Influence of metal matrix powder size on the tensile strength of a SiC$_p$/AlSi7Mg0,6 composite produced by field assisted sintering technique

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Abstract. In the present study, 35 vol% SiC$_p$/AlSi7Mg0,6 composites were prepared using field assisted sintering technique in order to investigate the effect of different particle fractions and size distributions of the AlSi7Mg matrix powder on the tensile properties of the produced composite material. In most usecases the size of the reinforcement phase is given by the application and is only variable within narrow limits ($\leq 20\mu m$ particle size in this work). On the other hand, there is potential for optimization of the matrix powder. In this investigation, fine ($d_{50} = 25\mu m$), coarse ($d_{50} = 52\mu m$), bimodal (50 wt% of fine + 50 wt% of coarse, $d_{50} = 36\mu m$) and as received ($d_{50} = 40\mu m$) aluminum powder was used as the matrix powder. Using fine matrix powder has improved yield strength by 5 % and ultimate tensile strength by 7 % compared to the as received condition. This is largely due to the lower porosity of the composite produced under the use of the fine matrix powder ((0.07 $\pm$ 0.04) %) in contrast to the composite using the as received aluminum powder ((0.62 $\pm$ 0.35) %). At the same time, the consumed heating energy of the composite was decreased by almost a third when using the fine matrix powder in comparison to the use of the as-received matrix powder. This paper presents results of an optimization approach for mechanical properties of aluminum matrix composites without any changes of the sintering parameters.

1. Introduction
Due to their widely adjustable properties, aluminum matrix composites (AMCs) promise high application potential in various industries. A wide range of applications is currently not given due to their complex and costly production. Therefore, different fabrication routes are the subject of current research. Field assisted sintering technique (FAST), often referred to as spark plasma sintering (SPS) in the literature, allows the production of various materials, among others ceramics [1], metals and their alloys [2], composites [3, 4] and even high entropy alloys [5]. It is not surprising that this promising process also seems to have potential in the production of AMCs [6, 7]. Almost pore-free AMCs with a homogeneously distributed reinforcement phase can be fabricated [8]. Therefore, both the matrix material and the reinforcing phase are mixed in powder form before the sintering process. One parameter to influence production and application properties is the powder size, both of the matrix and the reinforcement phase. The reinforcement particle size is mainly given by the application: nanometer range for improvements in strength properties [6, 9, 10] and micrometer range for the improvement of wear resistance [11, 12]. If the application is regarded as given, the variation of the matrix remains. Obviously, the smaller
Table 1. Chemical composition of the used metal powder according to DIN EN 1706 in wt%.

| Element | Si  | Fe  | Cu  | Mn  | Mg  | Zn  | Ti  | others | Al  |
|---------|-----|-----|-----|-----|-----|-----|-----|--------|-----|
| wt%     | 6.50–7.50 | 0.19 | 0.05 | 0.10 | 0.45–0.70 | 0.07 | 0.25 | 0.10 | balance |

Table 2. The used fractions of metal matrix powder and the SiC powder as the reinforcement phase provided by sieving. The combination of this description of the fraction and AMC is used in the text as an abbreviation for the corresponding mixed composites, e.g. "fine AMC".

| Fraction      | $d_{10}$ (μm) | $d_{50}$ (μm) | $d_{90}$ (μm) |
|---------------|--------------|--------------|--------------|
| Fine          | 9            | 25           | 41           |
| Coarse        | 35           | 52           | 80           |
| Bimodal       | 15           | 36           | 60           |
| As received   | 23           | 40           | 64           |
| SiC           | 12           | 21           | 33           |

the powder size, the higher the packing density and the amount of contact points between particles which leads to a better diffusion and neck forming in sintering processes [13]. But on the other hand, aluminum is a strong passivating metal: The smaller the powder size the larger the oxidized surface area which leads to a worse sintering result. In addition to that, diffusion is by far not the only compacting mechanism during field assisted sintering [14]. Among others, "particle rearrangement" and localized deformation are to be mentioned. Both effects have positive impact on the sintering process and are more pronounced with coarser particles [2].

