Investigation on Additive Manufacturing of tungsten carbide-cobalt by Selective Laser Melting

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Abstract
The production of special interior contoured tools made of cemented carbide is a time- and cost-intensive multistage sintering process. An alternative for economic, flexible and automated production is provided by a laser-based, additive manufacturing Selective Laser Melting process (SLM). In a shortened process chain near-net-shape and special interior contoured tools can be manufactured energy- and resource-efficiently in small batch sizes. An increase of the tool life is achieved by the use of wear resistant and tough cemented carbide material. This paper focuses on the process behavior of the agglomerated and pre-sintered tungsten carbide-cobalt (WC-Co) powder material in the SLM process. Depending on the exposure parameters, various types of micro structures can be generated and the original material profile can be significantly changed during the laser material interaction.

Keywords: additive manufacturing, selective laser melting, cemented carbides, tungsten carbide-cobalt

1. Introduction
The manufacturing of inner contoured special tools using carbides is a time- and cost-intensive multistage process. After the carbide has been directly pressed into a disk, a complex multistage cutting process is applied in order to achieve the required geometric complexity. The integration of cooling channels in conventional production leads to great difficulties because it involves high initial costs and is only achievable to a limited extent on a large scale. Nowadays, there is an increasing demand for individual products. Companies therefore require manufacturing technologies which are customizable and allow for the production of an increasing product range. One technology which meets the required criteria is Selective Laser Melting. In a shortened process chain, near-net-shape and inner contoured special tools can be manufactured in batches, thus saving energy and resources. Furthermore, there is a significant reduction in the conventional preparatory work as well as production stages. An increase in the freedom of design as well as the degree of innovation and efficiency in production can be achieved.

Selective Laser Melting is based on a remelting process in which the compaction of the material is determined only by the flow and wetting behavior of the melt [1]. In order to avoid undesired consequences such as material defects such as pores and cracks, delamination, component warping, uncontrolled changes of chemical and physical component properties, the exposure parameters (Figure 1) must be optimized, taking into account their mutual interactions.

The intensity of the laser beam and the specific material parameters determine the absorbed optical energy during the laser material processing. This energy is available as process heat in the material for further phase transformation processes [2].

Figure 2 shows a schematic representation of the three phases in the SLM process. The coupled laser energy primarily leads to local melting of the powder material. However, the powder grains in the outer area of the beam absorb less energy than the grains in the beam center. Below
the melting pool, the already solidified material layers and powder grains in the border areas of the laser beam are merely heated. In the center of the Gaussian laser beam, where the highest intensity is present, evaporation temperature of the material is however exceeded and pronounced spatter formation takes place. Compressive and reaction forces are thereby caused through the flowing steam [3, 4], which casts out the melting partially from the molten pool and causes spatter activity. This phenomenon is indeed typical for SLM, but is tolerated only in the context of robust processes and in the attainment of high-quality components. An excessively strong spatter formation affects the stability of the melting and can be an indication of a non-optimal energy input.

A robust and reliable processing of carbide materials using SLM therefore requires an in-depth interaction analysis of the influence factors and of the physical and chemical result quantities. The phenomenon of evaporation is to be taken into account, as this causes a change in the chemical composition of the processed tungsten carbide material [5].

The first researches on the laser sintering of tungsten carbide-cobalt were carried out at the University of Leuven in Belgium since the beginning of the current century. Mixed and mechanically alloyed WC-Co-powder were used which had been processed with low laser sintering typical volume energy densities. Generative built components indicated hereby a relative density $\rho_{rel}$ of 40%, therefore had to be redesified through time-consuming post-processes, mostly through infiltration, sintering, or hot isostatic pressing. [6, 7, 8, 9]

Based on the initial investigation, a thesis has been established that a complete dense sintering of WC-Co powder cannot be obtained in the laser sintering process. Therefore an approach was pursued at the University of Utah by means of laser sintering in order to generate a microstructure with open pores and subsequently closing them through infiltration with low-melting bronze. After the parameter optimization, 63% of the relative density $\rho_{rel}$ of additive manufactured components could be achieved. A subsequent infiltration of the components with bronze led to final relative density of 96%. [10]

Although it was possible to achieve a significant reduction in the porosity through subsequent infiltration within the scope of the mentioned research projects, the mechanical material properties were classified as deficient, so that ultimately no industrial use could follow [6, 7, 8, 9, 10].

