Improvement of the properties of additively manufactured steel parts by combination of heat treatment and hard coatings

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Abstract. In additive manufacturing using SLM (selective laser melting), the maraging steel 1.2709 is often used to produce metallic components for various applications. The work piece properties depend on the process control in the additive process, but also on the heat treatment and the final processing. Here, the heat treatment and the used finishing processes lead to changes in the work piece and material properties (e.g. residual stresses, strength). As part of the international "Ad-Proc-Add" project, the presented work took into account individual process steps of the process chain with consideration of: additive manufacturing - pre-treatment - functionalization - final processing when using different materials. In investigations of additive manufacturing of the material 1.2709, it could be proven that the thermal treatment determines the mechanical properties of the components. A PVD coating process was used to replace the usual heat treatment. Similar temperature-time regimes during the coating resulted in mechanical properties comparable to conventionally heat-treated additively manufactured components. In addition to modifying the microstructure and the component properties, the PVD coatings improve the surface wear resistance up to more than 60%. Further investigations to improve the adhesion strength of the coatings are planned.

1. Introduction

In Additive Manufacturing (AM) three-dimensional objects are built directly by layer-wise material deposition. Complex shaped parts with internal structures can be generated, which cannot be realized with conventional manufacturing methods or only at considerable expense [1, 2]. One way of additive manufacturing is the laser powder bed fusion (LPBF) method. Here a laser is used to selectively heat and melt powder in order to build up the layers sequentially. Depending on the source power and scanning velocity, a significant energy impact on the work piece takes place. This energy influences structure and properties, e.g. strength, hardness and residual stresses. In powder bed based AM, properties can be modified by heat treatment of the generated part [3].

Main challenges of AM technologies are the achievable surface quality and dimensional accuracy. Functional surfaces often have to be reworked in order to reach the required tolerances [4]. In addition, any existing support structures must be removed from the part. Post-processing steps are usually necessary for functional surfaces, due to the inherently insufficient surface quality after AM. Control of the so called-additive subtractive process chain is dependent on several printing and post-processing strategies. In particular, heat treatment for hardening of the material and removal of residual stresses are often used as well as machining processes such as milling, drilling and grinding for finishing [4, 5].
Based on these challenges, the European research project "Ad-Proc-Add" investigates additive-subtractive manufacturing chains in order to obtain a detailed understanding of the dependencies and interactions of material and component properties of additively manufactured and reworked work pieces with regard to process parameters, manufacturing strategies and boundary conditions. The aim is to be able to consciously adapt geometry, surface and boundary zone properties via additive-subtractive production chains in order to be able to meet predefined requirements. This enables the targeted design and implementation of additive-subtractive production chains in various industrial applications.

One of the tasks in the “Ad-Proc-Add” project is to determine the influence of the LPBF process and the process chain on the material properties of additively manufactured components of the maraging steel 1.2709 (X3NiCoMoTi18-9-5). This material is often used for the production of metallic components for various applications [6]. In addition to the printing direction, the heat treatment is an essential aspect. Of special interest are the mechanical and structural properties of the additively manufactured parts, depending on the used process chain. The objective is to investigate a correlation between the AM parameters and the material properties. From this, statements about the design of the additive-subtractive process chains can be derived.

2. Experimental setup

2.1. Process chain

In order to get a detailed understanding of the correlations in additive manufacturing, the additive-subtractive process chain shown in Figure 1 was used. The LPBF process is used to produce components from the material 1.2709. After the AM process, the components were separated from the build plate. To modify the mechanical properties, the work piece was heat treated. The final step was the finishing by machining processes (e.g. milling, drilling), as well as surface functionalization, e.g. by hard coatings.

The following investigations will focus on different heat treatment procedures and their influence on the properties of the additively manufactured parts. A PVD coating deposition process was used to combine heat treatment and surface functionalization in a single step.

