Consolidation behaviour of particle reinforced aluminium-matrix powders with up to 50 vol.% SiC_p

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Abstract. Aluminium-matrix composites (AMC) are produced in order to improve specific properties (e.g. mechanical, thermal or tribological) of the matrix alloy using discontinuous reinforcements like fibres or particles. If functional requirements of application specific components such as brake discs have to be met, reinforcement contents of more than 35 % may be required. Therefore, a new manufacturing route is currently being investigated at the Institute for Metal Forming Technology (IFU) of the University of Stuttgart, combining powder pressing with subsequent semi-solid forming to obtain near-net-shape components. A key challenge of this process route includes compacting the green bodies. In this contribution, therefore, powder pressing of AlSi7Mg0.6 and SiC_p powders with up to 50 vol.% SiC_p are investigated in order to gain a deeper understanding of the compacting behaviour of powder with particularly high reinforcement contents. During the investigations, homogenously mixed powders were consolidated to a cylindrical green body by uniaxial powder pressing in order to determine the compressibility of different AMC powder mixtures. At first, the influence of the consolidation pressure onto the reached density was analysed, resulting in compressibility curves as well as process limits for different amounts of reinforcement particles. Subsequently, the influence of particle size onto the achieved density as well as consolidation behaviour could be identified. Furthermore, the high tool wear due to the abrasive behaviour of the reinforcement is quantified in this paper.

Introduction

In comparison to monolithic aluminium alloys, aluminium-matrix composites (AMC) exhibit advanced properties such as increased stiffness, wear resistance, specific strength and vibration damping as well as a decreased coefficient of thermal expansion [1]. However, manufacturing of particle reinforced AMC still is facing technological problems in terms of relatively high processing costs and prevailing production difficulties, which are in the focus of different current research activities [1-3]. Manufacturing processes of AMC investigated in this context in general can be divided into liquid, semi-solid and solid routes. In liquid process routes, the matrix alloy is melted, while solid reinforcement particles are distributed in the melt. This can adversely affect its castability, thus leading to porosity [4] and agglomeration [5] within the final component´s volume. The particle reinforcement content achievable with liquid process routes is therefore up to now limited to 30 vol.% in most cases [6]. In contrast, in the semi-solid (e.g. [7]) and solid (e.g. [8]) process routes, both matrix and reinforcement are solid powders. Therefore, a consolidation step is required in these process routes to produce green bodies for further manufacturing steps. Several approaches can be used for this consolidation respectively cold or hot isostatic pressing, cold uniaxial pressing or direct pressure sintering. For the solid process route, a sinter [1], sinterforging [9] or extrusion [10] step can subsequently be used in order to bond as well as further consolidate the green bodies into the final shape for solid forming. Current investigations in the field of reinforced aluminium powder mixtures show the influence of size ratio, volume ratio between reinforcement and matrix alloy particles up to 30 vol.%. Hafizpour and Simchi displayed that a size ratio of 1:1 is recommended, especially for higher amounts of reinforcements, in order to reach a high density [3]. O'Donnell and Looney investigated the influence of lubrication onto the consolidation process using insert wall lubrication or mixed-in lubrication, showing no noticeable effects on the sintered specimens [1].

At the Institute for Metal Forming Technology of the University of Stuttgart (IFU), semi-solid routes were initially investigated in terms of producing continuous fibre-reinforced aluminium alloys [11]. Further investigations resulted in the manufacturing of a metallic end connection for pultruded fiber composite tensile rods, which were forged into a metallic end connection [12]. First investigations of particle reinforced AMC were produced by a process route combining direct pressure sintering with subsequent semi-solid forming, resulting in promising material properties as well as complex near-net shape components [2]. However, the direct pressure sintering step should be avoided in order to reduce energy costs. Therefore, a novel process route is currently investigated combining cold uniaxial compression with subsequent semi-solid forming. Thereby, particular focus is now being placed on the forming of AMC with particle loadings higher than 30 vol.% silicon carbide (SiC_p). Currently, no investigations of the compaction behavior of powders with SiC_p reinforcement higher than 30 vol.% were made, while for this novel process route further understanding of the compaction behavior is needed.

