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Review Article

Effect of severe plastic deformation process on microstructure and mechanical properties of AlSi/SiC composite

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ABSTRACT

AlSi11 matrix composites reinforced with SiC particles have been fabricated by high pressure die casting assisted by ultrasonic mixing. Severe plastic deformation process was performed on the as cast AlSi/2wt.%SiC composite using the modified channel with the twisted output (TCAP) at three temperatures of 350, 400 and 450 °C. The TCAP tool consists of helical part in the horizontal area of the channel allowing for simulation of back pressure and thus resulted in the increase of extrusion force and local plastic deformation. The most important issue of the presented investigations was to refine dendritic cast microstructure of the prepared composite during one pass of TCAP. As a result, a composite with a matrix containing equiaxed α(Al) grains of few microns depending on TCAP temperature and β(Si) particles of about 1 μm in size with twinned structure was produced. The dynamic recrystallization process during TCAP at 450 °C manifested by the grain growth of α(Al) and decrease of dislocation density was observed to affect the sample hardness causing its decrease. The mechanical properties of the composite extruded by TCAP were measured by compression testes and the maximum plastic deformation of about 35% and the compressive strength near 300 MPa were observed. These values were similar for all samples extruded at temperatures between 350 and 450 °C and generally are better comparing with AK11 alloy subjected to different plastic deformation processes.

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1. Introduction

Aluminium metal matrix composites (AMMCs) have attracted much attention since combining the advantages of both the aluminium alloys (low density, high ductility) and the reinforcements (high strength and high elastic modulus). As a consequence, they show superior specific strength, high specific stiffness, low coefficient of thermal expansion and outstanding wear resistance [1]. Development of a new processing techniques leads to the improvement of material properties and creates opportunities for new applications. One of them is the Severe Plastic Deformation (SPD) process realized in the Equal-Channel Angular Pressing (ECAP) [2] allowing the grain size refinement in the entire volume of the material without changing its external dimensions. During last years, many works related to the application of SPD techniques in the processing of aluminium, magnesium, titanium and copper base alloys have been reported, however still only a few papers concerning composites have been found [3–15]. Furthermore, most of them is related to composites based on magnesium [3,4], aluminium [5–14] and titanium [15] alloys reinforced with SiC [3,4,7,8,10,14] and Al_2O_3 [5], TiB_2 [13], TiC [16–18], carbon nanotubes [9], graphite [12] or Al–Cu–Ti metallic glasses particles [6].

Nowadays, the research interest in Al-matrix composites reinforced with various particles and subjected to intensive plastic deformation has substantially increased [19–26] due to their large application potential. In Ref. [19] the Al6082 alloy was reinforced by Si_3N_4 particles for further enhancing its strength and stiffness. When subjected to SPD the aluminium solid solution changed the grain size from micron to submicron level size by the application of load. The scanning electron microscope revealed the distribution of Si_3N_4 and increased hardness as compared to the as cast Al6082 alloy. In Ref. [20] the importance of using ECAP technique to improve ceramic particles incorporation in the metal matrix was confirmed in the case of AA7075-ZrO_2 composite. ECAP was found to improve the lightweight properties and superior performance of AA7075-ZrO_2. In Ref. [21], it was demonstrated that a nanoscale microstructure of Al–Mg hybrid system formed after high pressure torsion (HPT) processing contains a different types of intermetallic compounds within an Al matrix and may lead to the formation of metal matrix nanocomposites at the disk edges. The investigation showed a decrease of density at the disk edges confirming the potential of HPT process to fabricate materials with exceptionally high strength-to-weight ratios. Moreover, many advantages of using ECAP as a powder consolidation method for production of Al–SiC composites reinforced with ceramic particles have been presented in Ref. [22]. It was demonstrated that ECAP process has shown beneficial influence on consolidation of nano-sized particles eliminating problems relate to formation of conglomerates. In Refs. [23,24] has been reported that the mechanical strength of Al–SiC was improved almost 4 times after 8 ECAP passes, with respect to the as-stir cast composites. The distribution of SiC reinforcement particles was improved after applying ECAP while the size of both reinforcing particle size as well as porosity was reduced. Additionally, the composite remained ductile after the ECAP process. What is more, there are only a few papers concerning Al–Si base alloy strengthened with SiC particles. In Ref. [25], the SiC_p/Al–Si composites were consolidated by powder metallurgy (PM) technique and further processed by ECAP. The microstructure of samples after ECAP was significantly refined, the pores were eliminated and the reinforcement

Table 1 – Chemical composition commercial AlSi11 alloy used as a matrix for AlSi/2wt.%SiC composite.