![Figure 1. Additive-subtractive process chain.](image)

2.2. Additive manufacturing

The LPBF machine Lasertec 30 SLM 2nd from the company DMG MORI Germany was used to produce the AM samples from the material 1.2709. For quality assurance, the powder used is examined for its composition and particle size distribution and it is determined that it is in the range of a typical LPBF powder with the diameters: D10 = 24.45 µm, D50 = 35.47 µm and D90 = 47.79 µm. Simple geometries (discs, hollow cylinders, rods) were produced for the basic analyzes. Used process parameters, based on the recommendations of the manufacturer of the AM system, are shown in Table 1. Regarding the AM process parameters, only the influence of the buildup-direction was investigated (see Table 1).
Table 1. LPBF process conditions and work piece geometry.

| Process conditions | work piece geometry |
|--------------------|---------------------|
| Material           | 1.2709              |
| Layer thickness    | 65 µm               |
| Atmosphere         | Argon               |
| Build plate temperature | 200 °C        |
| Laser power (hatch)| 275 W               |
| Scan speed (hatch) | 750 mm/s            |

2.3. Heat treatment and hard coating

After the AM process, the components were separated from the construction platform and the support structures were removed by blasting and grinding operations. For mechanical investigations and the coating deposition, 26 samples were manufactured, each in horizontal and vertical build up direction. Then half of the samples were age hardened at 490°C for 6 hours which is a common heat treatment for the steel 1.2709 [7]. Currently it is uncertain if a solution annealing at temperatures of 820-850°C before hardening is really necessary to achieve sufficient mechanical properties. Some researchers found that the solution treatment before hardening plays only a minor role in the fracture process [7]. So according to the aim of the current research, no solution treatment has been carried out. This is planned for further investigations, but only for comparison purposes.

In order to examine the influence of different heat treatment procedures on the work piece properties, a PVD hard material coating was used instead of the heat treatment. This combination makes it possible to modify the material properties as well as to change the surface with regard to defined properties such as wear resistance or friction coefficient. For the investigations, the following PVD coatings were chosen: Diamond-like carbon (ta-C), a nano-structured (nACo2), oxygen-containing (AlCrON) and a wear-protective coating (TiCN-LT). The coatings differ in their special properties and the coating temperature. Table 2 shows the selected coating systems, the deposition temperature and time for the specific coating thickness as well as special coating features. The coating deposition was done using an arc-PVD process on commercial coating systems π211 and π311 from the company PLATIT AG from Switzerland.

Table 2. Properties and features of used PVD-hard coatings [8].

| Coating  | Deposition temperature | Deposition time | Thickness | Hardness | Features                                      |
|----------|------------------------|----------------|-----------|----------|-----------------------------------------------|
| ta-C     | 110 °C                 | 370 min        | 0,8 µm    | 65 GPa   | Tetraedrical amorphous DLC Extremely high hardness |
| TiCN-LT  | 300 °C                 | 360 min        | 2,5 µm    | 34 GPa   | Wear protective coating Low friction coefficient |
| nACo2    | 480 °C                 | 365 min        | 2,5 µm    | 41 GPa   | Nanostructured wear protection coating High wear resistance |
| AlCrON   | 550 °C                 | 370 min        | 4,0 µm    | 39 GPa   | Oxygen containing coating High thermal resistance |
The various temperature-time profiles for coating are compared with conventional heat treatment in Figure 2. The depositing temperature and the temperature profile of the nACo2 coating as well as the AlCrON-coating are nearly comparable with the conventional heat treatment. The holding times are almost the same for all variants. Differences can be seen in the heating and cooling rates - the heating rates of the hard coating systems are higher (14-16 K/min) compared to the conventional heat treatment (6 K/min), the cooling rates are higher as well (hard coatings: 5-6 K/min, conventional HT 2 K/min). For the ta-C coating, the heating (4 K/min) and cooling rate (1 K/min) are lower compared to the conventional HT.

Figure 2. Temperature-time-profile of different hard coating deposition processes compared to conventional heat treatment for the material 1.2709.