The present paper deals with the first step of the novel semi-solid process route and thus with the cold uniaxial compression of aluminium alloy powder with silicon carbide powder (SiC_p). Here, special attention is paid to the production of green bodies with as homogenous as possible particle and density distribution in the green body volume. In this paper the influence of two different composite particle sizes of the powder as well as of compression insert wall lubrication onto the consolidation behaviour is studied.

Materials and Methods

For the investigations of different AMC powder mixtures on the consolidation behaviour, two different particle sizes having a mean particle diameter of 250 µm and 63 µm respectively were analysed. For the matrix aluminium alloy (AlSi7Mg0.6), produced by Ecka Granules GmbH, Germany, nitrogen gas atomized powder with a nearly globular shape was used (Fig. 1a). During production of gas atomized powders larger particle sizes are undesirably produced, due to overspray. This normally would lead to a recycling of those larger particles, since established additive manufacturing routes commonly use particle sizes smaller than 63 µm. To utilize those larger particles for the production of green bodies, a powder particle size having a mean diameter of 250 µm was used. However, a conventional particle size for AM with a mean diameter of 63 µm was investigated as well. Commercially available SiC_p particles having an angular shape, as seen in Fig. 1b, were utilised as reinforcement particles. Dry mixing of both powders (size ratio 1:1) with a particle loading of 30, 40 and 50 vol.% SiC_P was performed by a turbular mixer. With a mixing speed of 20 min^{-1} and 15 min mixing time, a homogenous mixture of both powders could be guaranteed for all powder mixtures. The homogeneity was proven by an image analysis of the different volume fractions based on finite body tessellation and the coefficient of variance (COV) [13].

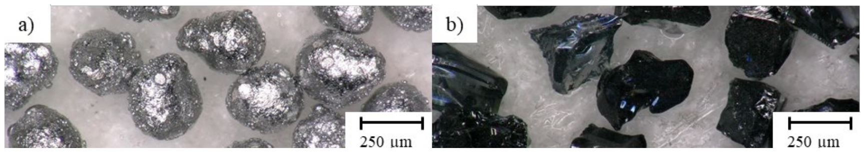
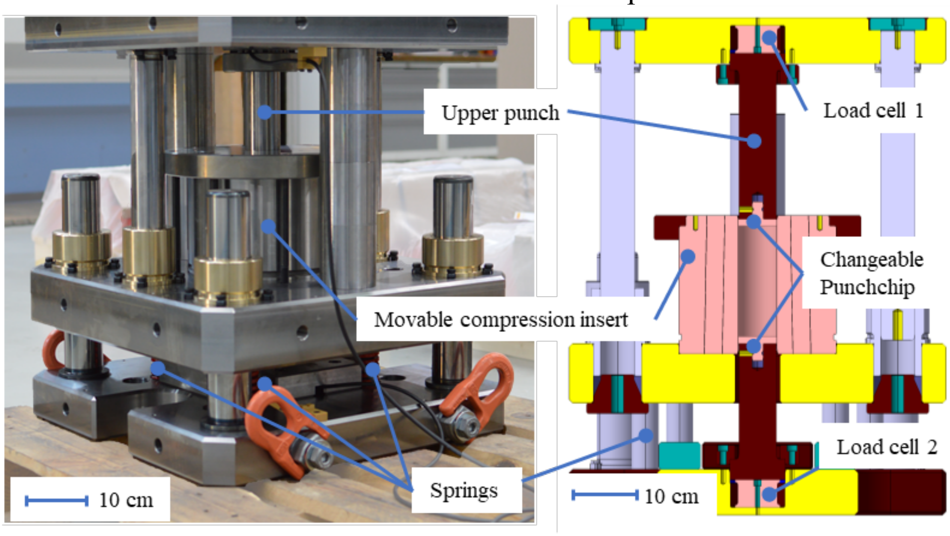


Fig. 1. a) Nitrogen gas atomized aluminium powder b) SiC powder (mean diameter 250 µm).

