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Investigation of In-Situ Low Copper Alloying of 316L Using the Powder Bed Fusion Process

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Abstract: This study investigated the allowability of materials in the laser powder melting process, with a focus on powder mixing as a means of adjusting the material composition quickly and cost-effectively. By mixing different powders, a desired alloy can be created during additive processing without the need to produce new powder, which can be expensive. However, one of the main challenges in this process is the segregation of powders, which can lead to non-homogeneous alloys. To address this challenge, the study examined the use of a single component 316L mixed with 1% and 5% copper powder in the additive processing. The results showed that homogeneous components with a uniform and targeted copper content could be produced. However, the mechanical-technological properties of both alloys were lower than those of 316L in situ. To optimize and extend this study, further investigation could be conducted to improve the homogeneity of the powder mixture and to enhance the mechanical-technological properties of the alloys produced. This could involve exploring different alloy designs, optimizing the laser powder melting process parameters, and using advanced characterization techniques to gain a deeper understanding of the microstructure and properties of the alloys. By addressing these challenges, the laser powder melting process could become an even more promising method for producing customized alloys with tailored properties.

Keywords: additive manufacturing; powder bed fusion; in situ alloying; 316L; copper

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1. Introduction

Powder Bed Fusion (PBF) is an advanced additive manufacturing technology that offers a flexible and efficient way to create complex three-dimensional structures from metallic materials. This technology works by applying fine powder coatings in layers and then melting them locally with a moving laser beam, creating melt tracks in the powder bed that overlap to form the individual layers [1]. This layer-by-layer production process eliminates the need for tooling and enables component complexity to be decoupled from production complexity, providing greater flexibility, better material utilization, and shorter production times [2]. One significant advantage of PBF technology is its sustainability, which has become increasingly important in recent times. Unlike conventional manufacturing processes, where a significant proportion of the material is lost or cannot be reused due to waste, PBF enables much higher material utilization. Used metal powder can be sifted and returned to the process, minimizing waste and saving resources. This sustainability aspect also contributes to the economic viability of PBF as a manufacturing process [3].

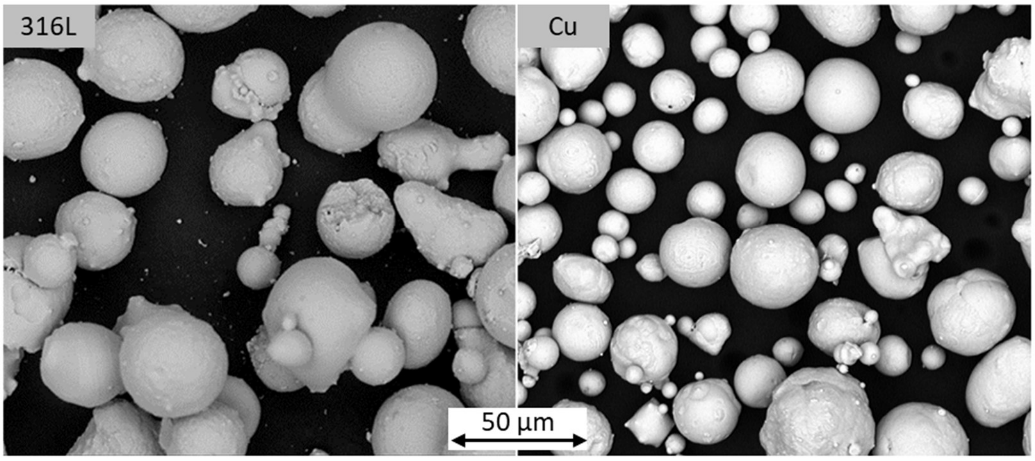
However, a potential limitation of PBF is its material selection. To be used in the PBF process, the material must first be converted to powder form, which often requires remelting followed by gas atomization. This process can limit the flexibility of the process in terms of material selection. Future research could investigate alternative powder production methods that allow for a wider range of materials to be used in PBF, further enhancing the technology's versatility and potential for customized manufacturing.

In order to address this issue, researchers have explored the use of in situ alloying, where different powders are mixed together and the desired alloy is created during the PBF process. However, previous studies have shown that powder mixtures tend to de-mix during the manufacturing process, highlighting the need for a more effective approach [4,5].