In this paper, the influence of the metal matrix particle fraction and size distribution on the mechanical properties of an AMC produced by field assisted sintering is investigated.

2. Materials and methods

Inert gas atomized powder "AlSi7Mg0,6 < 0,063 mm" by ECKA Granules Germany GmbH (for chemical composition see table 1) was used as the matrix material. The powder was sieved into two different fractions (see table 2), which were examined for their characteristic diameters by laser diffraction analysis using a ”Cilas 920” device. Together with the bimodal fraction (mixture of 50 wt% of fine and 50 wt% of coarse fraction) and the as received condition, four fractions and size distributions were examined. SiC-Powder ($d_{50} = 21 \mu m$) was used as a reinforcement phase. The four different matrix powder fractions were mixed with the SiC powder using a tumbler mixer to a composite powder with 35 vol% reinforcement content, each. According to DIN EN ISO 3953, the bulk density of all the powder mixtures were determined.

The powder mixtures were compacted using field assisted sintering by a ”SPS KCE FCT-HP D 25-SI” (FCT Systeme GmbH, Germany) in order to produce AMC variants. Three samples of each AMC variant with a diameter of 40 mm and a height of 8 mm were prepared in a random order. The used sintering tools (die and punches) were made out of graphite. In addition to that, a release agent in form of a 0.3 mm graphite foil was used between die and punches. To minimize reactions of both the sample and the tool with the atmosphere during sintering, the recipient was flushed with argon and evacuated to a low vacuum (< 1 mbar) twice. The sintering was performed with a constant current using the following manufacturing parameters: a sintering temperature of 520 °C, a constant pressure of 50 MPa, a heating rate of 100 K min$^{-1}$ and a holding
time of 5 min. The cooling to room temperature was uncontrolled with a resulting cooling rate between \((50 \text{ and } 100) \text{ K min}^{-1}\). By integrating the consumed heating power over time and corrected by the amount of energy used for a cycle in the empty sintering device, the heating energy per sample was calculated. The density of all samples was estimated by Archimedes’ principle using a Mettler-Toledo laboratory balance. From this the difference to the theoretical density could be determined. Porosity was determined optically using gray value correlation. According to figure 1, one specimen for metallographic investigations and three tensile specimens were eroded from the sintered samples. Cross-sections for the metallographic investigations were prepared by standard procedures. In total there were nine tensile specimens tested per condition. Tensile tests were performed with a ”Zwick Allround-Line 20 kN” (ZwickRoell GmbH & Co. KG, Germany) at room temperature using an initial strain rate of \(10^{-3}\text{ s}^{-1}\). The strain was measured by gray value correlation using a ”GOM Aramis” system. The toughness was calculated by integrating the stress-strain curve until failure.

3. Results and discussion
Figure 2 (a) shows the bulk density of the powder fractions. Obviously, this is highest with the fine AMC and lowest with the coarse AMC. The other two fractions fall in between and do not differ significantly from each other. A high bulk density means many contact points between the powder particles, which in turn promotes diffusion between the particles and leads to a high number of sinter bridges. A lower bulk density in turn allows intensive particle rearrangement and considerably larger local deformations, especially at the first stages of the sintering process. Furthermore, it should be noted that with decreasing particle size the total particle surface increases quadratically. In the case of a passivating material (e.g. the alloy considered here), this also means a significantly higher amount of oxide layers within the bulk material. Oxide layer could impede diffusion during the sintering process and may have a negative effect on microstructure formation.
Figure 2. (a) Bulk density of the powder fractions in relation to the theoretical density of a pore-free composite with the given composition. Furthermore, the consumed heating energy of the samples during sintering process, corrected by the amount of heating energy for the sintering apparatus, determined in an empty run. (b) Porosity of the compacted samples determined by microscopic investigations and by Archimedes principle using a laboratory balance, respectively.

