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# Influence of Reinforcement Particle Parameters on the Homogeneity of Particle Distribution in Semi-Solid Formed Aluminium Matrix Composites

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**Abstract.** Aluminium matrix composites (AMCs) offer improved mechanical and tribological properties compared to monolithic materials and therefore provide great potential for various applications. This particularly applies to particle-reinforced AMCs revealing comparatively high contents of reinforcement particles. However, these types of composites are difficult to manufacture due to their abrasive characteristics as well as their complex rheological material behaviour. An approach to produce such AMCs is semi-solid powder processing, combining powder pressing and sintering in one step in order to produce fully dense composites with currently only cylindrical shapes. Therefore, the tools as well as the powder mixture are first heated into the semi-solid temperature range of the aluminium powder and subsequently formed using low pressures under 200 MPa. Due to the shear thinning behaviour of the semi-solid aluminium matrix the porous structure of the pressed powder is filled during compaction, resulting in homogenous particle distributions in the component. However, this process results in high process times as well as energy costs, due to the heating inside of the die. In contrast to the semi-solid powder processing in one step, in this paper, a novel process route combining cold uniaxial compaction of particle reinforced aluminium powders having up to 50 vol.% SiC with subsequent semi-solid forming is presented. Here, a particle reinforced and cylindrically shaped green body is utilized as raw material, in order to produce complex components through semi-solid forming. The parts produced in this way are featuring varying wall thicknesses and are used in order to determine the process limits for manufacturing particle reinforced components having up to 50 vol.% SiC. Thereby, the influence of reinforcement particle size as well as particle loading on the homogeneity of the resulting particle distribution of an academic component is investigated. Future main objective of the process route is the manufacturing of complex parts with homogenously distributed particles.

## Introduction

High strength properties and low weight represent just a few of today´s increasing component specification, e.g. in the automotive, aeronautic or electrical engineering industries. Aluminium matrix composites (AMCs) meet those requirements by using reinforcements like fibers, whiskers, and particles to improve the mechanical properties of the matrix aluminium alloy [1]. However, to obtain optimum properties, a homogenous reinforcement distribution must be guaranteed.

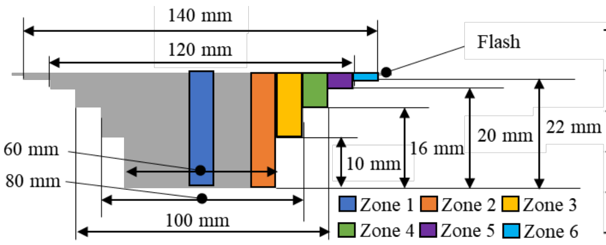
Manufacturing of AMCs can be divided into liquid state methods (e.g. casting), semi-solid state methods (e.g. semi-solid forging) and solid state methods (e.g. powder metallurgy). Stir casting processes, as part of the liquid state method, offer the advantages of low-cost manufacturing as well as simple process routes, producing near-net-shaped and complex geometries [2]. However, the wettability as well as the homogenous distribution of the reinforcement components are the main problems of stir casted components [3]. Furthermore, the high temperatures of the melt lead to interface reactions between matrix and reinforcement, resulting in brittle behavior at the interface [4]. Not least, the amount of reinforcement still ensuring homogenous reinforcement distribution is limited (< 30 vol.%) due to the settling of the reinforcement particles during freezing of the melt [5]. Powder metallurgy (solid state method) is using diffusion bonding to produce AMCs. Here, aluminium powder is mixed with the reinforcement particles and subsequently compacted using various compaction methods. Those compacted workpieces, so-called green bodies, are sintered at elevated temperatures under air, inert gas or vacuum, causing the matrix alloy to diffuse into a dense composite. One advantage of the solid state method is the low process temperature during sintering, which prevent interface reactions between reinforcement particles and matrix. Another advantage is the segregation free bonding during sintering, thus ensuring a homogenous particle distribution [2]. High material costs of the matrix powders [2] and tooling costs due to wear represent disadvantages of the sintering process, as well as residual porosity due to insufficient diffusion during sintering of green bodies with higher reinforcement contents [6]. Semi-solid state methods use raw materials coming form either in a liquid state (stir casting) or in a solid state (green body). The semi-solid material exhibits a paste-like dimensionally stable behavior, while providing time and shear rate dependent flow behavior [7]. The lower temperature compared to casting as well as the higher viscosity prevents reinforcement particle segregation and produces nearly full dense composites compared to sintering [8]. High particle loadings of up to 40 vol.% can be produced using semi-solid forming combined with direct pressure sintering of the semi-finished product, resulting in high density as well as homogenous particle distribution [9]. Currently, a novel process route combining powder pressing with subsequent semi-solid forming is investigated at the Institute for Metal Forming Technology (IFU) in Stuttgart to investigate the influence of even higher particle loadings onto the semi-solid forming process. While research results in the fields of the conventional routes (casting, powder metallurgy) have already revealed the influence of reinforcement on the process, this hasn't yet been fully explored for the semi-solid routes.