In addition, the non-uniform powder composition can significantly disrupt the manufacturing process itself [6]. Since the characteristics and properties of alloys can be optimized by adding a few masses percent of different elements, it is a promising approach to alloy an existing powder material with a few masses percent of one element, thereby reducing the risk of segregation described above. This is an interesting possibility, as alloying an existing alloy is a suitable method to optimize material characteristics for certain applications [7,8]. The use of computer-aided simulation methods for alloy design is of particular interest in this area of research [9]. There are a few examples in the literature where 316L has been treated with WC. For example, Yin et al. [10] investigated the effects of adding WC particles to the microstructure of 316L Al alloy by selective laser melting (PBF). Hu et al. [11] reported the use of Y_2O_3 particles to improve the ductility of 316L alloys. Zhou et al. [12] also reported that more nucleation sites were available after the addition of W powders, which limited grain growth and the formation of finer grains. As a result, the mechanical properties were improved. Furthermore, a powdery magnesium alloy with significantly smaller manganese and tin powder was alloyed in and successfully additively laser machined [13]. Han et al. [14] used graphene nanoparticles to improve the strength of 17.9 wt.% of printed 316L without affecting the ductility compared to 316L. There are examples in the literature of low levels of copper in 316L reducing the formation of bacteria by releasing copper ions on the surface of the material. For example, a copper content of 3.77 mass percent in [15] led to a broad-spectrum antibacterial effect against Staphylococcus aureus, Escherichia coli, and Staphylococcus epidermidis. In reference [16] it was shown that copper contents of 0, 2.5, and 3.5 mass percent in 316L lead to the release of copper ions on the surface. In the study, the manufactured 316L-Cu alloys had an austenitic microstructure and no obvious change to the pure 316L could be detected by the addition of copper. Furthermore, an improved corrosion resistance of the material was observed. In references [2,4,9,17], mass percent copper was added to 316L sintered components, which also led to an improvement in corrosion resistance. It is generally assumed that anodic dissolution is suppressed by the deposition of elemental Cu on the steel surface [18]. In the long term, it should be possible to produce 316L-Cu alloys using the PBF method, whereby the already good corrosion resistance of additively produced 316L [19] should be optimised. For this purpose, the manufacturability in the in-situ process is to be investigated with powder mixtures of 316L with a mass content of less than 10 wt.%. In this study, for the purpose of an initial feasibility investigation, powdered 316L was alloyed with one and five mass percent copper powder to produce corresponding alloys using the PBF process. The flow and segregation behavior of the powder blends was investigated, and production parameters were adjusted based on a melt track analysis. The mechanical properties and microstructure, including chemical homogeneity, were then investigated and compared to pure 316L. The main focus of this study was to achieve a uniform alloying of 316L and copper, with the results showing that homogeneous components could be produced with a uniform and targeted copper content.

2. Materials and Methods

In this study, gas-atomized powders of 316L and Cu were used to investigate the uniform alloying of 316L and copper in the PBF process. The 316L powder was obtained from m4p material solutions GmbH in Austria, while the Cu powder was obtained from Schlenk Service GmbH & Co. KG in Germany. The morphology and particle size of the powders were analysed using Scanning Electron Microscopy (SEM) and are shown in Figures 1 and 2, respectively.



(**a**) (**b**)

Figure 1. SEM Images of the 316L-(**a**) and the Cu-powder (**b**).

