Corrosion Resistance of 18Ni-300 maraging steel manufactured by LPBF

T Simson¹, J Koch¹, M Zetek², I Zetková²

¹OTH Amberg-Weiden, Kaiser-Wilhelm-Ring 23, Amberg 92224, Germany
²University of West Bohemia, Universitní 22, Pilsen 306 14, Czech Republic
E-mail: t.simson@oth-aw.de

Abstract. The study describes the corrosion behaviour of LPBF parts made of MAR 300 (EN 1.2709) in sulphuric acid. It includes the influence of the heat treatment on the part's properties as solution annealed and age hardened with reference to conventional samples. Laser remelted surfaces were examined as well. Due to the layered production of AM components, which is characteristic for this process, high surface roughness occurs. Therefore, for many applications and mostly on functional surfaces, complex post-processing of the surfaces is necessary. The reliable use of the manufactured components, demand knowledge of the specific influence of the selected post-processing methods on the electrochemical behaviour. The corrosion investigation takes place on different sample surfaces, such as milled, turned, sandblasted, untreated, grinded and laser-machined. The results show that there is no correlation between surface treatment and corrosion resistance. However, an increase in corrosion rate due to age hardening of the material can be observed for both LPBF and conventionally produced samples. By laser surface remelting of these age hardened samples, the corrosion resistance of these parts could be increased again, to the same level as in the solution annealed state.

1 Introduction
The laser powder bed fusion (LPBF) process is a part of additive manufacturing (AM). The advantages of AM processes are i.a. geometrical freedom, material flexibility, creating opportunities to fabricate innovative parts with enhanced functionality. [1] MAR 300 is a well-known maraging steel, which belongs to a special class of ultra-high strength steels, which differs from conventional steels by hardening via intermetallic precipitations without carbon. The quality of the used powder feedstock as well as the achievable product properties, corresponds to the powder morphology, including particle size distribution and shape. Therefore, for many applications and mostly on functional surfaces, complex post-processing of the surfaces is necessary. This post-processing is often carried out by conventional manufacturing and mechanical surface treatment techniques (e.g. grinding, milling, drilling or blasting). But also, laser treatment as a thermal post-processing method can smooth the surface and influence material properties. [2] For the reliable use of the manufactured components and due to the increasing relevance of AM processes in the industry it is necessary to know the specific influence of the selected post-processing methods on the electrochemical corrosion behaviour.

R. Schaller et al. [3] report on microstructural inhomogeneities such as pores or oxides on the sample surface effecting the corrosion behaviour of materials. Due to the manufacturing process by LPBF, samples are susceptible to pores, oxides and interconnectivities in the matrix which can affect the corrosion behaviour. According to the tested MAR 300 some publications
e.g. A. Ettefagh et al. [4] analyse that samples from stainless steel AISI 316L show decreased corrosion rates in comparison to conventionally manufactured ones. J. Suryawanshi et al. [5] on the other side mentioned that AM parts of MAR 300 show lower corrosion resistance caused by the higher porosity level compared with conventionally manufactured samples. In order to get to a substantiated statement of the comparability between the corrosion behaviour of LPBF and conventionally manufactured samples, corrosion measurements on the MAR 300 were carried out.

2 Experimental procedure

2.1 Material and Samples

The material used for this investigation is martensitic steel MAR 300 (EN 1.2709) with the chemical composition shown in Table 1. The powder was bought by Electro Optical Systems (EOS) Finland Oy and produced using gas atomization resulting in spherical partic composition shown in Table 1. The powder was bought by Electro Optical Systems (EOS) Finland Oy.

| Table 1. Chemical composition of new MAR 300 powder in wt% |
|------------------------------------------------------------|
| Fe  | Co  | Ni  | Mo  | Ti  | Al  | Cu  | Cr  | Mn  | Si  | C   | P   | S   |
|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| Balance | 8.76 | 18.25 | 4.80 | 0.64 | 0.06 | 0.03 | 0.12 | 0.06 | 0.02 | 0.01 | <0.01 | 0.01 |

The laser scanning strategy used zigzag pattern with 33.3° rotation between the superimposed layers. The process parameters used were laser power (P), scan speed (v), hatch spacing (h) and layer thickness (d), the values are shown in Table 2. Before starting the LPBF process, the base plate was preheated to 40°C by a heater placed inside the building platform, and the oxygen content in the process chamber was maintained below 0.04 % by pumping in nitrogen continuously. Further investigations by computer tomography determined for all samples a porosity in average of 0.0073 % which corresponds to a structure density, higher than 99.99 %.

| Table 2. Process parameters for different pattern |
|-------------------------------------------------|
| Pattern         | Laser power (W) | Scan speed (mm/s) | Hatch spacing (mm) | Layer thickness (mm) |
| In Fill         | 285             | 960              | 0.11               | 0.04               |
| UpDown          | 153             | 600              | 0.11               | 0.12               |
| DownSkin        | 145             | 2400             | 0.11               | 0.16               |

After the manufacturing process (as-built state) the samples were first solution annealed at 820 °C for 1 h in Argon atmosphere by using a gas-tight chamber furnace of the PKR 35/11 type from LAC followed by air-cooling down to room temperature. According to the testing conditions some samples were age hardened at 490 °C for 6 h. The aging was carried out using a chamber furnace from Linn High Therm GmbH with the type designation HK-70. The volume is 70 litres and the heating power 18 kW.