The tool used for the consolidation tests was designed with a movable compression insert mounted on four springs in order to ensure a two-sided uniaxial compression (Fig. 2). The insert diameter was 56 mm having a g6 fit, while the punches were processed to a H7 fit. The upper and lower punch was attached to a loading cell for measuring the press forces during consolidation. Contact faces of the punches to the workpiece were designed to be changeable due to the expected high abrasive wear by the SiC_p. This was achieved by using changeable punch-chips, which were mounted via a ball catch for a quick changeover. A hydraulic press was used to consolidate the green bodies with different press ram forces. Maximum force of the press is 2000 kN having a maximum stroke of 800 mm with a constant ram speed of 10 mm/s.



Mueller press	
Hydraulic press	
Max. Force [kN]	2000
Ram Speed [mm/s]	10
Max. Stroke [mm]	800

Fig. 2. Compacting tool and specification of hydraulic press used.

During the consolidation tests, mixed powders all having a size ratio of 1:1 (mean particle diameter of both powders) were compacted using pressures of 300, 450 and 600 MPa respectively (see Table 1). The coarser (F 60) powder was additionally compacted using a hydraulic press at a pressure of 1000 MPa, in order to determine the maximum force that can be applied without defects in the green body for this coarse size. In conventional powder metallurgic process routes often, lubrication is added to the powder mixture. However, for the subsequent semi-solid forming, lubrication is undesirable due to the included remaining lubrication in the final components. The presented experiments were carried out partly without lubricant and partly with molybdenum disulfide (MoS_2). Here, the MoS_2 was only applied to the inner side of the insert and the punch surfaces, aiming to minimize the amount of contamination inside of the green bodies produced. After ejection of the green bodies out of the insert, the green body density was measured using a volumetric approach. Weight measurement was performed on a precision balance with a deviation in measure of 0.001 g, while for dimensional measurement a micrometer with 0.01 mm deviation was used. For all experimental parameter combinations, three green bodies were produced and the mean deviation was used to compare the results in an objective manner.

Table 1. Experimental parameters used.

Experimental parameters				
Powder particle size [µm]	F 60 (250 – 400 µm)		F 220 (40 – 63 µm)	
SiC-Fraction [vol.%]	30	40	50	
Compaction pressure [MPa]	300	450	600	1000 (only F60)
Lubrication	None		Molykote (MoS_2)	

Results and Discussion

At first the obtained results of the unlubricated consolidation experiments are presented. The mixtures of powders with the larger particle size (F 60) could be pressed at all compaction pressure levels to unlubricated green bodies having relative densities dependent on the pressure value. The resulting compressibility curves are shown for all three investigated proportions of SiC-particles in Fig. 3. Thereby, the relative density was calculated from the reached density (volumetric approach) compared to the ideal density of the composite of each respective powder mixture. Lower particle loadings of 30 vol.% SiC_p showed the highest reached density for all pressure values. The reason for this is that the aluminium matrix is plastically deformed during densification, while the harder SiC_p particles are only elastically deformed. At the beginning of the densification a particle rearrangement occurs, as the pressure further increases the plastic deformation and thus the particle deformation of the aluminium particle begins. Elastic deformation of the SiC_p leads to a reduction of the plastic deformation and hence an insufficient bonding strength between the aluminium particles. Given by the uniaxial compression with a movable compression insert, the area of the lowest density is in the middle of the green body. If the plastic deformation is insufficient, a delamination plane appears in the green body, consisting of nearly non-compacted powder with low cohesion. Green bodies having a density below the depicted red line indicating a relative density of 77 % in fact showed such behavior with an insufficient densification. In general, a higher portion of reinforcement amount increases the absorbed elastic pressure and further limits the densification. Green bodies with 50 vol.% SiC_p showed on average a 4% lower relative density compared to green bodies with only 30 vol.% SiC_p. Maximum reached densities were 91.86 % (30 vol.%), 89.51 % (40 vol.%) and 87.69 % (50 vol.%). During ejection, abrasive wear of the insert as well as the punch-chips appeared, while with higher compaction pressure a higher ejection force as well as a higher wear behavior was determined.

