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Influence of Powder Properties on the Mixing Behavior of Metal Powders in LPBF

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Abstract:
Laser Powder Bed Fusion (LPBF) has become an attractive alternative to conventional processing routes over the past years. While some alloys are already processed in series production, the overall amount of available materials is low. Due to the high price of specially atomized powders, material variation is often neglected. In this work, metal powder blends are created and investigated in order to find the influences of powder properties on blending behaviour. The blending tests are based on 316L and various individually added element powders. This supports the understanding of influences of different parameters on the mixing behaviour. Analytic methods to define properties such as particle diameter, density, particle shape and mixing time are defined. The printing tests serve the verification of the results drawn from the blending processes. A threshold for a homogeneity of a powder blend is defined.

Keywords: *LPBF, powder blend, mixing behaviour*

1. Introduction

The mixing behaviour of dry powders is a complex issue that is influenced by a large number of factors. One of these factors is the choice of a mixing method, another one is the influence of particle shape on segregation during the mixing process. According to Tang and Puri (2007), no quantifiable results showing a strong influence of particle shape have been found so far [15]. This is based on the fact that systematic changes of particle shape within an experiment are difficult to realize. One factor that can affect mixing behaviour is density. If there are enormous differences in density, the powders tend to segregate [16]. In this case, the lighter particles tend to flow more to the surface of the mixture. The heavier particles settle to the bottom of the mixing vessel [1]. This can be counteracted by the appropriate choice of mixer.
According to Shenoy (2014), the influence of particle size is considered to be more important than previously mentioned influences [12]. Also according to Mikli et al. (2001), important material properties, such apparent density and flowability depend on the particle size [9]. This means that particle size is an important factor influencing mixing behaviour [38]. By using very fine powder, cohesive forces and the large specific surface area can cause fine powder to adhere to coarser particles, which can counteract segregation [16].

The building process increases the complexity of the processing of powder mixtures again by the coating and melting of the powder. The aim of coating is a uniform powder application and the creation of a homogeneous and a smooth powder layer. This leads to a uniform absorption of the energy introduced into the powder bed by the laser beam and produces a more homogeneous microstructure and lower porosity [3]. In order to achieve a homogeneous and flat powder application, a high flowability is required in laser additive manufacturing [8]. In addition to this, other factors are responsible for a uniform powder application, which indirectly influence the flowability. A spherical particle shape enables better flowability compared to powders with an irregular morphology [5].

2. Materials and methods

Powders

Goal of the investigation is finding the influences of different powder properties on blending behaviour. Therefore, all powder blends used in this work were made out of powders that differ greatly in their material properties. The used powders were mixed in a ratio of 80/20 vol%. The majority was 316L stainless steel, while the other powders were mixed individually. These mixtures were not designed for printing, but describe the mixing behaviour very well. 316L as base powder was used because it has a conventional particle size, spherical particles and has a medium density. The other powders were selected in a way that differences can be seen in terms of density, particle shape, or particle size

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distribution. In the following part of the paper, the terminology for the powder mixtures will be used in a way that a molybdenum mixture is a mixture of 316L and molybdenum (80/20). This applies equivalently to the other blends (chromium blend, sieved chromium blend, 316L BJ blend, and AlSi10Mg blend). The Particle size can be considered by comparing the fine and rough powders. Explicitly, the chromium can be considered as a blend component for this purpose, since the sieving makes the powder finer, and the particle shape and density of the powder remain identical. For the particle shape, the mixed components 316L BJ and molybdenum were investigated. Both powders have a relatively similar density and are very fine in particle diameter. For the isolated comparison of density, the AlSi10Mg and chromium (unsieved) blends in the 80/20 vol% mixture are compared. The particle size distribution is very similar. In addition, both powders have an irregular particle shape. The differences in density between the powders are obvious.