A significant breakthrough in the single-stage compression of the agglomerated and (before) sintered WC-Co powder using beam melting was achieved at research institutes of the Fraunhofer IPT in Aachen, Fraunhofer IPK in Berlin, IWB in Munich and BIAS in Bremen. A relative density to 98% could be increased by parametric studies [5, 11, 12, 13].

All project-investigated factor levels of laser power $P_L$ and scanning speed $v_s$ are summarized and presented in Figure 3. It is clear that so far no optimal process parameters for the beam melting of WC-Co were found and thus the qualification phase of WC-Co has not yet been completed. In the sub-optimal process window (100 ... 200 W and 20 ... 100 mm/s) densities of 95 ... 98% were indeed achieved, however all manufactured components more or less indicated pronounced cracks. With rising energy input, the crack tendency in addition to the relative density increased resulting in delamination of components [5, 12].

The aim of this study is to investigate the process behavior of WC-Co material considering the evaporation effect in SLM process. The primary objective is to understand the interaction
between different factors influencing the SLM process by using appropriate quality tools.

### Nomenclature

| Symbol | Definition |
|--------|------------|
| \(d_f\) | focus diameter, μm |
| \(E_v\) | volume energy density, J/mm³ |
| \(E_i\) | focus position, mm |
| \(h_s\) | scan line spacing, mm |
| \(HV\) | Vickers hardness, 1 |
| \(I\) | layer thickness, mm |
| \(P_L\) | laser power, W |
| \(l_z\) | layer thickness, mm |
| \(l_{SV}\) | scan vector length, mm |
| \(I\) | laser beam intensity, W/cm² |
| \(R_z\) | average surface roughness, μm |
| \(\rho_{rel}\) | relative density, % |
| \(\rho\) | relative density, % |
| \(\mu_m\) | chemical composition, particularly on the Co-content of the laser molten carbide specimens. For this purpose cuboid formed specimens are generated using spherical WC-Co 83-17 powder. The high content of Co should minimize the cracking and maximize the density of generated parts [5]. The metrological determination of the relative density \(\rho_{rel}\) is carried out using image analysis of metallographic grindings in order to obtain secure knowledge on this critical quality feature. The energy dispersive x-ray spectroscopy analysis is used to determine the chemical composition of the ground specimens. |
3. Experimental results

3.1. Relative density $\rho_{rel}$

The changes in the levels of average values can be compared qualitatively with each other in the main effect diagram (Figure 6). The laser power $P_L$ affects the response variable $\rho_{rel}$ most strongly. In addition, a main effect is present in the scanning speed $v_s$, line spacing $h_s$ and layer thickness $l_z$. The layer thickness $l_z$ was analyzed at the usual levels of 30 $\mu$m and 50 $\mu$m. With increasing layer thickness, an apparent reduction of the relative density can be seen. Furthermore, a highly curved function of the laser power $P_L$ is detected, indicating an optimum in the process window applied in the study. The interaction of factors can be described in a contour diagram (Figure 8). The laser power $P_L$ affects the response $\rho_{rel}$, line spacing $h_s$ and focus position $f_z$ show a weak interaction. As the focus position changes from negative to positive values, it leads to an increase in the line spacing which reinforces the effect. The effect increases between the focus position and the scanning speed is not as pronounced. The present interactions can be well illustrated by means of the common intersections of the line segments in Figure 10.

Fig. 6. Main effects plot of relative density $\rho_{rel}$ of generated specimens

In the process optimization, significant interactions between the factors must be considered. A highly significant positive interaction exists between $P_L$ and $v_s$ as well as $v_s$ and $l_z$. A negative interaction exists between $P_L$ and $h_s$. In the interaction diagram, the line segments of the laser power and scanning speed deviate particularly strong from parallelism (Figure 7). In addition, a weak positive interaction exists between $P_L$ and $h_s$.