2.4. Material and surface characterization
After additive manufacturing and the heat treatment as well as the coating deposition, the modified work pieces were characterized. Due to the large number of influencing parameters, the analysis is limited to essential parameters (hardness, strength). With regard to the mechanical properties of the AM work pieces, the notched impact strength at room temperature (pendulum impact tester PSd 300, company WPM Werkstoffprüfsysteme) and the hardness (DiaTestor 7521, company Wolpert) were determined according to the standards DIN EN ISO 6508-1, and ISO 148-1. An analysis of the failure behavior was also carried out on the fracture surface using a SEM (Phenom XL, company Thermo Fischer Scientific).

Furthermore the modified and coated surfaces were analyzed with regard to roughness (Taylor Hobson PGI 1500E, DIN EN ISO 4287:2009-11), coating hardness (Picodentor HM500, DIN EN ISO 14577-1) and coating adhesion (Rockwell indentation, DiaTestor 7521, DIN EN ISO 26443) to determine the influence of the different heat treatments on the mechanical properties of the AM work pieces. The abrasive wear resistance was also determined by spherical grinding with defined boundary conditions (ball on disc, grinding velocity 6m/min, grinding length 18 m, DIN EN ISO 26423). The influence of the heat treatment on the microstructure was determined on using SEM and EDX. The internal stresses of the components under the different thermal boundary conditions were also determined using X-ray diffraction (XRD) methods based on the sin²ψ method.

3. Results
3.1. Mechanical properties of the work piece
In the first step, the mechanical properties notched impact strength and hardness of horizontally (90°) and vertically (0°) printed samples were determined. The characterization was carried out on samples without and with conventional heat treatment (6h / 490 °C / air) after the AM process.
Figure 3 shows the hardness and impact energy of the analyzed samples for the different buildup directions with and without heat treatment (age hardening). The measured values correlate with information from the literature. For the work pieces, no significant difference in hardness between the different buildup directions can be determined. The heat treatment leads to a significant increase in hardness, which can be explained by the intermetallic segregations formed during age hardening [9].

With regard to the impact energy, there are small differences between the horizontally and vertically built work pieces without heat treatment. The specimens with a vertical direction (0° to the buildup direction) with a notch in the direction of the layer have slightly higher notched impact energies compared with a buildup direction of 90° with a notch vertical to the layer. As result of the heat treatment, the impact energy is reduced, and there is no longer any significant difference between the work pieces produced in different orientations. The reduced impact energy after the heat treatment correlates with the increase in hardness and is due to the formed intermetallic segregations, which limit the deformability of the material and lead to a more brittle material behavior.

![Figure 3. Hardness and notched impact strength of AM work pieces of 1.2709 with differently heat-treatment, comparison with information from literature [7, 10, 11].](image)

The fracture surfaces generated during the notched impact tests (Figure 4) can be used to give information about ductility. With a buildup direction of 0° without heat treatment, more dimples occur and the work pieces are more ductile. Heat-treated samples have fewer or smaller dimples, regardless of the buildup direction, and therefore have a significantly lower ductility [9]. These results are consistent to the measured impact energy.

![Figure 4. SEM images of the fracture surfaces of the notched impact work pieces with and without heat treatment.](image)
The results of the material characterization show that after the AM process, work pieces of the material 1.2709 with a buildup direction of 0° to the build plate have a higher notched impact energy (notch parallel to the layer) than with a buildup direction of 90°. In the hardened state, the hardness and impact energy are independent on the buildup direction.

3.2. Combination of heat treatment and hard coating
In order to examine the influence of different heat treatment procedures on the work piece properties, the heat treatment was replaced by a PVD coating deposition process. The resulting microstructure of the work piece as well as the work piece and coating properties were analyzed after each PVD coating process.