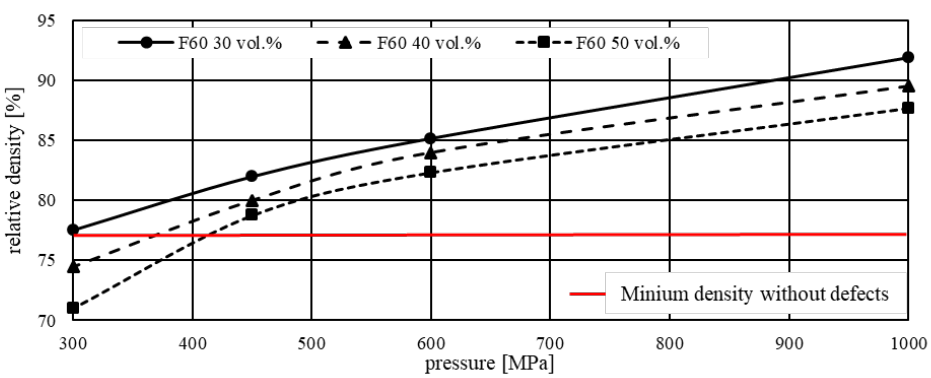


Fig. 3. Compressibility curve of the larger powder mixture without lubrication.

The finer powder of F220 could not be compacted without lubrication, since delamination and shear fractures occurred during ejection, as seen in Fig. 4 (1.). The reason for this is, on the one hand, that smaller aluminium particles have lower limiting strains, reducing the deformation ratio. On the other hand, particle rearrangement of the finer particles represents the dominant mechanism of densification as investigated by [3]. Another reason constitutes the higher friction between insert wall and powder mixture, which lead to sticking effects during ejection. Furthermore, the higher inner friction within the powder leads to a reduced deformation ratio, due to reduced force transmission. This promotes delamination and increases the density gradient inside of the green body, having areas of almost no compaction in the middle plane and highly compacted areas in the contact zones with both punches. Increasing the pressure up to 600 MPa shear cracks appear and the green body additionally cracks during ejection. Thus, the maximum pressure applied was reduced to 600 MPa for the following experiments.