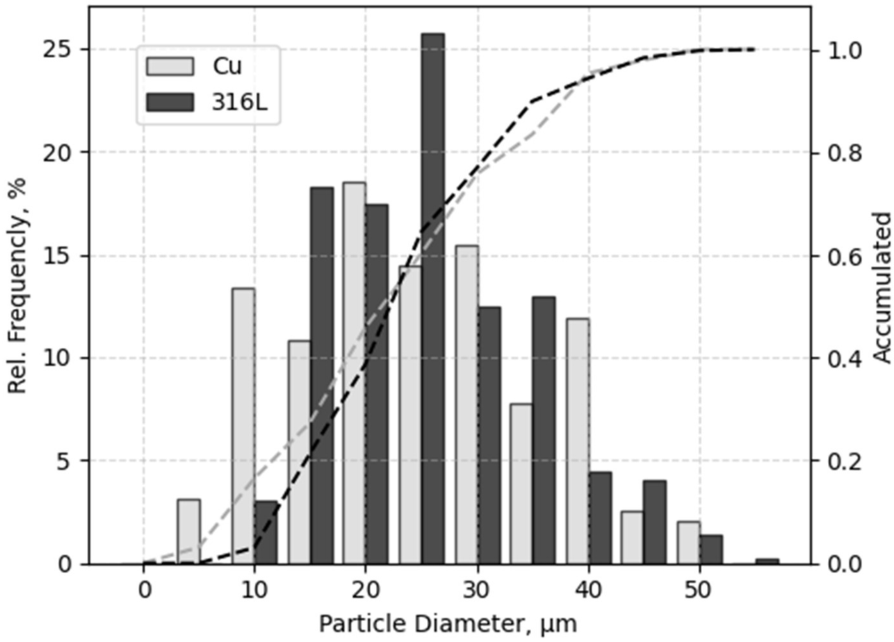


Figure 2. Distribution of powder particle sizes.

The average particle size of 316L and Cu powders was found to be (23.7 ± 9.1) µm and (24 ± 14) µm, respectively. The powders were then used in the MLab R system (Concept Laser, Bavaria, Germany) for the additive manufacturing process. Nitrogen was used as an inert gas in all tests to prevent oxidation of the powders during the manufacturing process. The use of nitrogen is common in PBF processes to prevent contamination and maintain the quality of the final product. The combination of these specific powders and the use of the MLab R system and nitrogen gas provided a controlled and standardized environment for the investigation of the alloying of 316L and Cu.

The experimental design, as shown in Figure 3, involves the use of three different groups of 316L powder. The first group is mixed with 1 wt.%Cu, while the second group is mixed with 5 wt.% Cu. The third group is left untreated and serves as a reference. The aim of the investigation is to determine the effects of alloying 316L powder with different percentages of copper on the properties of the resulting alloys. The mixed powders were prepared using a 3D shaker mixer for 30 min to ensure uniform distribution of the copper particles in the 316L powder. The mixer operates by combining a three-dimensional shaking motion with rotational movements to provide thorough mixing of the powders.

This method is commonly used in the production of alloys through powder metallurgy techniques. The use of different percentages of copper allows for the investigation of the effect of varying levels of alloying on the properties of the resulting alloys. The mixing process was carefully monitored to ensure that the powders were mixed thoroughly, and that no segregation occurred during mixing.