Table 1: Blended powders

Material	Density	Morphology	D50 (µm)
316L	7,9	spherical	36,6
316L BJ	7,9	spherical	9,7
Molybdenum	10,22	irregular	12,1
Chromium	7,15	irregular	33,2
Chromium (sieved)	7,15	irregular	23,3
AlSi10Mg	2,68	irregular	35,5

Mixing method

In the first step, a suitable mixing duration was determined for the mixing tests. For this purpose, a selected mixture (316L/Mo 80/20 vol%) was blended at four different mixing durations. The mixing duration was selected based on empirical values and values from literature. According to these, the Turbula mixer (three-dimensional shaking mixing) provides good mixing of the powder after a short mixing time [33]. Therefore, the mixing durations of 1 min, 8 min, 15 min and 60 min were used in the test series. A further increase in the mixing duration would be feasible, but not economically viable due to the time required. Furthermore, Obadele et al. found that more pores are formed with a mixing duration of 8 hours. In addition, the long mixing time results in a large number of very fine particles that agglomerate on coarser particles [10].

Powder Analysis

In static mixing technology, the coefficient of variation VarK has been established as a measure of blending quality. It is a relative measure of dispersion, which means that there is no dependence on the unit of measurement of the statistically investigated variable [6]. It is used to compare characteristics of the same kind, but of different dimensions [2]. The coefficient of variation is composed of the quotient of the standard deviation and the average [4]. The lower the quotient, the more homogeneous the mixture can be considered. The coefficient of variation can be used to eliminate the dependence of the standard deviation on the average value [11].

$$VarK = \frac{s}{\bar{x}}$$

In order to be able to make a statistically valid statement for the ten test samples in the container, three further samples were measured from each test sample. One sample corresponds to two heaped spoons, which correspond to an amount of approximately 6-7 grams. The size of the mixing container depends on the amount of powder. According to Sommier et al. (2001), the filling level has an influence on the measurement result when mixing with a three-dimensional shaker mixer [13]. Above a filling level of 80 %, no sufficient mixing takes place in the centre of the container. The size of the sample container was selected according to these aspects and was filled to a level of 60 % with 200 grams of powder [14].

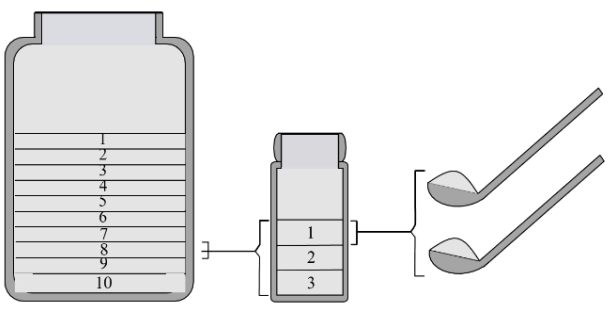


Figure 1: sample preparation

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To describe the concentrations between the base powder and the mixed components, volume percent was used, as this ensures better comparability of the samples. The reason for this is that the analysis system "Camsizer X2" shows the concentrations in volume percent and not in mass percent. Thus, the results of investigations of the mixtures are better comparable. Otherwise, only the number of particles could be given, but this is not useful for powders with a wide particle size distribution, because very fine particles would take up a much larger proportion.
The particle diameter and the specific surface area were used as a comparative measure for the fineness of particles. With the aspect ratio and the compactness it is possible to measure the particle shape.

Part production
In the last step, a printable blend was created in order to investigate the behaviour of the powder blends within the LPBF system. The blend was mixed taking all prior findings into account. The material was processed on a Trumpf TruPrint 1000 with a beam focus of 30 µm.
The parameters were fixed for all samples: a laser power of 195 W, a scan speed of 800 mm/s and a hatch distance of 50 µm. In combination with a layer thickness of 40 µm an energy density of 122 J/mm³ results for manufacturing these parts. The powder blend composition was chosen as follows:

Table 2: Results of the composition of the powder blend for LPBF by chemical analysis

Element	C	Cr	Mo	Ni	N	Si	Mn	Fe
	0,02	27,02	4,48	6,23	0,56	0,59	1,14	Bal.