For particle reinforced AMCs produced by the powder metallurgy route, both the amount of reinforcement particle [6] and the particle size [10] influence the resulting specific material properties. Surya and Gugulothu [6] showed that the amount of reinforcement particles ranging from 5 up to 15 vol.% silicon carbides ($SiC_p$) generally improves the strength properties of the composites compared to the matrix aluminium alloy. Furthermore, higher amounts of $SiC_p$ increased the density, hardness, impact strength and wear behaviour of the produced composites. Tan et al. [11] proved that larger particles increase the thermal conductivity while decreasing the mechanical properties of $Al/SiC_p$ composites. Inhomogeneous particle distribution of larger particles as well as pores reduced the mechanical properties as well as the reached densities according to Sun et al. [10]. Further, an additional extrusion step, which rearranged the particle distribution as well as a closing of the pores, improved the mechanical strength. A correlation between the particle size and the reached ductility was found, stating that composites with larger particles show lower tensile and yield strengths but higher ductility compared to composites with smaller particles [10]. All authors proved a high influence of the amount of reinforcement particles as well as particle size onto the reached material properties after sintering, while for semi-solid forming processes it is currently not investigated. In the investigations presented in this paper, the influence of particle size on the particle distribution as well as the process limits of semi-solid formed composites was examined. Therefore, green bodies having up to 50 vol.% $SiC_p$ from two different powder sizes were heated into a semi-solid state and subsequently formed, thus producing complex shaped parts with varying wall thicknesses.

## Materials and Methods

The semi-finished billets or green bodies having a cylindrical shape with a diameter of 56 mm and a height of around 65 mm were pressed according to [12] using a pressure of 600 MPa. This resulted in green body densities of about 84 % ± 3 %, depending on the amount of reinforcement and particle size. The monomodal aluminium alloy (AlSi7Mg0.6) and technical silicon carbide (SiC) powders used had mean particle diameters of 250 µm and 63 µm respectively. In order to produce the powder mixtures, a size range of 1:1 was chosen to exclude side effects during semi-solid forming due to different particle sizes of the $SiC_p$ compared to the matrix. Green bodies were compacted with reinforcement particle contents of 30, 40 and 50 vol.% $SiC_p$. Afterwards, these green bodies were formed in the semi-solid state into complex shaped parts with varying wall thicknesses, as shown in Fig. 1. Occurring volume variations were compensated by the flash marked in the left picture.

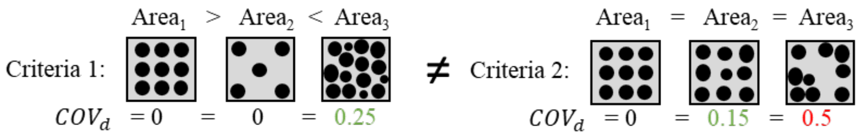
For the semi-solid forming process, the tool was heated to 350 °C and Molykote-Spray ($MoS_2$) was used as lubrication. Depending on the reinforcement particle content, the green bodies were heated to temperatures of 610 °C (30 vol.%), 630 °C (40 vol.%) and 640 °C (50 vol.%) using a Nabertherm LF15 furnace. After reaching the target temperature, it was held for 30 min. Subsequently, the billets were manually transferred to the foming tool and semi-solid formed by a ram speed of 100 mm/s. The forming tests were performed using a modified hydraulic press from BvH further described in [13].