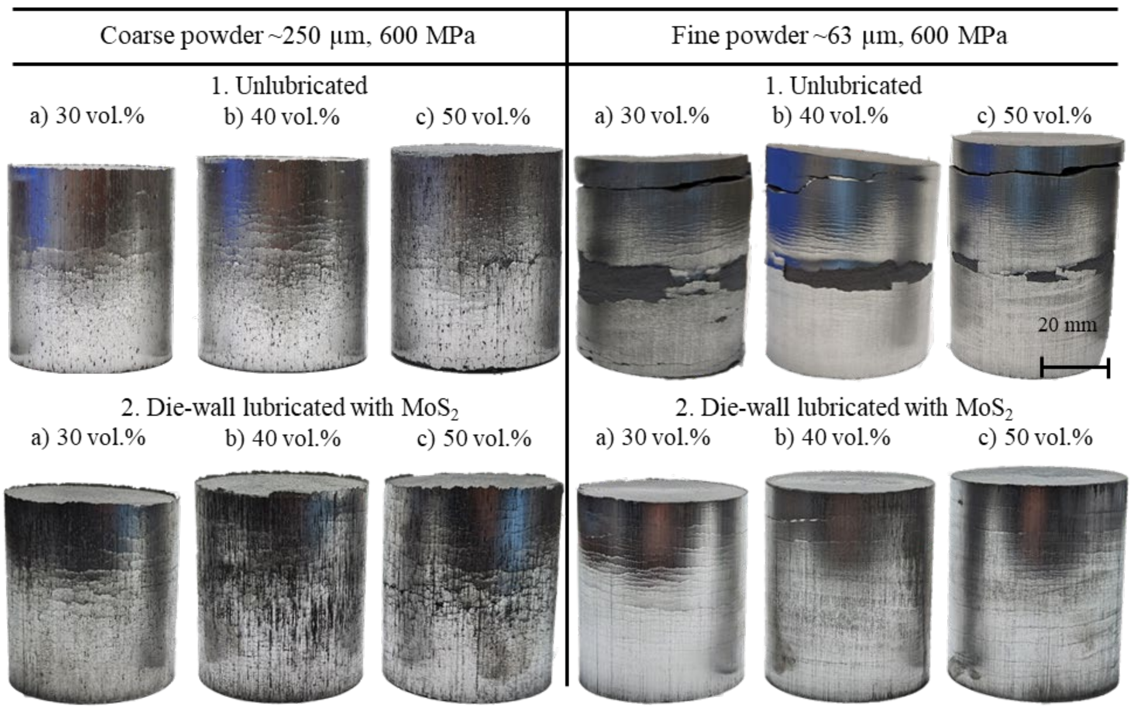


Fig. 4. Produced green billets with a constant pressure of 600 MPa, 1) Unlubricated 2) Insert-wall lubrication.

Compaction with a sprayed insert-wall lubrication, containing MoS_2, at 600 MPa achieved delamination-free green bodies for both the fine as well as the coarse powder mixtures (see Fig. 4 (2.). In the following, the resulting densities obtained from the compression experiments using insert wall lubrication (MoS_2) are presented in Fig. 5. The trend towards lower densities at higher particle loadings could be confirmed for both particle sizes and resulted from the higher amount of elastic energy absorbed by the SiC_p. Compared to the unlubricated densities of F60 particle size, an improvement in compressibility of approximately 2 % could be observed. Again, the lowest pressure of 300 MPa resulted in insufficient compaction for particle loadings of 40 and 50 vol.% as indicated by the red line in Fig. 5.

In conclusion, the fine powder showed a reduced compressibility compared to the coarser powder after consolidation. As already shown by the results of the unlubricated experiments, one reason for this is the friction between insert wall and powder, which is principally higher with fine than with coarser powder. As a consequence, higher friction results in lower consolidation pressures during densification. In this respect, the difference between measured force of the upper punch compared to the measured force of the lower punch, which corresponds to the friction loss, was 15.2 % higher for fine powder than for coarse powder at 300 MPa. At 450 MPa, the value was 32.0 % higher, and 51.5 % at 600 MPa. Another reason for the lower densification of the finer powder is the inner friction of the powder particles, since the porous structure has more contacts as well as smaller voids in the finer powder mixture.

In addition, the experiments carried out have shown that the limit for the lowest density, at which just sufficient compaction occurs in the middle plane of the green body, in general is nearly the same for fine powder compared to coarser powder. For all three particle loadings and both particle sizes, an insufficient consolidation is reached at densities below 77 % resulting in loose powder in the middle separation plane. Particle loadings of 40 and 50 vol.% SiC_p should be manufactured with pressures of 450 MPa or 600 MPa. Higher pressure than 600 MPa favors shear and delamination cracks without lubrication as well as increases the tool wear during ejection, due to the higher elastic energy absorbed by the SiC. Therefore, higher pressures should only be used for significantly higher required green body densities.