Element concentration [weight %]									
Si	Fe	Cu	Mn	Mg	Cr	Ni	Zn	Ti	Al
9.77	0.82	0.011	0.06	0.32	0.0039	0.0024	0.022	0.05	balance

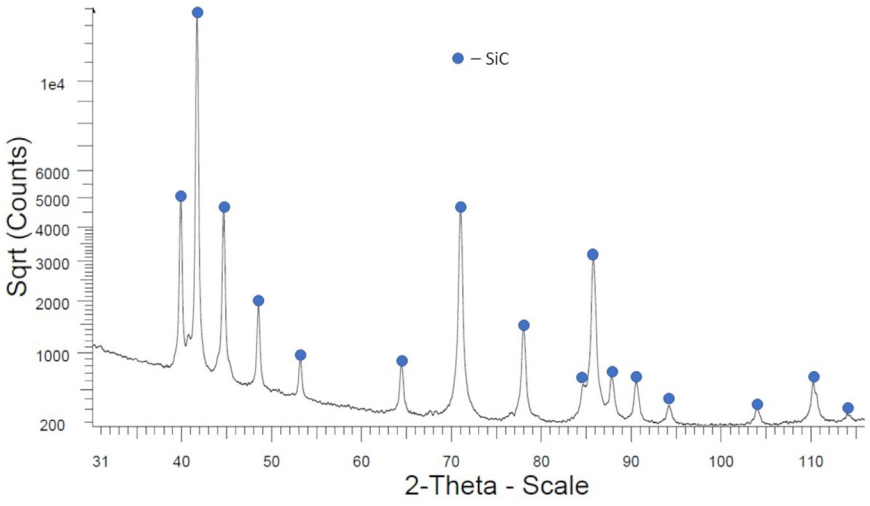

Fig. 1 – X-Ray diffraction pattern of SiC powders with declared particle size of about 1000 nm.

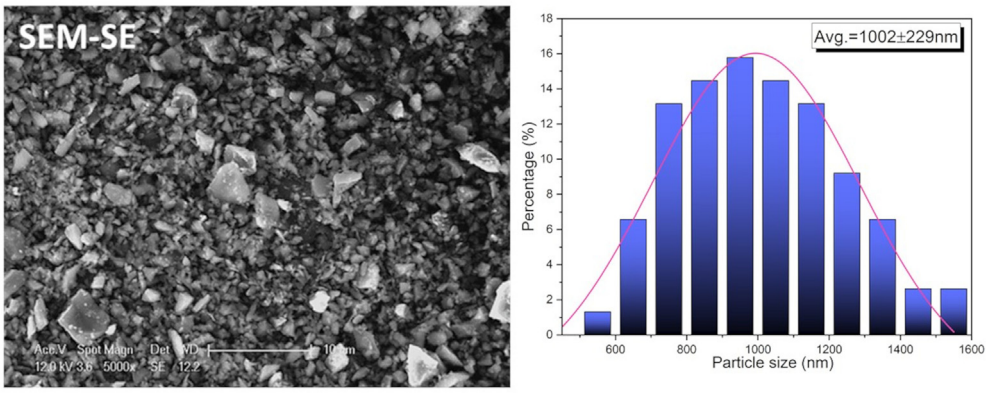

Fig. 2 – SE - SEM image of SiC particles with the size 1000 nm declared by the manufacturer and histogram of particle size distribution.

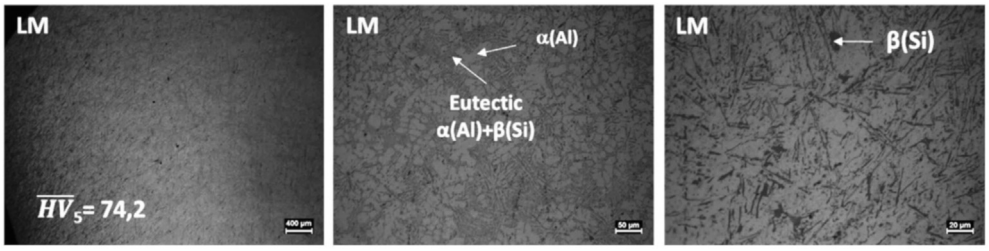

Fig. 3 – LM micrographs taken at different magnification showing microstructure of AlSi11 base alloy.