The post-processing of to sample surface was prepared with the same cutting edge radius by turning, milling and the cutting conditions were optimized to obtain the same surface input properties. The surface modifications were performed using an ANCA MX7 grinding machine with the cutting conditions, cutting speed (vc) 50 m/s, feed rate (vf) 100 mm/min and depth of cut (ap) 0.01 mm. Milling was done with a DMU 40 and turning with a multitask turning centre CTX BETA 1250TC. The cutting conditions were chosen to be the same for both machining with vc = 110 m/s, vf = 140 mm/min and depth of cut (ap) 0.5 mm. A manually controlled machine with a pressure of 4 bar and a Garnet 100 blasting abrasive was used for sandblasting. The laser treatment (LT) was carried out by a diode-pumped fibre laser from IPG Photonics of the type YLR-150/1500-QCW-MM-AC. The processing parameters selected were P = 93 W, v = 125 mm/s, h = 0.09 mm, the surfaces were remelted twice, each with an angular offset of 90°. Before laser processing, the samples were grinded with silicon carbide abrasive paper with a grain size of P180 (75 µm).
The surface roughness of the samples was determined with the Perhometer H1 from Garant. This is integrated into the system (Perthometer Concept) of the company Mahr (Mahr M1 Perhometer). The Rz values were evaluated and logged using the MarSurf 20 XR 20 V1.40-3 (SP1) software and determined by an average value from three measured points. The surface morphology was analysed by a scanning electron microscope (SEM) Leo Stereo Scan 440 (Figure 1).

![SEM micrographs 200-fold magnification with values of surface roughness](image1)

- **Rz = 5.19 ± 1.10 µm**
- **Rz = 3.27 ± 0.13 µm**
- **Rz = 11.78 ± 1.76 µm**

**Figure 1.** SEM micrographs 200-fold magnification with values of surface roughness (a) milled, (b) turned, (c) sandblasted, (d) as-build, (e) grinded

The laser treated surfaces were analysed by microscopy images displayed in Figure 2. The evaluation of the remelting depth was carried out on the cross-section (perpendicular to the building direction) using the BX60M reflected-light microscope from Olympus and the DMC2900 camera from Leica. For image processing the software IMS Client V17Q4 from Imagic was used. Figure 2 a) shows a partial area of the laser treated surfaces with a roughness of 2.64 ± 0.62 µm. To determine the melting depth of the laser treatment metallographic examinations were carried out. Figure 2 b) shows a melting depth d of 75 µm, the heat-affected zone is 46 µm.

![Micrographs with values of surface roughness](image2)

- **Rz = 2.64 ± 0.62 µm**
- **d = 75 µm**

**Figure 2.** Micrographs with values of surface roughness (a) laser treated, (b) microsection of laser treatment

### 2.2 Corrosion measurements

The corrosion behaviour was investigated by using a potentiostatic measurement system, type Avesta Cell. The performed research work examined corrosion potentials for different surface structures of
solution annealed LPBF parts in comparison to conventionally produced material. The parts for the corrosion investigations (diameter $D$ 35 mm, thickness $t$ 16 mm) were prepared to have different surface qualities, such as milled, turned, sandblasted, as-build, grinded and laser treated ones. The test set-up includes the potentiostat PGS 95 and the software CPC934s1 V 5.1 from Bank Elektronik. The reference electrode used was Ag/AgCl in a 1nH$_2$SO$_4$ electrolyte. Experiments were carried out at 21°C. In addition, a Haber-Luggin capillary was included in the structure. The samples were cathodically polarized at the beginning of the measurement at a potential $\varepsilon_R$ (starting in the anodic area) and driven with $d\varepsilon/dt$ from 0.2 mV/s to $\varepsilon_R$ ± 200 mV. The total measuring area of the individual samples is 0.28 cm$^2$. The evaluation of the current density-potential curves was done with Microsoft Excel. In this case, current per area $I/A$ (mA/cm$^2$) is plotted logarithmically against the normal hydrogen electrode (NHE). The potentiodynamic polarisation parameters, corrosion potential ($E_{corr}$) and current density ($I_{corr}$), are evaluated values for assessing the corrosion behaviour determined by Tafel plots according to Figure 3. The intersection of the cathodic (yellow line) and the anodic (green line) tangents determine the corrosion behaviour for all measurements. [6] All sample were measured three times to calculate an arithmetic mean value of the corrosion behaviour.

