

Tailored Hardness Profiles Through a Combination of Specialized PBF-LB Processing Strategies with Subsequent Heat Treatment for Graded High-Strength Components Made of Maraging Steel

Niki Nouri ^{1,*1}, Gregor Graf ², Stefan Dietrich ¹ and Volker Schulze ¹

¹ Institute for Applied Materials – Materials Science and Engineering (IAM-WK) / Karlsruhe Institute of Technology (KIT), 76131 Karlsruhe, Germany

² Rosswag GmbH, 76327 Pfinztal, Germany

This study investigates the utilization of laser powder bed fusion for fabricating functionally graded parts using a novel maraging steel named Specialis[®]. By manipulating the processing strategy involving single and dual laser exposures, along with remelting, graded parts are successfully produced. Both the as-built and heat-treated specimens exhibit hardness variations of up to 80 HV across distinct regions. Notably, a reversal of hardness levels occurs after heat treatment, with the originally harder as-built dual laser zone becoming the softest zone following the post heat treatment. This offers innovative approaches and eliminates the necessity for complex and time-consuming intrinsic heat treatment steps. Instead, the manufactured parts can be efficiently furnace-hardened while retaining their graded properties. These findings facilitate the production of components with tailored hardness profiles, precisely suited for their intended applications. The versatility of additive manufacturing, combined with the unique capabilities of Specialis[®], opens a new avenue for creating functionally graded parts with improved mechanical properties.

Keywords: dual laser based powder bed fusion, maraging steel, post heat treatment, precipitation hardening, functionally graded material, tailored hardness

1. Introduction

Functionally graded materials (FGMs) designate a group of advanced materials with a progressive variation in properties regarding the spatial positions by varying the material, microstructure, defects or macrostructure ¹. The fabrication of metal FGMs by means of additive manufacturing (AM) has been of great interest in recent years, as the freedom in geometry during the process could simplify the approach and enable a specific adaption of components to the application. Numerous studies have explored the gradation of parts fabricated by AM by varying the chemical composition ², the density ³, the structure ⁴, or the process parameters which result in a change in the microstructure ⁵. An intrinsic heat treatment (IHT) during the AM process could also change the microstructure and open new possibilities of creating FGMs, as already achieved for a maraging steel ⁶. Maraging steels have been extensively investigated as suitable materials for laser powder bed fusion (PBF-LB). To achieve peak hardness, these steels must undergo a long aging heat treatment (~6 h), during which precipitation hardening occurs. Nevertheless, there has been evidence of hardness increase after short aging times ^{7,8} due to early precipitation formation ⁹.

In this work a novel maraging steel for PBF-LB has been analyzed regarding the manufacturing of a graded microstructure by varying the processing strategies. Maraging steels manufactured by PBF-LB commonly find use in applications demanding complex geometries and high strength, such as molds. Therefore, the investigations were carried out before and after heat treatment.

2. Experiment

2.1 Material

The investigated novel maraging steel, called Specialis[®],

has been developed for PBF-LB by SpecMaterials and processed by Rosswag Engineering. Table 1 contains the chemical composition of the material, which is based on the commonly used maraging steels 18Ni300 and 18Ni350, with an addition of V and Al. The focus of earlier investigations was on the optimization of material qualification process ¹⁰, process parameters ^{11,12}, heat treatment ¹², as well as the analysis of phase transformations and strengthening mechanisms (manuscript submitted for publication). Specialis[®] surpasses the hardness of conventional 18Ni300 while requiring a shorter aging time of 2 h. The main strengthening effects observed after heat treatment include intermetallic precipitations and grain refinement.

Table 1: Chemical composition of Specialis[®] in wt%

C	Ni	Co	Mo	Ti	V	Al	Fe
0.01	18.27	10.69	4.22	1.75	1.61	0.19	Bal.