The energy consumed by the samples during the sintering process is shown in Figure 2 (a). The as received AMC consumed a heating energy of \((376 \pm 49)\) J, which is on a similar level to the coarse AMC that consumed \((348 \pm 83)\) J. A significant reduction in energy can be observed in both the fine composite and the bimodal AMC. The bimodel AMC consumes an energy of \((308 \pm 84)\) J, which means a reduction of about \(20\%\) compared to the as received AMC. In fact, the fine AMC consumes only an energy of \((261 \pm 49)\) J, which means a reduction of almost one third \((31\%)\) compared to the as received AMC. This is probably due to the fact that there are significantly more contact points between the powder particles, especially at the beginning of the sintering process, which leads to a better conversion of the electric current into joule heat. Therefore, this temperature-controlled process requires less electrical power to reach and maintain the set temperature.

Figure 3 shows the metallographic cuttings of the four different AMC conditions. For the fine metall matrix powder there are an almost pore-free microstructure with a homogenous dispersion of the reinforcement phase. Almost no clusters of SiC-particles are visible. In contrast, the AMC with the coarse metall matrix powder shows huge clusters of the reinforcement phase and clearly visible porosity. This is due to the missing content of fines. There are no fines which could penetrate into the clusters evolved during sintering process. The results are large voids inside the SiC-clusters. This conclusion is supported by the other two AMCs. Despite there are a less homogenous distribution of SiC-particles inside the bimodal AMC with a tendency to clustering, there are no significant voids visible. This is most likely due to the mixed in fines of this composite. The bimodal AMC and the as received AMC have a similar average powder size,
Figure 3. Micrographs of the different AMCs produced: (a) fine AMC, (b) coarse AMC, (c) bimodal AMC and (d) as received AMC. The bright phase is the aluminium alloy, the dark phase is the SiC and the black areas are pores.

but the content of fine fractions in the as received composite is much smaller. As a consequence, in this samples voids can be found similar to the coarse AMC. It seems, that not the average powder size of the metal matrix powder, but a critical amount of fine fraction influences the occurrence of voids.

The qualitative microstructure observations are confirmed in the difference to theoretical density $\Delta \rho_{th}$ and the porosity. The fine AMC has a $\Delta \rho_{th}$ of $(0.35 \pm 0.07)\%$, only a porosity of $(0.07 \pm 0.04)\%$ can be identified. This means a nearly non-porous material. The coarse AMC, on the other hand, shows the highest $\Delta \rho_{th}$ of $(1.87 \pm 0.02)\%$, porosity measured of $(0.95 \pm 0.33)\%$, as suspected when looking at the microstructure. A $\Delta \rho_{th}$ of $(0.86 \pm 0.05)\%$, porosity of $(0.32 \pm 0.24)\%$, is detected for the bimodal AMC, which allows a practical application of this composite. This porosity level is in the range of gravity casting, a potential competitive method. The bimodal AMC is also less porous than the AMC produced with the as received powder. The latter shows a $\Delta \rho_{th}$ of $(1.38 \pm 0.02)\%$, optically measured of $(0.62 \pm 0.35)\%$.

It is noticeable, that the $\Delta \rho_{th}$ calculated by Archimedes’ principle is higher than the porosity determined by optical microscopy for all samples. This is due to the density measurement of the whole sample by laboratory balance. In contrast, the microscopic determination is performed at some cross sections of the sample, not the sample as a whole. The higher standard deviation supports this conclusion. So, it is assumed and in accordance with other authors (e.g. [15]), that the Archimedes’ principle is more reliable than microscopic determination for absolute values.
Figure 4. (a) Representative engineering stress-strain curves of the four different AMCs and the same matrix alloy without reinforcement with initial strain rate of $10^{-3}$ s$^{-1}$ until uniform elongation. (b) Yield strength, ultimate tensile strength and toughness of the four different AMC conditions. The vertical stress-axis in (a) and (b) have the same scale.