3.2.1. Microstructure. Figure 5 shows a cross-section through the structure of the differently heat treated and coated work pieces in a horizontal buildup direction. For a better visibility of grain boundaries and structural components, the structure was etched using the etchant Nital (solution of 3% HNO3 in ethanol). The grain boundaries of the individual layers are particularly visible in the variants without heat treatment and after the ta-C coating (deposition temperature 110 °C). The closer the deposition temperature meets the temperature of the conventional heat treatment the more comparable is the resulting microstructure to the conventional heat treatment. In particular, at a coating temperature of 480 °C, martensitic structures result that are comparable to those of conventional heat treatment.

The same results apply to a horizontal buildup direction. It can be concluded, that a PVD hard coating is suitable for replacing the heat treatment. For best results in terms of hardening of the coating substrate the coating temperature should be as close to 490 °C as possible. In the present case, the nano-structured nACo2 coating with a deposition temperature of 480 °C meet this requirement. The buildup direction has no influence on the resulting structure.

![Figure 5](image_url)

**Figure 5.** Microstructure of the additively manufactured and differently heat-treated / coated work pieces (horizontal build direction, 90° to build plate).

3.2.2. Mechanical properties. Figure 6 shows the hardness of the additively manufactured work pieces after coating deposition. It can be seen that work pieces have a low hardness of approx. 370 HV10 directly after the AM process. With a following martensitic age hardening (6h / 490 °C / air), the hardness increases to 550 HV10. Using a PVD coating, similar results can be achieved due to the analogous temperature profile. At temperatures of 110 °C and 300 °C, the hardness of 370 HV10 (110 °C) and 490-510 HV10 (300 °C) does not reach the level of conventional heat treatment; the usual hardness values can be achieved at temperatures of 480 °C and 550 °C. For 480 °C (coating nACo2), slightly higher hardness can be achieved based on the slightly higher cooling rate at the PVD process. Only small differences in hardness can be seen between the horizontal and vertical buildup direction.

The residual stresses in the surface near areas of the work pieces is shown in Figure 7. Due to the effort of the XRD measurement, only horizontal samples have been analyzed. On the surface
Compressive stresses are measured, these have a favorable effect for improved wear resistance. There are only small differences in the residual stress between the AM work pieces with and without conventional heat treatment, which are within the tolerance for XRD methods. For the PVD coated work pieces, higher internal compressive stresses result at lower deposition temperatures. At higher coating deposition temperatures, the internal stresses are reduced compared to conventional heat treatment. This indicates that the higher heating and cooling rates under vacuum conditions of the PVD coatings have a significant influence on stresses close to the surface. Further analyses are necessary to verify the results.

**Figure 6.** Hardness of the AM work pieces with different heat-treatments / coatings in different buildup directions.

**Figure 7.** Residual stresses (XRD) of the AM work pieces with different heat-treatments / coatings.

### 3.2.3. Coating and surface properties

PVD coating can be used to increase the wear resistance of the work pieces, therefore the surface and coating properties are important. Figure 8 shows the coating adhesion determined by Rockwell indentation test as well as an example of a Rockwell indentation for the different deposition temperatures. In comparison with coatings on conventionally produced high speed steel (HSS) material 1.3433, which is a common substrate material for PVD coatings, on the AM material 1.2709 the coatings show a lower coating adhesion (higher adhesion class). For the ta-C coating with the lowest deposition temperature, large-scale flakes and insufficient coating adhesion are visible. The reason for this are the high internal stresses of the coating due to the extremely high coating hardness (see also figure 9). All other heat treatments / coatings show adequate adhesion, the coating adhesion is enhanced with increasing deposition temperature. For the further coating development on AM produced work pieces, an improvement is necessary to ensure coating adhesion during the use of coated AM parts.

**Figure 8.** Coating adhesion (Rockwell indentation method) and sample Rockwell indentations for different coatings.
The hardness of the coatings with different deposition temperatures is shown in Figure 9. For the same coating system, there are only small variations with respect to the buildup direction and a previous heat treatment. The highest hardness of about 22-23 GPa shows the ta-C coating. The coating hardness for the ta-C coating can be increased to about 25 GPa by conventional heat treatment before coating deposition, because of the better support due to the higher substrate hardness. All other coatings show significantly lower coating hardness between 11 and 15 GPa. The hardness of coatings on AM work pieces is lower, compared with hard coatings on conventional base material due to different substrate properties (e.g. hardness, microstructure).