2.2. Processing Strategies

All samples were fabricated by the PBF-LB system SLM[®]280 HL Twin 400 W with gas flow upgrade by SLM Solutions Group AG. A schematic overview of the processing strategies and their influence on the thermal history of the melt pool is shown in Figure 1. The following three processing strategies were used with the optimized parameters, conducted from earlier studies ^{11,12}:

- Single laser (SL): only one laser with the power $P1 = 200$ W is used.
- Dual laser (DL): A second laser with the power $P2 = 190$ W follows the same path as the first laser with the power $P1$, keeping a constant distance of $\Delta s = 5$ mm. This would extend the cooling time and the resulting IHT.
- Remelting (RM): The second laser has a reduced power of $P3 = 125$ W compared to the DL strategy and the distance to the first laser is increased. There

*1 PhD Student, Karlsruhe Institute of Technology (KIT)

is a time offset of $\Delta t = 3$ s between both lasers, allowing the material to reach a homogenous temperature level before the second laser approaches. The following scanning parameters were kept constant for all strategies: 800 mm/s scanning speed, 0.85 mm hatch distance, and 40 μ m layer thickness.

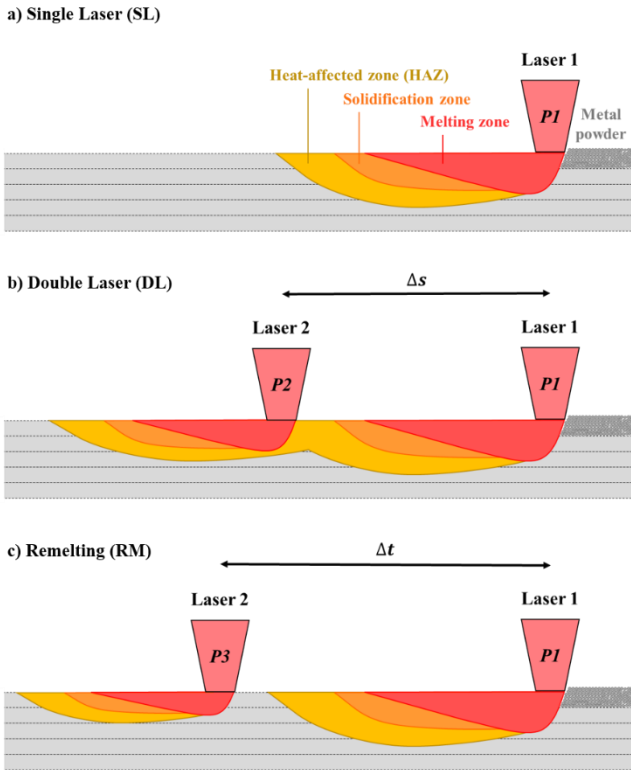


Figure 1: Overview of the influence of a) SL, b) DL, c) RM processing strategy on the thermal history of the melt pool.

2.3 Methods

A DIL 805 dilatometer from TA Instruments was used to perform the short time aging heat treatments at 500 °C for 5, 10, 25, 50 and 100 s. The process involved inductive heating within a vacuum chamber filled with helium to prevent oxidation or evaporation. To regulate the temperature, a type S thermocouple was spot welded at the center of a 10 mm-long cylindrical hollow sample, fabricated with SL strategy.

To explore the feasibility of manufacturing graded components, cubic samples were vertically divided into three distinct zones. The fabrication was carried out by using three processing strategies in each layer. The chemical composition of these graded samples was determined using an iCAP 7600 DUO inductively coupled plasma-optical emission spectrometry (ICP-OES) instrument from Thermo Fisher Scientific. At least three samples per strategy were analyzed to obtain a mean value.

Micro Vickers hardness measurements at $HV_{0.1}$ were conducted using a Qness Q30a+ tester, following the DIN EN ISO 6507-1 standard¹³. The samples were ground and polished prior to the measurements. For each sample, three horizontal hardness profiles and one vertical profile per area (SL, DL, RM) were generated. The horizontal measurements started and ended 1 mm from the side edges to eliminate the influence of any border effects. For the same reason the vertical measurements started 0.5 mm away from the top

surface. The hardness has been measured both in as-built and heat treated states. Heat treated samples were subject to a vacuum furnace aging treatment at 500 °C for 3 h.

3. Results

The outcomes of the horizontal hardness measurements conducted on graded components are exhibited in Figure 2. The position of the horizontal measurement (top, middle, bottom) does not have any noteworthy influence on the hardness. Nevertheless, clear distinctions can be observed among the three employed processing approaches. While SL and RM exhibit comparable vertical mean hardness values in their as-built states (433 ± 7 and 441 ± 9 $HV_{0.1}$, respectively), DL results in a higher mean hardness (460 ± 11 $HV_{0.1}$). These disparities become even more pronounced following heat treatment, wherein a surprising outcome arises: DL demonstrates a diminished mean hardness (629 ± 18 $HV_{0.1}$) compared to SL and RM (712 ± 11 and 703 ± 18 $HV_{0.1}$, respectively). These trends remain consistent for samples produced with alternate sequences of processing strategy zones (DL|SL|RM or SL|RM|DL).