Fig. 7. Interaction plot of relative density $\rho_{rel}$ of generated specimens

The interaction of factors can be described in a contour diagram. In this diagram, the functional relationship between the response $\rho_{rel}$ and two factors is represented in two dimensions. By means of the contour diagram in Figure 8, the process window in which a high relative density is achieved can be located. It has become clear in previous presentations that a high relative density can be expected from a high laser power, a low scanning speed, a small line spacing and a low layer thickness.

Fig. 8. Contour plots of interacting factors which affect the density $\rho_{rel}$ of cemented carbide parts

3.2. Cobalt content

As a result of the laser based processing of tungsten carbide-cobalt material, its original chemical composition changes [5]. The question which process parameters have an influence on this, has not yet been researched in detail. An important indicator for material cohesion is the binder content in the cemented carbide. This is detected by energy dispersive X-ray spectroscopy on the cross-section polish of each specimen and statistically evaluated in this study.

From the main effect diagram (Figure 9) it can be extracted that the laser power $P_L$ influences the evaporation of the cobalt binder the most. The second largest leverage is caused in the cemented carbide. This is detected by energy dispersive X-ray spectroscopy on the cross-section polish of each specimen and statistically evaluated in this study. From the main effect diagram (Figure 9) it can be extracted that the laser power $P_L$ influences the evaporation of the cobalt binder the most. The second largest leverage is caused in the cemented carbide. This is detected by energy dispersive X-ray spectroscopy on the cross-section polish of each specimen and statistically evaluated in this study.

Fig. 9. Main effects plot of cobalt content of generated specimens

In addition, scan line spacing $h_s$ and focus position $f_z$ exhibit a strong interaction while scanning speed $v_s$ and focus position $f_z$ show a weak interaction. As the focus position changes from negative to positive values, it leads to an increase in the line spacing which reinforces the effect. The effect increases between the focus position and the scanning speed is not as pronounced. The present interactions can be well illustrated by means of the common intersections of the line segments in Figure 10.

A plausible representation of the detected interactions is shown in a response surface diagram (Figure 11). In order to minimize cobalt evaporation, scanning speed and line spacing must be increased and the focus position held in the negative
range. However, the required factor level combinations lead to an extremely low material density, which is between 52.6% and 62.3% at a layer thickness of 30 μm.

3.3. Porosity and cracks of multilayer structure

A qualitative comparison of the generated multilayer structures (Figure 12) confirms conclusions compiled within the statistical evaluation:

- An increasing in laser power enhances the continuity of the melting pool. Individual tracks and layers are seamlessly joined together. Cavities are closed, and the porosity decreases.
- A reduction in scanning speed causes an expansion of the melting pool and leads to a significant increase in density.
- With smaller scan line spacing, the degree of overlap of individual tracks increases and fewer pores arise in the overlapping regions.
- A variation of the focus position causes no noticeable change in the porosity.

All specimens show a pronounced tendency to crack regardless of varying process parameters. In porous structures, the single cracks are mostly between the pores. In dense test specimens, the cracks run considerably more continuously than in porous ones.

Fig. 10. Interaction plot of cobalt content of generated specimens

There is a clearly pronounced conflict concerning response optimization: As a result of the increase in energy density, a dense and brittle composite material is generated. If the energy input is reduced, substantial porosity and a high cobalt content is maintained.

Fig. 11. Contour plots of interacting factors which affect the cobalt content of cemented carbide parts

3.4. Material structure

Parameter variation similarly causes an evident change in the microstructure (Figure 13). To sum up, the following conclusions can be retained concerning structural analysis:

- An increase in laser power reinforces the development of single melting beads. In this case, penetration of the laser beam increases significantly.
- With increasing scanning speed, a certain inhomogeneity can be recorded in the laser melted structure.
- As a result of reduced line spacing, the number of the melting beads increases and the area between individual melting tracks decreases.
- A variation of the focus position causes no noticeable change to the carbide structure.