Powders used for the composition

Powder	D2205	316L	CrN	Mo	Cr
Share	60 %	30 %	2 %	2 %	6 %

The powder blends were filled into the machine in a default way as any other powder to have a realistic process. The container was turned upside down into the powder storage.
In order to examine the composition of the parts an EDX analysis was conducted.

3. Results

Identification of optimal blending time
In Figure 2, the coefficients of variation of the specific surface area S_V are plotted over mixing time t. Each of the measured values show results from the measurement of 30 samples. It can be observed that for the Mo blend, mixing time history can be divided into three phases. In the first phase, the amount of material is continuously split and after the change of place it is split and mixed again. The groups of a powder which are not yet mixed are rapidly reduced by this process. In area II, the phases of convective mixing and shear mixing occur. During shear mixing, slip planes form in the bulk material, at which the coherent particle groups are slowly broken up and the different powders continue to mix with each other. Area III is determined by diffusion mechanisms, where a random movement of the powder particles takes place [17] [10].

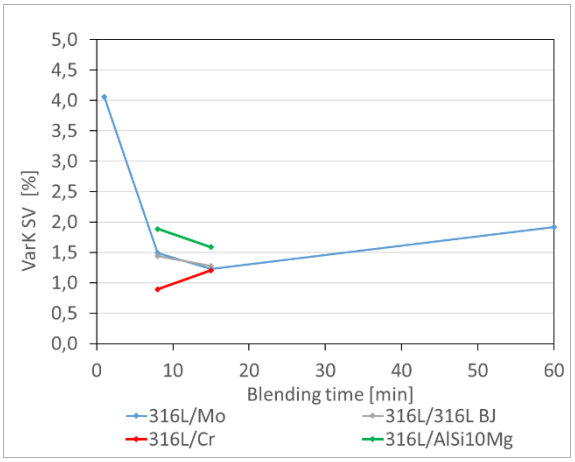


Figure 2: Influence of blending time

The degree of mixing shows significantly stronger deviations from the mean value at a blending time of one minute compared to the other measurements. The minimum, i.e. the smallest coefficient of variation, occurs at a mixing time of 15 min. It can be seen that the coefficient of variation increases at a mixing time of 60 minutes. This is due to possible segregation.
The coefficients of variation for the AlSi10Mg and 316L BJ mixes show a comparable trend to the Mo mix for mixing durations of 8 min and 15 min. The coefficient of variation for the mixing duration of 15 min lower than for the mixing duration 8 min. The course of the Cr mixture differs from the other mixtures in this point. The lower coefficient of variation was obtained for a mixing duration of 8 min. Based on the results, 15 min provide better mixing in three out of four cases. For further tests, the mixing time of 15 min was defined as the

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default value. Consequently, the mixing durations of 1 min and 60 min were excluded for the further mixing tests.