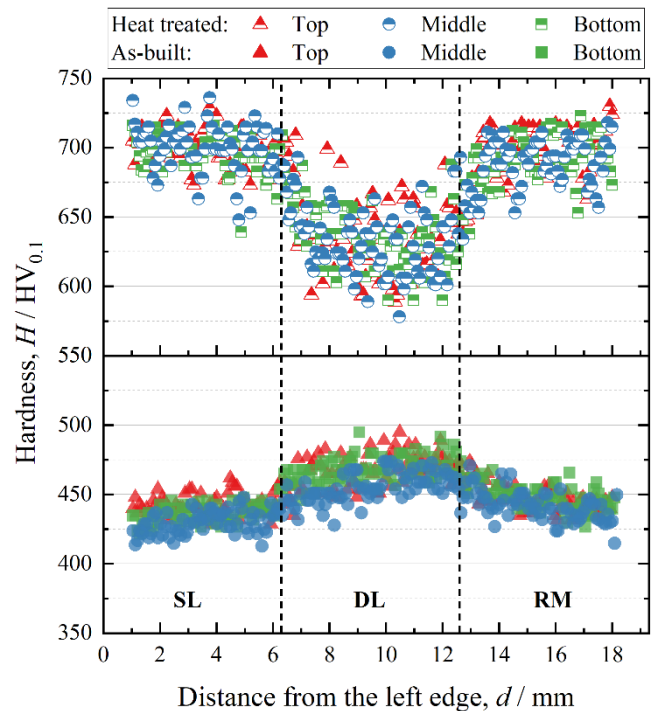


Figure 2: The horizontally measured hardness profiles of a sample in as-built and heat treated states at three different positions (top, middle, bottom). Dashed lines approximately mark the transitions between different processing strategy zones (SL, DL, RM).

Short time aging treatments were carried out on SL samples to investigate potential early stages of precipitation hardening. The evolution of hardness over aging durations up to 100 s is depicted in Figure 3. The results clearly indicate that even brief aging times induce an increase in hardness for Specialis® (~ 75 $HV_{0.1}$ after 10 s). The relationship between hardness H and the logarithm of aging time t_A in seconds can be described by the equation

$$H = 428 + 75 \log(t_A),$$

illustrated as an orange dashed line in Figure 3. The intercept of this line with y-axis at 428 aligns closely with the hardness value of the SL sample in its as-built state ($433 \pm 7 \text{ HV}_{0.1}$).

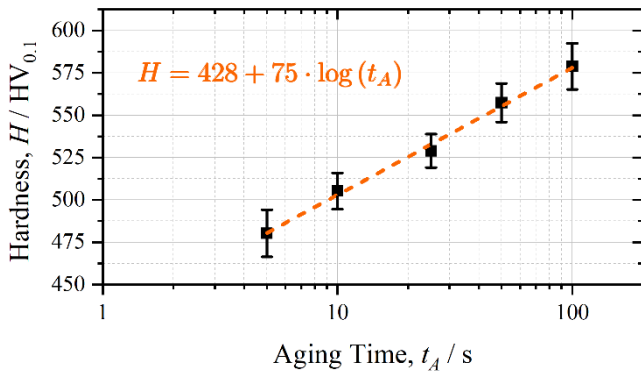


Figure 3: The evolution of hardness H depending on the aging time t_A after short time aging heat treatments of samples processed with SL. The dashed orange line represents the linear fit between H and $\log(t_A)$ in seconds.