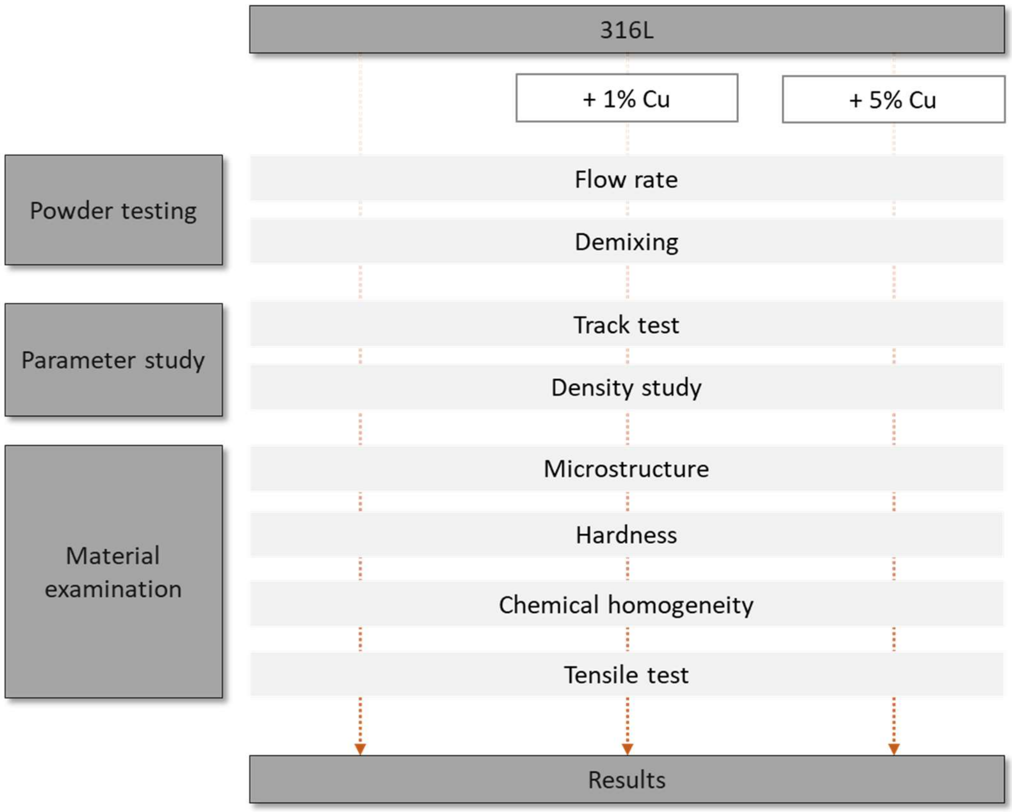


Figure 3. Experimental design.

To assess the behavior of the powders during layer application, a flow analysis in accordance with the guidelines of the German association of engineers on material under inspection in PBF production (VDI 3405) [20] was carried out using a Ø 5 mm powder nozzle. Furthermore, the powders were examined after 30 min on a vibrating plate to assess segregation. These tests provided a good estimate of the behavior of the powders during layer application in additive manufacturing. Powder samples of approximately 200 g were carefully observed to evaluate the flowability of the powders during the printing process. The results of the flow analysis showed that the mixed powders exhibited good flowability and no signs of segregation.

In addition to evaluating the behavior of the powders during layer application, melt track studies were also conducted to investigate the effect of the starting material on the manufacturing process and the manufacturing parameters. For parameter adjustment, eight melt tracks of 20 mm length were applied to rolled sheets of 316L for each powder set. The evaluation was conducted optically in plain view and by metallographically prepared cross section. A laser beam power of 90 W and a scan speed of 800 mm/s were used. Based on the measured melt track widths, the parameters for further investigations were adjusted.

Cubes of $10 \times 10 \times 10$ mm^3 were produced to investigate the density of the fabricated parts. The cubes were hydrostatically weighed to determine the density of the parts. The fabricated cubes were further used to investigate the microstructure in both the longitudinal and transverse directions. For this purpose, the samples were metallographically treated for etching at 70 °C with a V2A etchant (50 wt.% HCl, 10 wt.% HNO_3, 40 wt.% H_2O). They were also subjected to a Vickers HV30 hardness test to assess the hardness of the parts.

To study the chemical composition and homogeneity, samples of 20 × 20 × 2 mm^3 were prepared and analysed by optical emission spectrometry (OES) and energy dispersive X-ray spectroscopy (EDX) mapping.

Finally, round tensile specimens were tested according to VDI 3405 [21] on a universal testing machine to investigate the mechanical properties of the powder blends. The results of the tests showed that the mixed powders had poorer mechanical properties compared to the untreated powder.

3. Results

3.1. Powder Testing

In the flow test, a small amount of 316L powder was poured into a 5 mm diameter funnel and the time taken for the powder to flow through the funnel was measured. The results show that the 316L powder had the shortest flow time: (7 ± 0) seconds were required to flow through the funnel. In contrast, the 316L + 1 wt.% Cu powder required (17 ± 1) seconds, whilst the 316L + 5 wt.% Cu powder required (14 ± 2) seconds to flow through the same hopper. However, no significant differences were found between the powders in the segregation test. Segregation is the segregation of particles in a powder bed due to differences in particle size, density, or shape. The powder samples were vibrated on a vibrating plate for 30 min and the segregation was evaluated visually.

3.2. Parameter Study

The widths of the fusion lanes for each powder set were measured and recorded in Table 1. These measurements were crucial in designing the lane overlap, which was set to 30 wt.%. Additionally, the remaining parameters of the set and the material densities achieved with them were also recorded. Figure 4 illustrates a cross section of a melt track, which was used to evaluate the quality of the fusion process. The cross-section images were analysed thoroughly, and no significant differences were found between them. Similarly, the top view measurements of the melt tracks also showed no significant differences.

Table 1. Results of the parameter study.