| Experimental parameters | | | |
|---|---|---|---|
| Powder particle size [µm] | F 60 (250 – 400 µm) | F 220 (40 – 63 µm) | |
| SiC-Fraction [vol.%] | 30 | 40 | 50 |
| Tool temperature [°C] | 350 | Forming speed [mm/s ] | 100 |
| Lubrication | Molykote ($MoS_2$) | | |

Figure 1: Dimensions of the part geometry and experimental parameters used

To determine the process limits with regard to the microstructural homogeneity of the components produced, particle distribution analysis were performed. For this, specimens from the different component zones (see Fig. 1) were cut out using a precision cut-off machine (ATM) and subsequently prepared for metallographic investigations with a Keyence-vhx 5000. Multiple images of the six zones were taken and subsequently analysed using the finite body tessellation in order to identify the near neighbours of all particles according to [14]. To determine the homogeneity of the particle distributions in the components, the coefficient of variance (COV) was used according to [15]. The COV value is described as the standard deviation of the area or near neighbour distance ($\sigma_d$) divided by the average area or mean near neighbour distance (d) according to Eq. 1 [15].




$$COV_d = \frac{\sigma_d}{d} \quad (1)$$

Figure 2: Comparison of $COV_{area}$ vs $COV_d$ and equation to determine the COV

Two criteria for the components as seen in Fig. 2 need to be met to guarantee a homogenous particle distribution. On the one hand in every zone of the component the same amount of reinforcement should be present, therefore, the $COV_{Area}$ was determined. While on the other hand for equal amount of reinforcements the clustering can differ tremendously, therefore, the $COV_d$ of the mean near neighbour distance in all zones of the component was also determined.

## Results and Discussion

**Component analysition and process limits.** Fig. 3 shows a comparison between two components produced by the process route proposed in this paper. The components were manufactured from powders with two different particle sizes, once with particle size F60 and once with particle size 220, but with the same reinforcement particle content of 50 vol.% $SiC_p$. Hereby, a clear difference in particle distribution was observed. Using the powder with larger particles resulted in small entrapped voids between the particles, while with the smaller particles a nearly porous-free and homogenous microstructure was obtained. As shown in Fig. 3, for both particle sizes, only the matrix alloy without reinforcement particles could be found in zone 6 having a thickness of 2 mm. In contrast, reinforcement particles could be identified in this zone for components produced with 30 and 40 vol.% $SiC_p$. Thus it can be assumed that due to the higher amount of solid $SiC_p$, the semi-solid material is hindered and interfered by the $SiC_p$, resulting in a lower flowability and a lower entrainment of the particles. As a result the process limit for both particle sizes of such highly particle reinforced components is dependent on the particle loading. An increase in particle loading leads to a decrease in achievable wall thickness. The influence of the semi-solid forming parameters (billet/tool temperature, forming speed) on the forming behaviour will be investigated in future work to determine the improvement of process limits and particle distribution through varying semi-solid forming parameters.

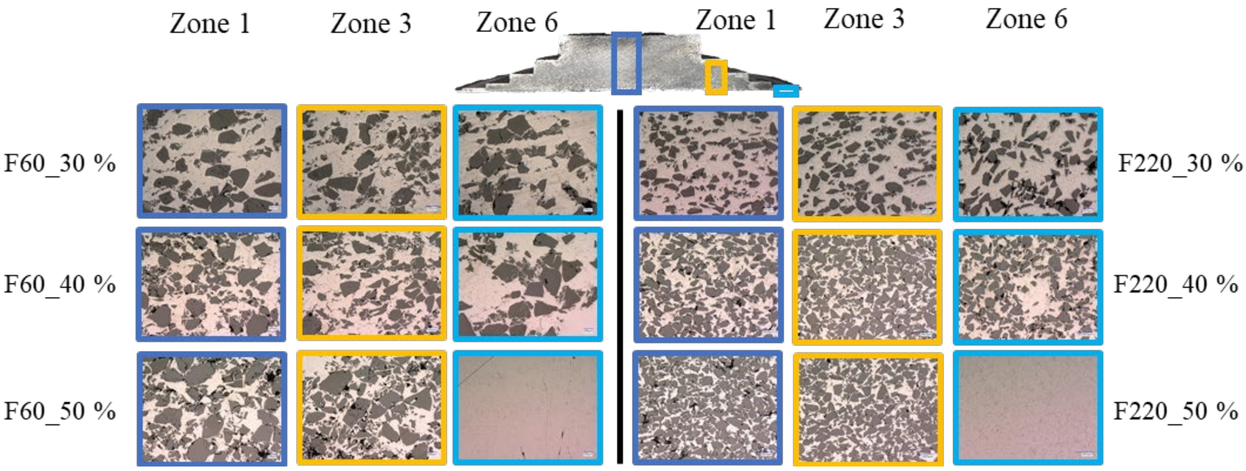



Figure 3: Microscopic view of the different zones for all AMC components with 30, 40, 50 vol.%