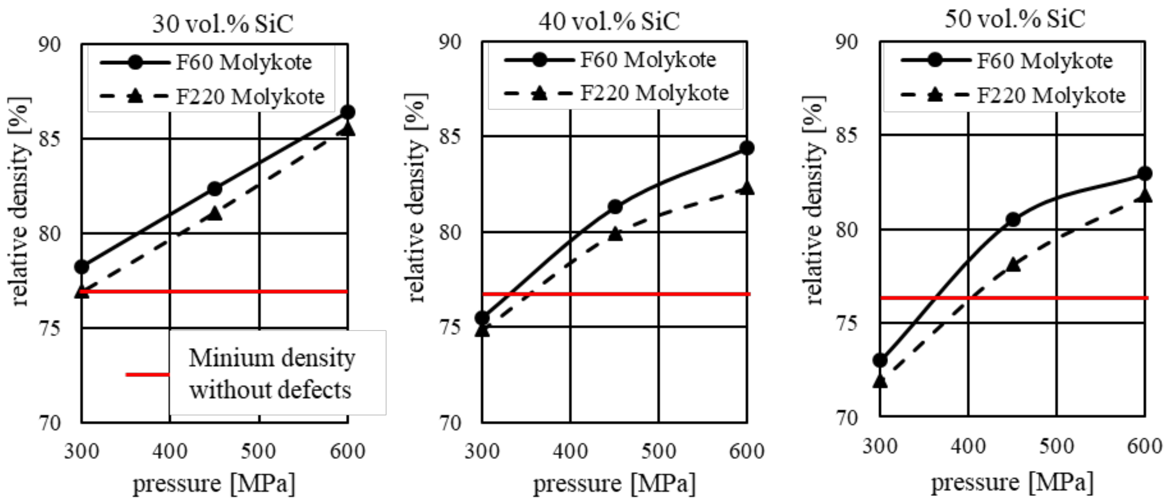


Fig. 5. Compressibility curves for both particle sizes using insert wall lubrication.

Summary

In this paper, the consolidation process of highly reinforced aluminium powders with SiC_p contents up to 50 vol.% were investigated. Thereby, the influence of particle loading and size of the powder mixture on the achieved density of the green bodies to be consolidated was analysed. Subsequently, the compaction parameters regarding compaction pressure as well as lubrication effects onto the produced densities of the green bodies were determined. The main findings are summarized according to the obtained results:

- The coarser powder mixture exhibited a lower wall friction as well as a higher compressibility during compaction, resulting from lower inner friction as well as a high plastic deformation of coarser aluminium particles. In general, it was found for all powder mixtures investigated that the green body must exhibit a density of at least 77 % after consolidation in order to ensure sufficient plastic deformation of the aluminium powder and thus avoid a delamination plane in the middle of the green body.
- Finer powder mixtures showed a higher insert wall friction, especially during the ejection of the green bodies, resulting in shear and delamination fractures. Green bodies without insert wall lubrication could not be compacted. One reason for shear and delamination defects was the higher friction inside the powder. For finer powders the main densification mechanism is particle rearrangement, resulting in high particle to particle contacts as well

as smaller voids inside the green body compared to the green bodies with coarse powder. Thereby, the inner friction reduced the force transmission inside the powder, causing high density gradients inside the green body height and failure of the form-locking bonds.

- Particle loading reduced the achieved density, due to the absorbed elastic energy by the SiC_p for both particle sizes of the powder decreasing the plastic deformation of the aluminium powder. A higher amount of SiC_p results in lower densities of the final green bodies.
- Compaction pressure improved the density achieved for both particle sizes of the powder, which can be attituded to the increased plastic deformation of the aluminium particles and the resulting bonds between the particles.
- Unlubricated experiments using fine powder mixtures resulted in delamination in the middle plane of the green bodies as well as shear cracks near the contact areas of the punches, due to high insert wall friction during compaction as well as during ejection. Insert wall lubrication enabled the manufacturing of green bodies with the finer powders, and reduced the ejection forces acting on the green body. In general, insert wall lubrication improved the achieved densities compared to the unlubricated green bodies by 2 % for the coarser powder mixtures, due to the reduced friction energy absorbed by the insert. Coarser powder reached higher densities compared to the finer powder, resulting from the higher friction energy absorbed by the insert as well as the higher inner friction in the finer powder mixture during compaction.