The wear resistance to abrasive wear is shown in Figure 10. Compared to an uncoated, additively manufactured component, all coatings reduce the wear rate (increase the wear resistance). The results show, that the ta-C coating can strongly reduce the abrasive wear. As well as it can be seen, that the buildup direction or a previous heat treatment has only a small influence on the wear rate.

4. Discussion and Conclusion
The analyses of different heat treatments of AM work pieces of the martensitic steel 1.2709 show that heat treatment is essential for optimal work piece properties. It could be shown that the heat treatment not only influences hardness and ductility, but also the properties of a subsequent coating. Hence, the complete additive-subtractive process chain with the specific treatment steps must be taken into account. Investigations have shown that both the buildup direction and the heat treatment have an influence on the mechanical properties of additively manufactured components. Work pieces with a buildup direction of 0° to the buildup plate have a higher notched impact energy (notch parallel to the layer) compared to a buildup direction of 90°. After the heat treatment, the hardness is increased from 32-36 HRC up to 51-52 HRC and the notched impact energy is reduced from 44-60 J down to 12-14 J while the buildup direction has no significant influence on these properties.

If the usual heat treatment for the material 1.2709 is replaced by a PVD coating, comparable hardness can be achieved. Here a deposition temperature of about 480 °C is necessary. The combination of heat treatment and coating deposition allows to set similar microstructures, but also to improve the wear resistance of the coated work pieces. With this properties application areas comparable to conventionally manufactured components can be used.

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References

[1] Vaneker T H J 2017 The role of Design for Additive Manufacturing in the successful economical introduction of AM Procedia CIRP 60 181-186

[2] Schmidt M, Merklein M, Bourell D, Dimitrov D, Hausotte T, Wegener K, Overmeyer L, Vollertsen F and Levy G N 2017 Laser based additive manufacturing in industry and academia CIRP Annals 66 561-583

[3] Riemer A, Leuders S, Richard A R and Kullmer G 2017 Optimierung der Werkstoffperformance lasergeschmolzener metallischer Werkstoffe. In: Additive Fertigung von Bauteilen und Strukturen ed H A Richard and B Schramm (Wiesbaden: Springer Vieweg) p 173-188

[4] Klocke F, Arntz K, Teli M, Winands K, Wegener M and Oliari S 2018 State-of-the-art Laser Additive Manufacturing for Hot-work Tool Steels Procedia CIRP 63 58-63

[5] Levy G N, Schindel R and Kruth J P 2003 Rapid manufacturing and rapid tooling with layer manufacturing (LM) technologies, state of the art and future perspectives CIRP Annals 52 589-609

[6] Moshiri M, Candeo S, Carmignato S, Mohanty S and Tosello G 2019 Benchmarking of Laser Powder Bed Fusion Machines Journal of Manufacturing and Materials Processing 3 85

[7] Bajaj P, Hariharan A, Kini A, Kürnsteiner P, Raabe D and Jägle E A 2020 Steels in additive manufacturing: A review of their microstructure and properties Materials Science & Engineering A 772 138633

[8] Compendium, Platit AG 2021, 62nd Edition

[9] Kučerová L, Zetková I, Jandová A and Bystrianský M 2019 Microstructural characterisation and in-situ straining of additive-manufactured X3NiCoMoTi 18-9-5 maraging steel Materials Science & Engineering A 750 70-80

[10] Simson, T, Koch J, Rosenthal J, Kepka M, Zetek M, Zetková I, Wolf G, Tomčík P and Kulhánek J 2019 Mechanical properties of 18Ni-300 maraging steel manufactured by LPBF Procedia Structural Integrity 17 843-849

[11] 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 19 746-758