### 3.1 EBSD analysis

The EBSD analysis provided the information on the development of the microstructure of individual states. The colour Inverse Pole Figure (IPF) maps represent random grain orientations along the building direction of all states (Figure 4). The pole figures which enable the representation of the preferred orientation (texture) in the analysed material (Figure 5) proved that no strong texture was formed during the additive production by the L-PBF method and subsequent heat treatment. The texture in the as-built condition presented in Figure 5a showed that no clear  $\langle 100 \rangle$  alignment occurred in the building direction during the AM process. After solution annealing at 840 °C, there was a slight grain growth and grain rearrangement, as observed by comparing the IPF and PF shown in Figure 4, 5 a - c. The texture after the aging treatment with prior SA retained almost the same as in the as – built state, as shown in Figure 5c.

The amount of retained austenite detected by EBSD analysis was found to be 1 - 2 % with no significant differences between individual states. The lowest fraction was observed by solution annealed state. These results were verified by X-ray diffraction with the similar results.



**Figure 4.** IPF maps: a) IPF as-built; b) IPF solution annealing; c) IPF precipitation hardening.



**Figure 5.** Pole figures: a) As-built state; b) solution annealed state; c) precipitation hardened state.

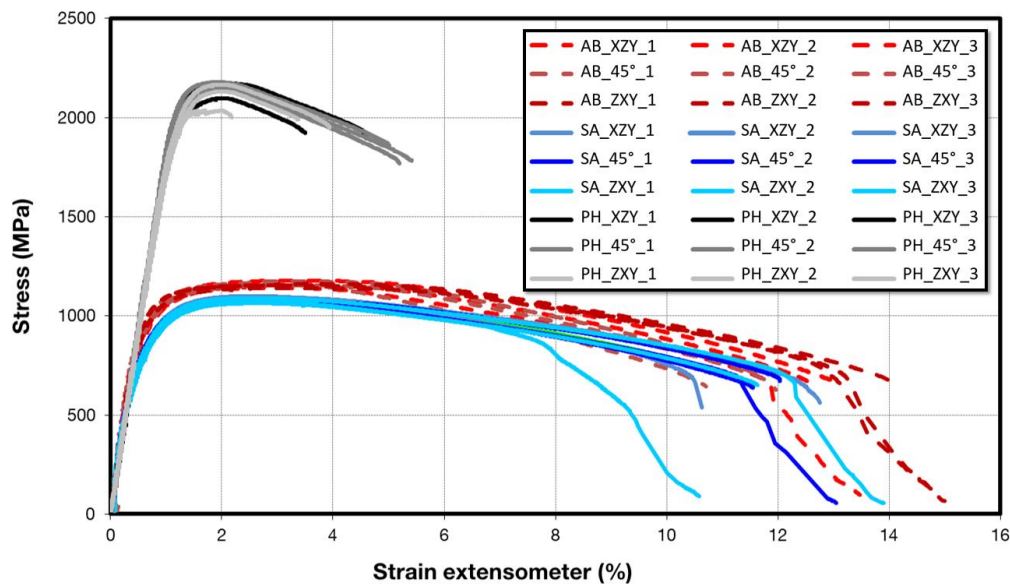
### 3.2 Micro tensile testing (M-TT)

Micro tensile testing of the supersaturated homogeneous state after solution annealing is characterized by lower strength properties in comparison to the as-built state as the microstructure of the as-built state consisted by a mixture of martensite and tempered martensite, which was formed by further applied layers during the laser powder bed fusion.

The precipitation hardening caused a dramatic increase in the ultimate tensile strength at the cost of decreased elongation at break (EI). M-TT testing in XZY, 45° and ZXY directions proved no significant build orientation based anisotropy of mechanical properties in as-built and heat treated samples (Table 3, Figure 6). The prolonged stress - strain curves were caused by further deformation of the sample after a crack was formed, but at the time of the crack the sample was not completely torn. The decrease in hardness confirmed the trend of decreasing yield strength and ultimate tensile strength after solution annealing, whereas precipitation hardening led to the improvement of the hardness (Table 3).

**Table 3.** Mechanical properties of researched material.

| Specimen                     | Specimen | YS [MPa]  | UTS [MPa] | EI [%] | Z [%]   | HV10    |
|------------------------------|----------|-----------|-----------|--------|---------|---------|
| As-built (AB)                | AB_XZY   | 931 ± 17  | 1158 ± 19 | 12 ± 1 | 69 ± 2  | 359 ± 1 |
|                              | AB_45°   | 959 ± 22  | 1163 ± 11 | 11 ± 1 | 63 ± 7  |         |
|                              | AB_ZXY   | 981 ± 24  | 1152 ± 11 | 13 ± 1 | 66 ± 0  |         |
| Solution annealing (SA)      | SA_XZY   | 842 ± 21  | 1099 ± 2  | 11 ± 1 | 61 ± 1  | 330 ± 2 |
|                              | SA_45°   | 814 ± 8   | 1078 ± 8  | 11 ± 0 | 59 ± 5  |         |
|                              | SA_ZXY   | 801 ± 12  | 1077 ± 13 | 11 ± 2 | 60 ± 10 |         |
| Precipitation hardening (PH) | PH_XZY   | 2049 ± 89 | 2145 ± 41 | 3 ± 1  | 51 ± 7  | 618 ± 2 |
|                              | PH_45°   | 2066 ± 28 | 2157 ± 20 | 4 ± 0  | 54 ± 7  |         |
|                              | PH_ZXY   | 1989 ± 22 | 2111 ± 67 | 2 ± 1  | 50 ± 6  |         |



**Figure 6.** Engineering stress-strain curves for XZY, 45° and ZXY specimens of as-built (AB), solution annealing (SA), precipitation hardening (PH) states.