However, it should also be mentioned that $\Delta \rho_{\text{th}}$ determined by Archimedes’ principle is related to the theoretical density of the composite. In practice, this can be subjected to slight fluctuations due to the mixing process. In relation, both measurement methods are consistent with each other.

Representative stress-strain curves of each composite are shown in Figure 4 (a) and the yield strength (YS), ultimate tensile strength (UTS) and toughness are shown in Figure 4 (b). The as received AMC shows a YS of $(179 \pm 5)$ MPa and an UTS of $(222 \pm 6)$ MPa, while the toughness was calculated on $(228 \pm 38)$ J cm$^{-3}$. The fine AMC shows both, the best strength and toughness. Out of all tensile specimens a YS of $(188 \pm 2)$ MPa (increase of 5 %) and an UTS of $(238 \pm 2)$ MPa (increase of 7 %) was measured with a toughness of $(390 \pm 22)$ J cm$^{-3}$ (70 % more than the as received AMC). The bimodal AMC shows a similar curve shape to the fine AMC, which results in similar strength values, namely a YS of $(187 \pm 2)$ MPa and an UTS of $(233 \pm 1)$ MPa, respectively. The lower toughness of $(296 \pm 18)$ J cm$^{-3}$ is apparently due to the lower uniform elongation. In contrast to the composites with fine content, the coarse AMC shows the worst strength and toughness values with a YS of $(177 \pm 4)$ MPa, an UTS of $(206 \pm 15)$ MPa and a toughness of $(131 \pm 51)$ J cm$^{-3}$. In particular, the toughness is over 40 % less than the as received AMC. Also worth mentioning is the greater fluctuation of the measured values between the parallel samples, especially for the UTS and the toughness. This is probably due to the pores and clusters, described at the microscopic observations, which act as inner notches.

Thus, the $\Delta \rho_{\text{th}}$ and the porosity of the samples were reflected one-to-one in the mechanical properties. A composite that is as dense as possible is therefore obtained by the presence of a certain amount of fines. It is therefore clear that the composite sintered exclusively from
a very fine fraction has the highest density and the best mechanical properties. A significant influence due to the increasing proportion of oxide films with smaller particles does not seem to exist in this order of magnitude (micro-scale). On the other hand, adding a fine fraction to a coarse fraction has a very strong effect, as the bimodal composite proves. Compared to the as received AMC (and of course to the coarse AMC), the density and thus the tensile strength was significantly improved. This circumstance possibly allows the utilization of fine and coarse fractions by sintering, which are discarded in other manufacturing processes, e.g. in thermal spraying. An effect that coarse particles can have a positive impact on the FAST process (see [2]) could not be determined for the SiC$_p$/AlSi7Mg0.6 composites examined in this work.

4. Conclusions

SiC$_p$/AlSi7Mg0.6 composites with different powder fractions and particle sizes as a matrix, fine ($d_{50} = 25\,\mu m$), coarse ($d_{50} = 52\,\mu m$), bimodal (50 wt% of fine + 50 wt% of coarse, $d_{50} = 36\,\mu m$) and as received ($d_{50} = 40\,\mu m$), were fabricated by field assisted sintering technique and examined for their microstructure and mechanical properties. The following key aspects can be summarized from this study.

(i) Heating energy consumed by the fine AMC was over 30 % less than the as received AMC. Both, coarse and bidmodal AMC showed no analyzable differences to the as received AMC.

(ii) The fine AMC shows a nearly pore-free microstructure with a homogenous distribution of the reinforcement phase, whereas the coarse AMC shows the highest porosity with partly huge clusters of SiC particles. Bimodal powder fraction can improve density in comparison to the as received powder fraction.

(iii) Using the fine aluminum powder as a matrix increases the yield strength about 5 % and the ultimate tensile strength about 7 % compared to the as received powder. The toughness was increased by about 70 %. The bimodal specimen achieves similar strength values, but with a considerably lower toughness. The coarse AMC had the worst mechanical properties, especially tensile strength (decrease of 7 %) and toughness (decrease over 40 %) are significantly lower than in the as received composite.

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