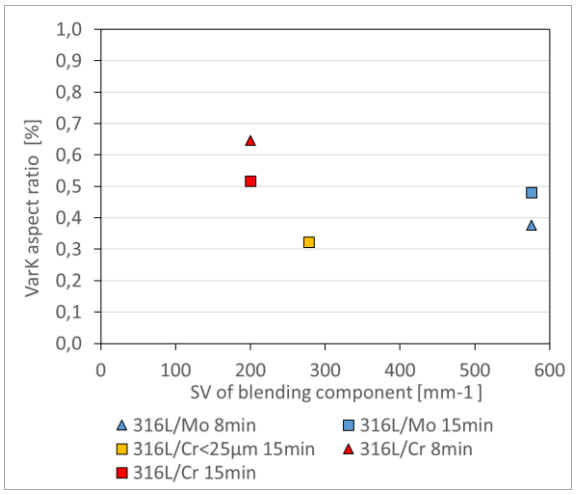


Figure 3: Influence of particle size

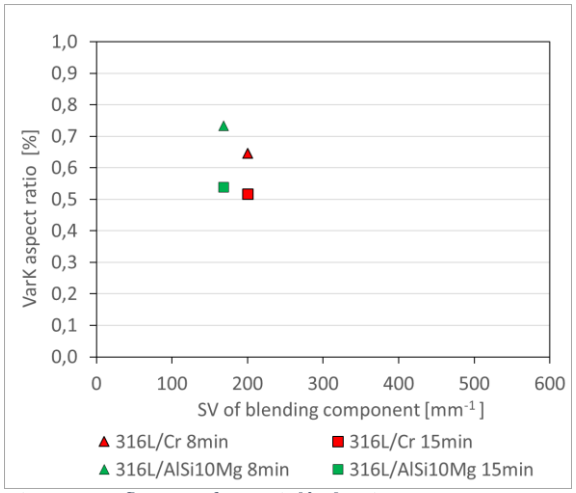


Figure 4: Influence of material´s density

Influence of particle size

In order to measure the influence of the particle size, the chromium powder was sieved to reach a lower d50 value. Due to the particle diameter being smaller, one factor was changed in isolation. The density and particle shape of the powder remained constant, while the particle size was varied. In order to take the mixing time into account, 8 min and 15 min are illustrated in Figure 3. The VarK of the aspect ratio is plotted over particle´s specific surface area (S_V) because (S_V) represents different particle size distributions best, while the aspect ratio shows the difference between 316L and the blended powders. The results show that the coefficient of variation for the sieved chromium powder achieves a significantly lower value compared to the unsieved powder. Since only the particle size was varied, the better mixing behaviour is due to the finer particle size distribution. Additionally, the finer Molybdenum powder shows a lower VarK than the unsieved Chromium. This means better mixing, consequently better homogeneity**.**

Influence of material density

The investigation of the influence of density was carried out between the Cr mixture and the AlSi10Mg mixture (Figure 4). Both mixture components have a very similar particle size. Both components consist of non-spherical particles. The strong difference between the two mixing components is the density. Cr has a density of ρ(Cr) = 7.15 g/cm³, while the AlSi10Mg powder has a much lower density, of ρ(AlSi10Mg) = 2.68 g/cm³. Both powders have similar d50 values compared to the base (316L) and both consist of irregularly shaped particles. Due to the difference in particle shape to the base powder, the coefficient of variation of aspect ratio was used to measure the homogeneity of the blends. Also these results show a big impact of blending time but a rather low impact of the density. Both powders behave very similar and no major effect can be documented.

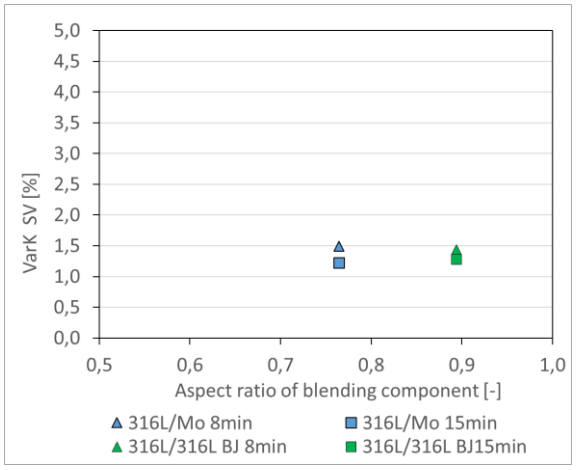


Figure 5: Influence of particle shape:

Influence of particle shape

For the investigation of the influence of particle shape, the Molybdenum blend and the 316L BJ blend were compared. The comparability was ensured by the fact that both powders (Mo & 316L BJ) have a very similar particle size distribution. The difference in density is present, but not very strong. According to the prior results the influence of density on the measurement result is considered to be small. In terms of particle shape, both powders are very different. The molybdenum powder consists of particles with an irregular shape, while the 316L BJ powder consists of particles with spherical shape.

For the investigation of the influence of the particle shape, the VarK of the spec. surface S_V was used (Figure 5**Error! Reference source not found.**).