![Figure 3. Corrosion evaluation by Tafel plots on an additive sample (grinded surface and solution annealed heat treatment)](image)

### Results and discussion

According to the post-processes such as milled, turned, sandblasted, as-build, grinded and laser treated (compare Figure 1 and 2), different surface roughness was realized. The arithmetic mean values of the electrochemical measurements on the different post-processes for annealed samples were summarised in Table 3. The results of the corrosion measurements show that the corrosion potential $E_{corr}$ – varying in a range of -78 ~ -74 mV – is comparable for all post-processes. Also, the current density as a value for the corrosion rate $I_{corr}$ – varying between 0.38 ~ 0.70 mA/cm$^2$ – does not show any influence by the surface roughness. The results of the samples and the wrought material show that there is no correlation between the surface roughness and $E_{corr}$ or $I_{corr}$.

| Surface treatment | Sample | Heat treatment | $E_{corr}$ (mV NHE) | $I_{corr}$ (mA/cm$^2$) |
|-------------------|--------|---------------|---------------------|----------------------|
| Milled            | Additive | Solution annealed | -75 +/- 2.0 | 0.48 +/- 0.03 |
| Turned            | Additive | Solution annealed | -75 +/- 1.5 | 0.55 +/- 0.15 |
| Sandblasted       | Additive | Solution annealed | -78 +/- 0.5 | 0.70 +/- 0.10 |
| As-build          | Additive | Solution annealed | -74 +/- 1.0 | 0.38 +/- 0.03 |
| Grinded           | Additive | Solution annealed | -78 +/- 0.5 | 0.65 +/- 0.05 |
| Grinded + LT      | Additive | Solution annealed | -77 +/- 1.5 | 0.50 +/- 0.10 |
The measured corrosion behaviour of the conventional and LPBF samples in the heat treatment stages solution annealed and age hardened is summarized in Table 4 including the mechanical (grinding) and thermal (laser treated) post-processes. It can be observed that, contrary to the statement of Jyoti Suryawanshi [5], the LPBF samples do not deteriorate the corrosion resistance compared with conventional manufactured samples. The investigations revealed that the manufacturing methods have no measurable effect and show similar corrosion behaviour.

**Table 4.** Average $E_{\text{corr}}$ and $I_{\text{corr}}$ of LPBF and conventional manufactured samples in different heat treatment stages and mechanical and thermal post-processed surfaces.

| Surface treatment | Sample  | Heat treatment | $E_{\text{corr}}$ (mV NHE) | $I_{\text{corr}}$ (mA/cm²) |
|-------------------|---------|---------------|-----------------------------|-----------------------------|
| Grinded           | Conventional | Solution annealed | -74 +/- 1.0 | 0.53 +/- 0.03 |
| Grinded + LT      | Additive | Solution annealed | -77 +/- 1.5 | 0.50 +/- 0.10 |
| Grinded + LT      | Conventional | Solution annealed | -81 +/- 2.0 | 0.70 +/- 0.05 |
| As-Build          | Additive | Age hardened | -79 +/- 3.5 | 1.25 +/- 0.05 |
| Grinded           | Additive | Age hardened | -73 +/- 2.5 | 1.20 +/- 0.15 |
| Grinded           | Conventional | Age hardened | -71 +/- 1.5 | 1.55 +/- 0.15 |
| Grinded + LT      | Additive | Age hardened | -78 +/- 0.5 | 0.40 +/- 0.05 |
| Grinded + LT      | Conventional | Age hardened | -79 +/- 0.5 | 0.50 +/- 0.05 |

However, it can be observed that the corrosion of the solution annealed samples is lower than that of the age hardened samples. The corrosion potentials $E_{\text{corr}}$ for the samples according to Figure 3 are in the range of -81 ~ -71 mV (values referring to the potential of the normal hydrogen electrode NHE). The corrosion current densities of the solution annealed samples are in a range of $I_{\text{corr}} = 0.50 \sim 0.7$ mA/cm². The corrosion current densities of the age hardened samples (without LT) vary between $I_{\text{corr}} = 1.2 \sim 1.55$ mA/cm², which corresponds to a factor of about two. The corrosion current density is directly proportional to the corrosion rate, which doubles in this case. This may be due to the fact that these samples are solution annealed and that this heat treatment results in a homogeneous microstructure. A single phase or homogeneous metal resisted the corrosion attack better than a multiphase or inhomogeneous metal. [7]