Zone 6 of the considered component is adjoined by a flash with a height of 0.5 mm in order to compensate for potential volume variations of the green body in the forming process. In this flash, only the matrix alloy was found for components with both 50 vol. % and 40 vol.% $SiC_p$, which again can be explained by the mutual obstruction of the solid fractions. While according to Bramann et. al. [16], the solid phases in a semi-solid melt tend to refine under shear loads and thus to fill smaller wall thicknesses, the size of the $SiC_p$ does not decrease due to the high hardness, resulting in segregation of matrix alloy and reinforcement. Since the particle size distribution of a monomodal powder has also an amount of smaller particles than the mean particle diameter, the effect can be investigated here as well. For the F60 50 vol.% component in Fig. 3 (left), the smaller particles of the $SiC_p$ are found in zone 5. In components with 30 vol.% $SiC_p$ made from both powder sizes, some particles can be detected in the flash zone, but inhomogeneously distributed and a considerably lower amount compared to the other zones.

**Analysiation of Criteria 1 (Area distribution of particles).** For AMC components, the homogenous distribution of the reinforcement particles is essential to ensure increased material properties compared to the monolithic matrix alloy. In the investigations presented here, the area distribution in each zone of the component was evaluated using the $COV_{Area}$. Tab. 1 shows the $COV_{Area}$ values determined in this way, whereby according to Yang et. al [15] a value of 0.36 ± 0.02 or lower indicates a homogenous random distribution of particles. The determined $COV_{Area}$ values imply that a homogenous area distribution could be obtained in the components with 30 and 40 vol.% $SiC_p$ made from both particle sizes. Hence, the amount of particles in each zone is nearly the same, while a lower value of the $COV_{Area}$ can be observed when using a particle size of F220. This results from the lower obstruction due to the smaller solid particles inside the semi-solid matrix and thus the improved entrainment of the $SiC_p$. However, the $COV_{Area}$ value of the components with a SiC content of 50 vol. % indicate an inhomogeneous area distribution of particles, which mainly can attributed to zone 6, wherein no reinforcement particles could be detected. In contrast, the $COV_{Area}$ calculated for these components without zone 6 resulted in a homogenous area distribution. The area distribution showed no dependency of the size of the $SiC_p$, while the size ratio was not investigated.

Table 1: Results of the area coefficient of variance ($COV_{Area}$) for the produced AMC components

| | F60 30% | F60 40% | F60 50% | F60 50% without zone 6 | F220 30% | F220 40% | F220 50% | F220 50% without zone 6 |
|---|---|---|---|---|---|---|---|---|
| $COV_{Area}$ | 0,15 | 0,23 | 0,45 | 0,22 | 0,10 | 0,16 | 0,45 | 0,13 |

**Analysiation of Criteria 2 (Particle clustering).** The $COV_{Area}$ does not account for the formation of $SiC_p$ agglomerates during forming, which means the area of particles can be homogenously distributed, while particles can still form clusters (see Fig. 2). Such particle clusters do negatively affect the material properties of the AMC components, due to the influence on recrystallization, precipitation and crack formation [14]. Therefore, the mean near neighbour distance ($COV_d$) was additionally used to detect particle clustering and thus characterize the homogeinity of the components´microstructures. Here, $COV_d$ values of up to 0.38 [15], as marked with the red line in Fig.4, indicate homogenous distribution or low clustering. In this regard, Fig. 4 shows that a larger particle size of the $SiC_p$ (F60) as well as the matrix alloy rather tends to form reinforcement clusters. Here, the components with a particle loading of 30 vol.% $SiC_p$ show inhomogeneous particle distributions in all zones of the component, while an increase in $COV_d$ values towards the outer component zones can be investigated. Similar behaviour can be investigated for the components produced with 40 vol.% $SiC_p$, while the segregation effect due to the higher particle loading increases. For the highest particle loading of 50 vol.% $SiC_p$ a nearly constant $COV_d$ value for zone 1 to 4 can be seen, this can be attributed to the highest green body temperature (100 % liquid fraction) before forming. For zone 6 the $COV_d$ value for 50 vol.% cannot be calculated due to the lack of particles for both particle sizes. In general, a clustering dependent on particle loading can be investigated, showing an increase in inhomogeneity towards zone 6, due to the higher obstruction and hindering during forming of the additionally added large solid particles of $SiC_p$, resulting in a process limit 2 mm wall thickness.