Experience from previous research works [8], [9] monitored the effect of several etchants on revealing different microstructural features. The use of etchants e.g. Adler and Villela – Bain resembled the conventional microstructures with barely revealed traces of 3D process. Nital etching was used to reveal the traces of 3D printing of the researched material. Nital highlighted the laser tracks and cellular microstructure, as it attacks alloy-rich segregations (Ni, Mo, Ti) and solidification structures [8]. The deficiency of texture along building direction of all states is probably caused by changes in the laser paths among layers, which resulted in altering the heat flux direction [2].

The M - TT results are in accordance with theoretical knowledge in [3], that the supersaturated homogeneous state (SA – low carbon lath martensite) is characterized by lower strength properties in contrast to the equilibrium heterogeneous state (AB state - a mixture of martensite and tempered martensite). The decrease in UTS may also be related to a significant reduction in the residual stress found in the material after laser powder bed fusion production and can be mitigated by post heat-treatment [9], [14], [15], i. e. solution annealing.

The effect of subsequent heat treatment on the microstructure and mechanical properties of maraging steel proved that the precipitation hardening caused almost doubling of the values of ultimate tensile strength at the expense of reduced elongation at break. M-TT testing proved no significant build orientation based anisotropy of mechanical properties in as-built and heat treated samples. The achieved results obtained with the use of the micro-tensile tests samples are in good accordance with the results using the standard specimen sizes declared not only by the powder manufacturer, but also other publications [2], [8] - [14].

#### 4 Conclusions

Maraging steel produced by additive manufacturing using the laser powder bed fusion technology was analysed in as-built and two heat treated states by metallographic analysis by means of light, electron microscopy, and electron backscatter diffraction and the results were supported by micro tensile testing.

Metallographic analysis of the as - built state proved the occurrence of a typical line-shaped morphology denoting melt pools. The direction of the grain growth tends to follow the direction of the heat dissipation. Heat treatment of individual states reduced the effect on the melt pool boundaries and fine cell structure. Solution treatment resulted in slight grain growth of the matrix.

The trace amount of retained austenite detected by EBSD and verified by the X-ray diffraction analysis proved no significant differences between individual states. There was also the absence of remarkable texture along building directions.

Micro-tensile tests in the XZY, 45° and ZXY directions reliably measured the material properties. Testing of M - TT is advantageous for additively manufactured materials, which can be tested from a minimum amount of experimental material and thus the costs of AM production can be reduced. Future research will focus on modification of the alloy's chemical composition and its influence on microstructural and mechanical properties.

### Acknowledgements

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### References

- [1] LaserForm Maraging Steel (B) Datasheet. <https://www.3dsystems.com/materials/maraging-steel>
- [2] Tan C and Zhou K 2018 Microstructural characterization and properties of selective laser melted maraging steel with different build directions *Science and Technology of Advanced Materials* 19:1 pp 746-758
- [3] Kraus V 2000 Tepelné zpracování a slinování (Pilsen: University of West Bohemia) chapter 8.3 pp 130 - 131
- [4] Dzugan J and Sibr M 2017 Mechanical properties determination of AM components. *IOP Conference Series: Materials Science and Engineering* 179 012019
- [5] Dzugan J and Seifi M 2018 Effects of thickness and orientation on the small scale fracture behaviour of additively manufactured Ti-6Al-4V *Materials Characterization* 143
- [6] Podany P and Reardon C 2018 Microstructure, mechanical properties and welding of low Carbon, medium manganese TWIP/TRIP steel *Metals* 8 (4) 263
- [7] Pehlivan E and Roudnicka M Effects of build orientation and sample geometry on the mechanical response of miniature CP-Ti Grade 2 strut samples manufactured by laser powder bed fusion 2020 *Additive Manufacturing* 35 101403
- [8] Hrbáková T and Simson T 2018 The effect of heat treatment on mechanical properties and microstructure of additively manufactured components. *IOP Conf. Ser.: Mater. Sci. Eng.* 461 012026
- [9] KucEROVA L and ZETKOVÁ I 2019 Microstructural characterisation and in-situ straining of additive-manufactured X3NiCoMoTi 18-9-5 maraging steel *Materials Science & Engineering A* 750 pp 70–80
- [10] MUTUA J and NAKATA S 2018 Optimization of selective laser melting parameters and influence of post heat treatment on microstructure and mechanical properties of maraging steel *Materials and Design* 139 pp 486–497
- [11] Tan C and Zhou K 2017 Microstructural evolution, nanoprecipitation behavior and mechanical properties of selective laser melted high-performance grade 300 maraging steel *Materials & Design* 134 pp 23–34
- [12] Simson T and Koch J 2019 Mechanical Properties of 18Ni-300 maraging steel manufactured by LPBF Proc. Int. Conf. on Structural Integrity 17 pp 843–849
- [13] Kempen K and Yasa E Microstructure and mechanical properties of Selective Laser Melted 18Ni-300 steel *Physics Procedia* 12 pp 255–263
- [14] Valenta J 2018 Heat treatment of maraging steel processed by SLM 2018 Bachelor's thesis VUT Brno
- [15] Hermann Becker T and Dimitrov D The achievable mechanical properties of SLM produced Maraging Steel 300 components 2016 *Rapid Prototyping Journal* 22(3) pp 487–494