4. Discussion

In the context of the present study, the influence of three different manufacturing methods (SL, DL, RM) on the hardness were examined in as-built and heat treated states. In its as-built state, DL showcases a notably higher level of hardness compared to SL and RM counterparts. This phenomenon can be attributed to the IHT generated by the second laser employed in the DL process, which leads to the early stages of precipitation hardening. This proposition is convincingly validated by the results obtained from short time aging experiments, wherein the hardness of SL specimens surpasses 500 HV even following a mere 10 s aging at 500 °C. However, an unexpected deviation emerges after post-heat treatment, where DL's hardness registers lower than that of SL and RM. In order to verify if any elements were evaporated due to high temperatures during PBF-LB, a chemical analysis was carried out using ICP-OES. Yet the results showed no difference in the chemical composition of the three areas. Given the observed uniformity in the chemical composition, the diversion in hardness implies a modification within the microstructure of DL zone, resulting in diminished precipitation formation. Therefore, a graded part was successfully fabricated, which can conserve its graded microstructure even after heat treatment. It is yet remarkable that there is a reversal in terms of which area exhibits the highest hardness. Detailed microstructural investigations, including the potential formation of elemental clusters, are crucial to unveil the mechanisms behind this intriguing shift.

5. Conclusions

In Summary, this study involved the PBF-LB fabrication of graded components out of Specialis® maraging steel, utilizing three distinct manufacturing approaches: SL, DL, and RM. The following can be concluded:

- DL showcased elevated initial hardness owing to early precipitation stages, a consequence of the IHT introduced by its second laser.

- However, in contrast, following heat treatment and the progression of precipitation hardening, both SL and RM exhibited superior hardness, although it was confirmed through chemical analysis that no elements were evaporated during the processes.
- Nevertheless, graded parts with a variation of up to $\sim 80 \text{ HV}_{0.1}$ were successfully fabricated in as-built and heat treated states.

The findings open up the possibility of manufacturing complex geometries out of the maraging steel Specialis® with a graded microstructure. For instance, it enables the achievement of surface hardening without incurring a hardness decline in overaged regions. Furthermore, the heat treatment can be performed throughout the entire part in a furnace, rather than being limited to the surface. This feature allows complex geometries to be topologically optimized and contain a tailored hardness profile specifically adapted to the application.

Acknowledgments

The authors would like to thank Dr. Thomas Bergfeldt (KIT, IAM-AWP) for conducting the chemical analysis.

References

- 1) A. K. Mishra, K. Yadav, and A. Kumar: *Kumar, Mittal et al. (Hg.) 2022 – Advances in Additive Manufacturing*: pp. 281–297.
- 2) Y. Zhang, Z. Wei, L. Shi, and M. Xi, *Journal of Materials Processing Technology* **206** (2008) pp. 438–444.
- 3) M. Fousová, D. Vojtěch, J. Kubásek, E. Jablonská, and J. Fojt, *Journal of the Mechanical Behavior of Biomedical Materials* **69** (2017) pp. 368–376.
- 4) S. Y. Choy, C.-N. Sun, K. F. Leong, and J. Wei, *Materials & Design* **131** (2017) pp. 112–120.
- 5) T. Niendorf, S. Leuders, A. Riemer, F. Brenne, T. Tröster, H. A. Richard, and D. Schwarze, *Adv. Eng. Mater.* **16** (2014) pp. 857–861.
- 6) P. Kürnsteiner, M. B. Wilms, A. Weisheit, B. Gault, E. A. Jägle, and D. Raabe, *Nature* **582** (2020) pp. 515–519.
- 7) J. Marcisz and J. Stepień, *Archives of Metallurgy and Materials* **59** (2014) pp. 513–520.
- 8) A. Shekhter, H. I. Aaronson, M. R. Miller, S. P. Ringer, and E. V. Pereloma, *Metall Mater Trans A* **35** (2004) pp. 973–983.
- 9) E. V. Pereloma, R. A. Stohr, M. K. Miller, and S. P. Ringer, *Metall Mater Trans A* **40** (2009) pp. 3069–3075.
- 10) G. Graf, M. Neuenfeldt, T. Müller, J. Fischer-Bühner, D. Beckers, S. Donisi, F. Zanger, and V. Schulze, *Advanced Materials Research* **1161** (2021) pp. 27–36.
- 11) G. Graf, N. Nouri, S. Dietrich, F. Zanger, and V. Schulze, *Materials (Basel, Switzerland)* **14** (2021) pp. 4251.
- 12) N. Nouri, Q. Li, J. Damon, F. Mühl, G. Graf, S. Dietrich, and V. Schulze, *Journal of Materials Research and Technology* **18** (2022) pp. 931–942.
- 13) Deutsches Institut für Normung, *Metallische Werkstoffe - Härteprüfung nach Vickers. Teil 1: Prüfverfahren* [DIN EN ISO 6507-1] (2018).