For the smaller powder size (F220) a similar general behaviour of the particle loading can be investigated for zone 6. For all three particle loadings the $COV_d$ compared to the F60 specimen is decreased, which relates to the smaller particle size of the $SiC_p$ during forming. Although for the specimens with a particle loading of 30 vol.% $SiC_p$, still inhomogeneity can be investigated. This can be attributed to the temperature used in the process, compared to the temperature of the green bodies of 40 and 50 vol.% a 20 /30 °C lower temperature was used, resulting in a lower liquid fraction of the matrix alloy. Nearly homogenous distributions could be investigated in the F220 40 and 50 vol.% components up to zone 5, showing the feasibility of this process route to produce AMCs with high particle loadings up to 50 vol.% $SiC_p$. As a result the influence of particle size as well as particle loading onto the semi-solid forming of AMC green bodies could be proven in this paper. Homogenoues particle distributions can be produced using smaller particles (F220), while larger particles (F60) tend to form clusters. Higher particle loadings show an earlier process limit, due to the hindering and obstruction of the $SiC_p$ and thereby decreased entrainment during forming.

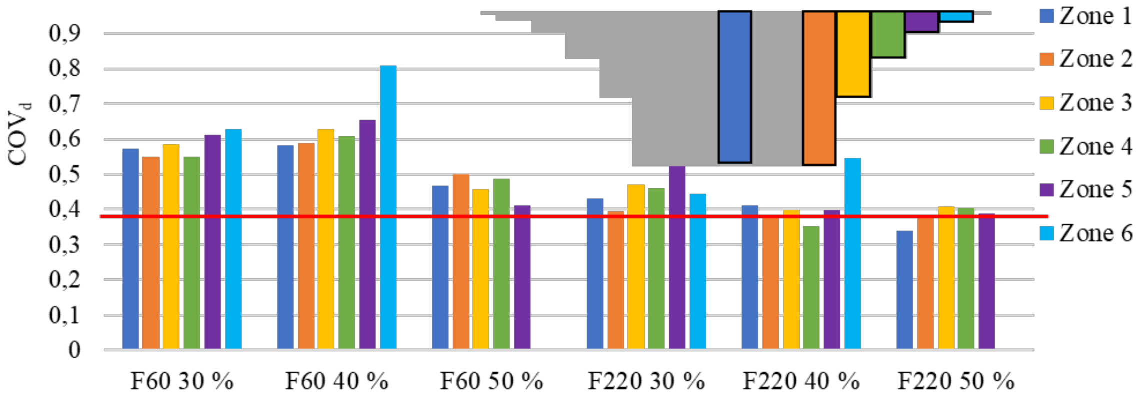



Figure 4: $COV_d$ values for the produced components

## Conclusion

In this paper, the influence of particle size and loading on the semi-solid forming behaviour of green bodies with comparatively high reinforcement particle contents of up to 50 vol.% $SiC_p$ was investigated. Thereby, the main findings are:

- The novel process route is feasible to produce AMCs with up to 50 vol.% $SiC_p$ with a homogenous particle distribution when using a powder size of F220
- For both particle sizes a homogenous area distribution could be investigated, showing no dependency on particle size
- Finer $SiC_p$ powders produce homogenous AMC components with high particle loadings, resulting from the higher entrainment of the particles during semi-solid forming
- Currently, for these semi-solid forming parameters, the process limit to produce components with a homogenous particle distribution is a wall thickness of 4 mm, due to the obstruction introduced by the solid $SiC_p$ in the semi-solid melt.

In future work, the influence of different semi-solid parameters on the particle distribution as well as the process limits of the so-produced components will be investigated. Furthermore, the material properties of those components will be determined.

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