	316L	316L + 1 wt.% Cu	316L + 5 wt.% Cu
Laser power [W]		90 W	
Scan speed [mm/s]		800 mm/s	
Layer thickness [μm]		25	
Measured track width [μm]	88 ± 3	83 ± 9	85 ± 8
Hatching (chosen) [μm]	62	58	59
Density [g/cm^3]	7.996	7.98	7.68
Relative density [%]	99.95	99.63	95.38

The investigation of the melt track width revealed a reduction in width for the two powder blends in comparison to the untreated 316L powder. The reduction in width is approximately 15 wt.% for both powder blends, and the melt track depth remains constant at approximately 70 μm for all three samples. This reduction in width is attributed to the lower laser absorption rate of copper compared to 316L. Additionally, the increased scatter is due to the inhomogeneous powder composition. An example of one of the examined melt traces in the metallography-prepared cross-section is shown in Figure 4, which shows the anisotropy created by the powder bed fusion process. To compensate for the change in melt track width, the hatching was adjusted accordingly. During production, there were no anomalies or defects observed.

The density of the finished parts was determined using Archimedean weighing, whilst the ideal density was calculated based on the chemical composition. The density decreases as the copper content increases, which is partly due to the fluctuating melting curves and partly due to the poorer flowability of the powder. The powder blend with 1 wt.% Cu showed a density of 98.7% of the ideal density, while the powder blend with 5 wt.% Cu showed a density of 96.4% of the ideal density.

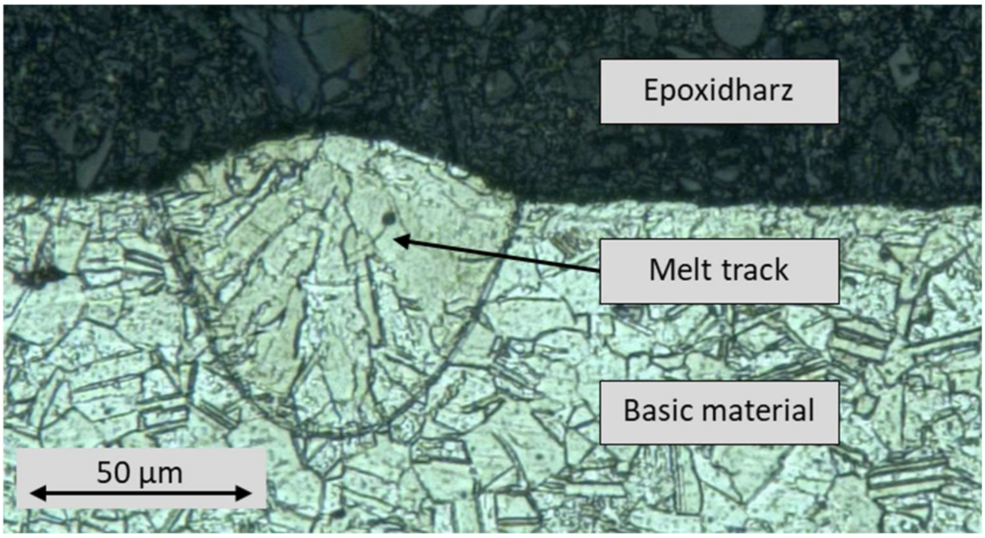
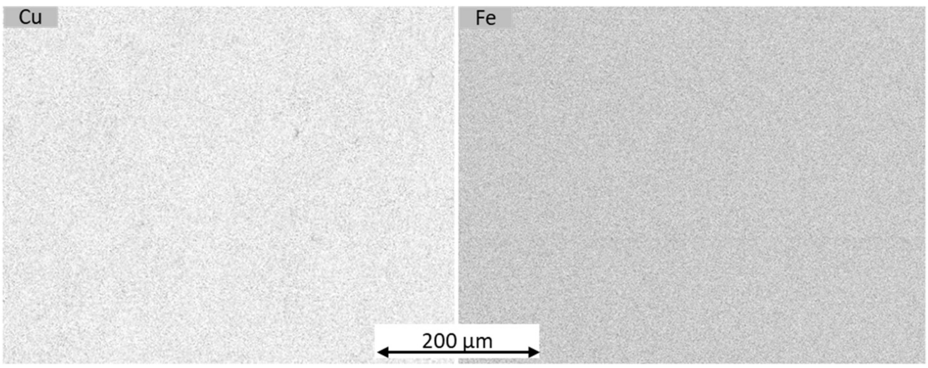


Figure 4. Image of a metallographic cross-section of a melting track.