In this condition, the samples contain a martensitic matrix consisting predominantly of iron and nickel. As a result, the corrosion behaviour is comparable to that of iron and nickel. In the case of MAR 300, the precipitations formed during aging have been identified as complex of nickel-cobalt-molybdenum-titanium (Ni3Mo and Ni3Ti). [8]–[9] These intermetallics have different compositions therefore their electrochemical behaviour differs from the matrix. [10]–[13] Due to the different crystal structures and lattice parameters, stress fields formed around the precipitations. These composition differences lead to a lower corrosion resistance of age hardened samples due to the galvanic effect than the solution annealed ones. [14]–[17] The corrosion current densities of the age hardened and subsequently laser treated samples show a similar corrosion behaviour as the solution annealed states ($I_{\text{corr}} = 0.4 \sim 0.5$ mA/cm²). As a result of the laser treatment, the precipitations in the melted area of the microstructure dissolve again and thus increase the corrosion resistance of the material again. In principle, all corrosion attacks are uniform surface corrosion.

4 Conclusion
The results demonstrate that samples from MAR 300 processed by LPBF with high structure density and with a low number of pores deliver similar results as conventionally manufactured ones.

- It can be observed that different surface qualities such as milled, turned, sandblasted, as-build and ground have no significant influence on the corrosion resistance.
- However, the corrosion resistance of age hardened samples is lower than of solution annealed samples. This leads to a lower corrosion resistance of the age hardened specimen due to a galvanic effect. For the solution annealed samples, the corrosion current densities are in a range of $I_{\text{corr}} = 0.38 \sim 0.7$ mA/cm². The corrosion current densities, approximately twice as high, were measured for them, $I_{\text{corr}} = 1.2 \sim 1.55$ mA/cm².
By laser treatment, the precipitations could be dissolved in a thin surface layer of 75 µm and thus the corrosion rate decreases to the values of the solution annealed condition. The influence of the laser treated surface on the mechanical properties must be examined further.

The manufacturing method of the additive or conventional material samples has no measurable effect on the corrosion resistance. The investigations show that LPBF components show a similar corrosion behaviour as the conventionally produced samples.

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References
[1] Rüßmann M, Lorenz M, Gerbert P, Waldner M, Justus J, Engel P and Harnisch M 2015 Industry 4.0: The Future of Productivity and Growth in Manufacturing Industries Boston Consulting Group: Boston MA USA Volume 9
[2] Frenk A, Kurz W 1992 Laser Surface Treatment: Microstructural Aspects, Springer Science and Business Media Dordrecht
[3] Schaller R F, Taylor J M, Rodelas J and Schindelholz J 2016 Corrosion Properties of Powder Bed Fusion Additively Manufactured 17-4 PH Stainless Steel Sandia National Laboratories Albuquerque
[4] Ettefagh A H and Guo S 2016 Electrochemical behavior of AISI316L stainless steel parts produced by laser-bed powder bed fusion process and the effect of post annealing process ScienceDirect Additive manufacturing: Louisiana State University Baton USA
[5] Suryawanshi J, Baskaran T, Prakash O, Arya S B and Ramamurty U 2018 On the corrosion resistance of some selective laser melted alloys ScienceDirect Materialia: Indian Institute of Science Bangalore India
[6] Buchanan R A and Stansbury E E 2012 Handbook of Environmental Degradation of Materials Elsevier 2nd ed.(4) pp 113-117
[7] Wadhwa V C 1965 Corrosion behavior of heat treated 18 per cent nickel maraging steel
[8] Floreen S 1968 The physical metallurgy of maraging steels Metallurgical Reviews 13:1 pp 115-128
[9] Tewari R, Mazumder S, Batra I S, Dey G K and Banerjee S 2000 Precipitation in 18 wt% Ni maraging steel of grade 350 Acta Mater 48(5) pp 1187–1200
[10] Sedriks D W 1992 Metals Handbook ASM International 9th ed. volume 13
[11] Poornima T, Nayak J and Shetty A N 2010 Corrosion of aged and annealed 18 Ni 250 grade maraging steel in phosphoric acid medium Int. J. Electrochem. Sci 5 pp 56-71
[12] Ansell T Y, Ricks J P, Park C, Tipper C S and Luhrs C C 2020 Mechanical Properties of 3D-Printed Maraging Steel Induced by Environmental Exposure Metals 10(2) 218.
[13] Tan C, Zhou K, Kuang M, Ma W and Kuang T 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
[14] Avelino A F, Araújo W S, Dias D F, dos Santos L P M, Correia A N and de Lima-Neto P 2018 Corrosion investigation of the 18Ni 300 grade maraging steel in aqueous chloride medium containing H2S and CO2 Electrochimica Acta 286 pp 339-349