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The reason for this is the spherical shape of the 316L BJ powder. It differs only slightly from the likewise spherical base powder. With regard to the small particle size of the 316L BJ and Molybdenum powder, a differentiation via the particle size is more meaningful, since the differences to the base powder are more pronounced. The mixing durations of 8 min and 15 min are shown so that the time is taken into account. It can be seen that the coefficients of variation of the two blends are very similar. The mixing with the duration of 15 min is better for both mixtures than the mixing for 8 min. In relation to all other mixtures, the 316L/ Mo mixture are the closest in terms of the coefficient of variation. This suggests that the powders behave similarly despite the different particle shape. Therefore, the influence of particle shape on mixing can be considered small. This is supported by the statements of Tang and Puri (2007) [15].

Composition of LPBF parts

The results of one EDX measurements are provided in **Error! Reference source not found.**, **Error! Reference source not found.**, Figure 6 and Figure 7. The measured powder composition is provided as reference. Three different positions where analysed. It can be seen that all values are very similar to the powder values, which leads to the conclusion that the different powders, including the elemental one, are well melted and mostly homogeneously distributed within the volume of the sample. This also confirms the findings of the powder blending investigations. The powders were well spread over the whole volume.

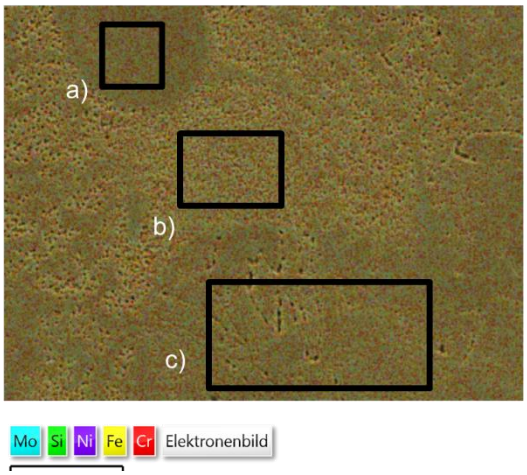


Figure 6: EDX measured areas

Table 3: Chemical composition of the sample measured by EDX

Element	a) %	b) %	c) %	powder %
Si	0,64	0,62	0,63	0,59
Cr	27,67	27,74	26,80	27.02
Fe	Bal	Bal	Bal	Bal
Ni	6.55	6,62	6,83	6,23
Mo	4,44	4,73	4,68	4,82

Figure 6 and Figure 7 show an area of roughly 120 x 120 µm², which equals four scan vectors of the laser. Nevertheless a few non melted particles of Molybdenum were found. Figure 7 shows three of those unmelted particles. Those appear very locally in certain areas of the parts. Currently, it is impossible to say, why those particles were not melted and how this is influenced. Right next to the Molybdenum particles in the square, the expected chemical composition can be measured again as listed in **Error! Reference source not found.**.



Figure 7: Sample with pores and unmelted Molybdenum

Table 4: Measured chemical compositions by EDX

Element	measured %	powder %
Si	0.63	0,59
Cr	27.17	27.02
Fe	Bal	Bal
Ni	6.65	6,23
Mo	4.49	4,82

4. Conclusions

In this study different metal powder combinations were blended and a methodology of mixing and measuring of homogeneity was examined. The particle size, particle shape, material´s density and blending time were included into the investigations. Following conclusions can be drawn from the

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work:

- The mixing time has a major impact on the homogeneity of powder blends. Most effective seems a time between 8 – 15 minutes when using a Turbula mixer and a 60% filling.
- The particle size has the biggest impact on blending compared to particle shapes and material´s densities, which only have minor impacts and could be neglected. However, those sizes, shapes and densities of the used powders are relatively close to each other. Thus, a broader field of properties could lead to different results.
- The parts built by using the powder blend were built successfully and the chemical composition suits the analysed powder composition.
- Nevertheless, some Molybdenum particles were not melted and were found caught in the samples, suggesting that the LPBF parameters have to be taken into account.

Additional research needs to be carried out to identify a parameter set for stable chemical and microstructural homogeneity. To bring these alloys into industrial application a reliable process with homogeneous parts is required. Further studies should include a systematic analysis of the heat treatment conditions and the resulting mechanical properties.

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