3.3. Material Examination

The analysis of the chemical composition of the fabricated samples is crucial to evaluate the success of the alloying process. Table 2 shows the measured chemical composition of the manufactured samples. The results show that the 316L powder was effectively alloyed with copper powder. The 316L + 1 wt.% Cu sample had a copper content of (1.002 ± 0.051) wt.%, while the 316L+ 5 wt.% Cu sample had a copper content of (5.22 ± 0.19) wt.%. The low standard deviations indicate that there were no significant fluctuations in the copper content, suggesting that the alloying process was successful. In addition, an EDX mapping was performed on a 316L + 5 wt.% sample, as it showed a stronger scattering of the Cu content. Figure 5 illustrates the copper content on the left (Figure 5a) and the iron matrix of 316L on the right (Figure 5b) for comparison. The mapping confirms the homogeneity of the copper distribution, indicating that the alloying process was uniform and achieved the desired copper content.



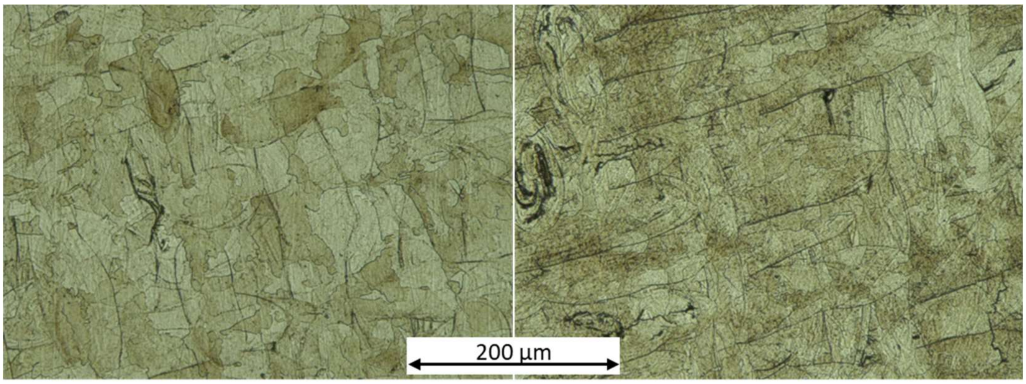
(**a**) (**b**)

Figure 5. EDX-mapping of the 316L + 5 wt.% Cu sample. (**a**) shows the copper content and (**b**) the iron matrix of the 316L.

Table 2. Material examination.

	316L	316L + 1 wt.% Cu	316L + 5 wt.% Cu
Cu [wt.%] (n = 13)	0.048 ± 0.001	1.002 ± 0.051	5.22 ± 0.19
Hardness HV 30	221 ± 5	209 ± 2	197 ± 5
Yield strength [MPa]	504 ± 8	493 ± 9	476 ± 12
Tensile strength [MPa]	576 ± 5	547 ± 15	536 ± 13
Elongation [%]	22.0 ± 0.6	20.6 ± 0.4	12.8 ± 0.4

The microstructure shown in Figure 6a depicts 316L + 1 wt.% Cu, while Figure 6b shows 316L + 5 wt.% Cu. Apart from the typical microstructure of the PBF process, build-up defects are visible in Figure 6a, which are even more clearly visible in Figure 6b. These defects might be caused by improper laser settings or an insufficient supply of powder, leading to a discontinuity in the melt track.



(**a**) (**b**)

Figure 6. Macrostructure of the in situ alloyed materials manufactured around PBF; (**a**) with 1 wt.% Cu and (**b**) with 5 wt.% Cu.

The experimental data from the mechanical–technological tests are presented in Table 2, while the results of the tensile test are shown in Figure 7.