For future research, the process parameters of lubricated insert wall with a compaction pressure of 600 MPa will be used, as the results showed defect-free green bodies for both particle sizes with sufficient densification. For semi-solid forming of brake disc-shaped components the green bodies will be heated into a semi-solid state and subsequently formed using different process parameters. Thereby, the influence of the process parameters onto the particle distribution of the final component will be further investigated.

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References

[1] G. O’Donnell, L. Looney, Production of aluminium matrix composite components using conventional PM technology, Mater. Sci. Eng. A 303 (2001) 292-301. https://doi.org/10.1016/S0921-5093(00)01942-0
[2] A. Storz, T. Schubert, T. Weissgärber, C. Seyboldt, K.R. Riedmüller, Efficient processing of Metal-Matrix-Composites by combining Direct Pressure Sintering and subsequent Thixoforging, Mater. Sci. Forum 825-826 (2015) 167-175. https://doi.org/10.4028/www.scientific.net/MSF.825-826.167
[3] H.R. Hafizpour, A. Simchi, Investigation on compressibility of Al-SiC composite powders, Powder Metall. 51 (2008) 217-223. https://doi.org/10.1179/174329007X22250
[4] S.N. Ahmad, J. Hashim, M.I. Ghazali, The effects of porosity on mechanical properties of cast discontinuous reinforced metal- matrix composite, J. Compos. Mater. 39 (2005) 451-466. https://doi.org/10.1177/0021998305047096
[5] J. Hashim, L. Looney, M.S.J. Hashmi, Particle distribution in cast metal matrix composites - Part I, J. Mater. Process. Technol. 123 (2002) 251-257. https://doi.org/10.1016/S0924-0136(02)00098-5

[6] J. Grilo, V.H. Carneiro, J.C. Teixeira, H. Puga, Manufacturing methodology on casting-based aluminium matrix composites: Systematic review, Metals 11 (2021) 1-30. https://doi.org/10.3390/met11030436
[7] Y. Wu, G.Y. Kim, Compaction behavior of Al6061 and SiC binary powder mixture in the mushy state, J. Mater. Process. Technol. 216 (2015) 484-491. https://doi.org/10.1016/j.jmatprotec.2014.10.003
[8] J. Sun, G. Chen, B. Wang, G. Chen, W. Tang, Fabrication, microstructures, and properties of 50 vol.% SiCp/6061Al Composites via Hot Pressing, J. Mater. Eng. Perform. 28 (2019) 2697-2706. https://doi.org/10.1007/s11665-019-04071-6
[9] M. Balog, P. Krizik, M. Nosko, Z. Hajovska, M.V.C. Riglos, W. Rajner, D.S. Liu, F. Simancik, Forged HITEMAL: Al-based MMCs strengthened with nanometric thick Al2O3 skeleton, Mater. Sci. Eng. A 613 (2014) 82-90. https://doi.org/10.1016/j.msea.2014.06.070
[10] L.A. Dobrzański, A. Włodarczyk, M. Adamiak, The structure and properties of PM composite materials based on EN AW-2124 aluminum alloy reinforced with the BN or Al2O3 ceramic particles, J. Mater. Process. Technol. 175 (2006) 186-191. https://doi.org/10.1016/j.jmatprotec.2005.04.031
[11] K. Siegert, P. Unseld, J. Baur, F. Kauffmann, E. Arzt, K.v. Niessen, Thixoforging of continuous fiber-reinforced AlSi/AlMg-alloys, Int. J. Mach. Tool. Manuf. 46 (2006) 1227-1232. https://doi.org/10.1016/j.ijmachtools.2006.01.037
[12] K.R. Riedmüller, M. Liewald, Material efficient production of complex (hybrid) components using semi solid forming processes, AIP Conf. Proc. 1769 (2016) 030001. https://doi.org/10.1063/1.4963417
[13] X. Deng, N. Chawla, Modeling the effect of particle clustering on the mechanical behavior of SiC particle reinforced Al matrix composites, J. Mater. Sci. 41 (2006) 5731-5734. https://doi.org/10.1007/s10853-006-0100-1