All carbide specimens, having a dense composite material due to sufficiently large volume energy density, possess an evident and layered periodic inhomogeneity. Here it concerns the molten structures which arise after the cooling of the local melting pool (Figure 14). At these points, extremely low cobalt content was detected by means of EDX point analysis, with a poor amount of 1% at a laser power of 150 W. All carbide components in the molten beads accordingly must have had a fully liquid state. The temperature of the molten pool must have exceeded 2900 °C, since cobalt initially starts to evaporate at this value, thus escaping the process zone.
Layer thickness $l_z$

They start to grow while the molten carbides cool. Due to the molten beads are the strongest thermally activated ones. Crystal-growth processes take place. The WC grains bordering the solidification front, is located below the melting pool, where toughening of the material and therefore minimize crack growth.

The WC grains of the material embrittlement.

A typical sintered structure is generated between the layers. The outer contour area of the component was exposed with the molten beads, which contains only a small weight percentage of cobalt. This results in an enormous material embrittlement.

A typical sintered structure is generated between the layers and the melting tracks. It contains approximately the initial amount of cobalt binder. In addition, no changes in the carbide grains are visible in the intermediate areas.

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4. Validation of results

Within the scope of the validation studies, first special tool prototypes could be laser molten from carbide powder (Figure 15). Special tools are an important subcategory of cutting tools, which are often used in industrial production processes. Additively manufactured inserts or drills with integrated cooling channels have the potential to increase productivity enormously. Through internal and conformal cooling of the tools, dry machining without cooling lubricants is possible. This reduces pollution on the environment as well as on the machine operator. In addition, heat accumulations can be eliminated from the tool. Cycle period as well as warping of the work piece can be diminished. Wear resistance and ductility of cemented carbide favor an increased product life during machining production.

The validation studies aim to check the predicted quality features for the optimized factor level combinations. The results of the validation test are presented in Figure 16. Both predicted response variables are clearly within the confidence intervals. The estimated regression models for the relative density $\rho_{rel}$ and the cobalt content thus describe the correlation between input and output variables in sufficient approximation.

The outer contour area of the component was exposed with a low energy input ($E_V = 185 \text{ J/mm}^3$) in order to sustain the toughness of the material and therefore minimize crack growth. Thereby a typical sintering micro structure is generated. Due to the low energy input as a result of low laser power and high scanning speed, mainly liquid phase sintering
processes take place there. The original content of cobalt binder in the laser-sintered structure remains almost constant. In addition, no changes in the carbide grains can be seen. An adverse effect however is the distinct porosity in the contour area, thus a lower resistance against the permeation is available.

Fig. 17. Characteristic of the laser molten WC-Co structure in different areas

5. Conclusion

Numerous interactions were statistically verified between the investigated process parameters. In order to achieve an effective optimization of the critical quality characteristics, the intensity and direction of the respective 2-factor-interaction must be considered. A summary of the gained findings can be obtained from Table 1.

A clear conflict between the output variables \( \rho_{\text{rel}} \) and Co has been identified. Their optimization directions are opposed to each other. This means that either a high relative density can be reached by a large energy input (PL, vs) or a high cobalt content can be maintained by a low energy density (EV). A simultaneous optimization of both variables is only possible to a limited extent.

In conclusion, depending on the exposure parameters used, various types of micro structures can be generated with laser melting:

- A high energy density \( E_V \) of 1667 J/mm\(^3\) results in dynamic remelting and evaporation processes which contribute to the development of a coherent and closed molten bath in the interaction zone of the laser beam and the powder material. Although this results in a high relative density, it simultaneously leads to strong embrittlement of the processed tungsten carbide material. Thermally induced cracks can spread unobstructed through the brittle structure.

- A low energy density \( E_V \) of 185 J/mm\(^3\) leads to unwanted residual porosity. The original content of cobalt binder in the laser sintered structure remains approximately constant, whereby the toughness and thus the resistance to fracture or crack growth is preserved. The microstructure exhibits an overall homogeneous composite as a result of the liquid phase sintering process.

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