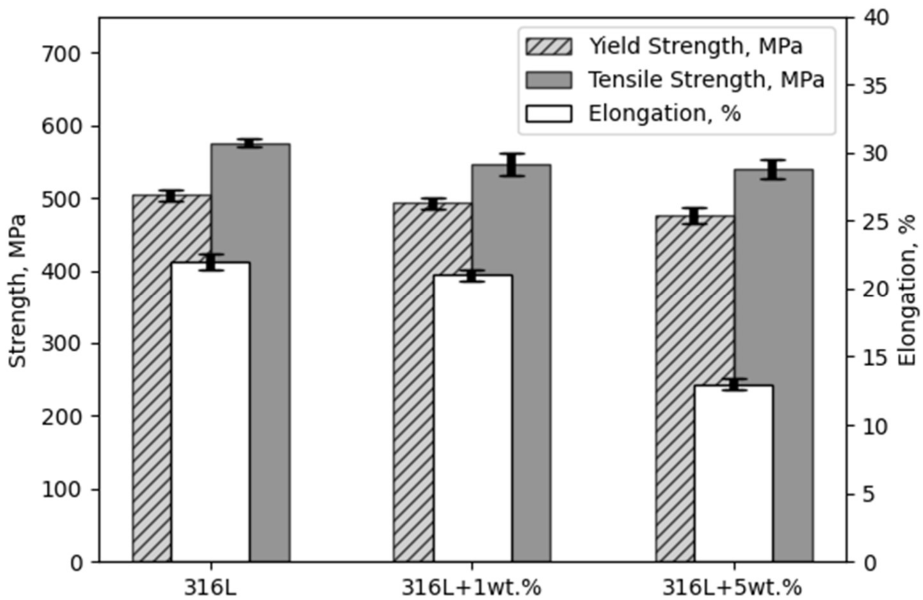


Figure 7. Results of the tensile test.

4. Discussion

4.1. Powder

These studies indicate a significant deterioration in the flowability of the powders due to the addition of copper. The longer flow time of the powders containing copper can be attributed to the fact that copper is a denser material than 316L and, therefore, has a higher flow resistance. No settling of the heavier copper powder on the bottom was observed in the segregation test, indicating good workability. The poor flowability of the copper-containing powders observed in the flow test is consistent with the segregation test results, as poor flowability can lead to less efficient packing and therefore less segregation. Overall, the results indicate that the addition of copper has a negative effect on the flowability of the powders, but does not significantly affect the segregation behavior.

4.2. Parameter

These results indicate that the chosen manufacturing parameters were suitable for all powder blends, and no significant differences were observed between them, suggesting that the addition of copper did not significantly affect the fusion process.

This decrease in density with increasing copper content could affect the mechanical properties of the fabricated parts, and further investigations are necessary to assess this effect.

4.3. Material

The investigation of the EDX mapping revealed some small accumulations of copper in localized areas where intermixing occurred. Despite this, no correlation between the chemical composition and the build-up direction was observed, indicating that there was no segregation of the powder due to vibrations in the PBF system. If such segregation had occurred, there would have been a constant change in the copper content over the z-axis due to increasing mixing during continuous production. The PBF system used in this study has a powder chamber in which the powder is stored and not fed through tubes or similar structures. However, it is uncertain whether similar results can be obtained using a tube-fed system, as there is a higher risk of segregation with such systems. These results are also confirmed by the optical emission spectrometer analyses performed, as there was no significant variation between different measurement points.

It is obvious that the mechanical–technological characteristic values of the tensile test decrease significantly with increasing copper content. This can be attributed to the decreasing component density caused by the addition of copper. As the copper content in the alloy increases, the flowability of the powder decreases, resulting in a less dense product. Consequently, the tensile strength, yield strength, and elongation at break of the alloy decrease with increasing copper content. Furthermore, the increasing scatter of the data is most likely due to the non-uniform distribution of copper in the final product due to the inhomogeneous powder composition. It is precisely the inhomogeneous distribution of the copper particles that can lead to fluctuating laser absorption and thus further disrupt the process. Despite these observed effects, the mechanical-technological properties of all tested samples are in the acceptable range for the intended application, even though they may need to be further optimized for specific applications. A slight decrease in the mechanical–technological properties was also observed in other studies [12].

5. Conclusions

In conclusion, this study successfully demonstrated the in situ alloying of 316L steel with copper powder using the PBF process. The resulting material exhibited uniform and desired concentrations of copper, as confirmed by the chemical composition measurements. However, it should be noted that the PBF system used in this study employs a powder chamber, and repeatability with plants using a powder fed via a pipe system may not be guaranteed.

The study also found that the addition of copper powder led to a decrease in melt track widths, indicating a deterioration in laser absorption. The resulting mechanical–

technological properties of the alloyed material were also found to decrease with increasing copper content, possibly due to the decreasing component density.

Overall, this study contributes to the understanding of the effects of in situ alloying on the PBF process and provides insights into the potential challenges and limitations of the method. Further research could explore different powder mixing ratios and examine the effect of other alloying elements on the resulting material properties.

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