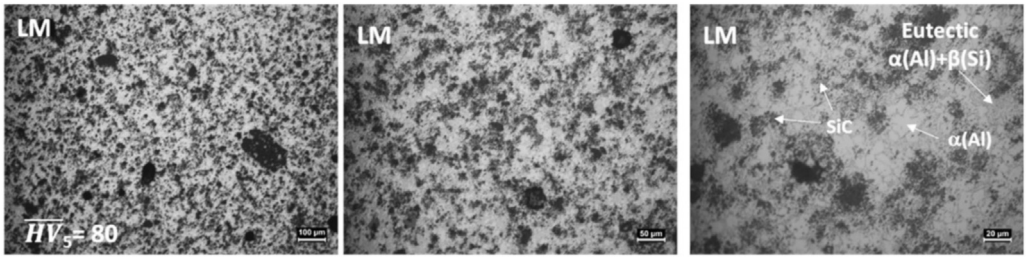

Fig. 4 – LM micrographs taken at different magnification showing microstructure of AlSi/2%SiC composite obtained by high pressure die casting assisted by ultrasonic mixing.

particles were homogenously embedded in the metal matrix. The composite after 8 ECAP passes consisted of ultrafine Al matrix grains (~600 nm) modified by uniformly dispersed Si and SiC_p particles, while the composite relative density approached 100%. Both, hardness and wear resistance of the composites were markedly improved as compared to the PM composite. Accumulative roll bonding (ARB) process was used as a very effective method for improving the microstructural and mechanical properties of the Al356/SiCp composite in Ref [26]. It was found that with increased number of ARB cycles the aforementioned advantages were found:(i) the uniformity of the Si and SiCp in the aluminium matrix was improved, (ii) the Si particles became finer and more spheroidal, (iii) the free zones of Si and SiC particles disappeared, (iv) the porosity of composite decreased, (v) the bonding quality between SiCp and matrix improved, and (vi) mechanical properties of the composites were improved. Overall, SPD processes are carried out at the lowest possible temperatures in order to avoid the recovery and recrystallization processes, which deteriorate the mechanical properties due to the grain growth. Nevertheless, for hard-deforming materials such as magnesium alloys [27] or in order to improve the degree of densification of composites produced by powder metallurgy [6], the temperature of SPD processes are carried out at elevated temperatures. Authors in Ref. [27] stated that higher temperature of ECAP of magnesium base alloys promotes dynamic recrystallization processes during strain accumulation, therefore well-defined grain boundaries and a low dislocation density within grains were observed. On the other hand, the application of higher consolidation temperature of Al–Cu–Ti metallic glass reinforced aluminium matrix composite leads to higher deformability of powders, the effectiveness of shear deformation and degree of conformation between neighboring particles were therefore improved [6]. Therefore, the influence of temperature during SPD processes is a complex phenomenon and mostly depends on the type of processing material.

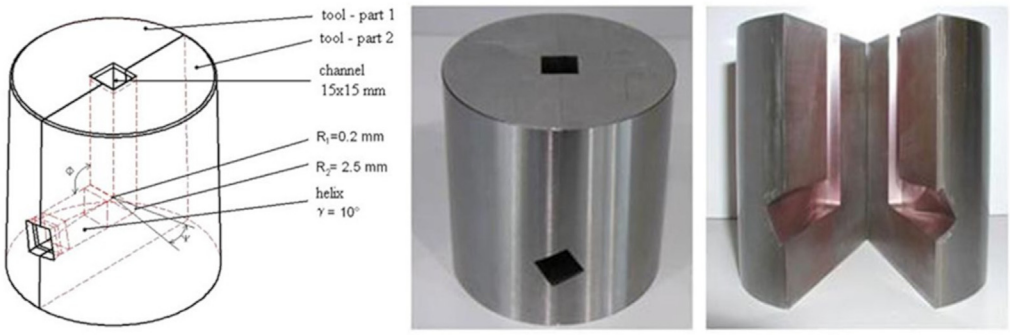

Fig. 5 – Geometry of tool and photographs of tool used in TCAP process.

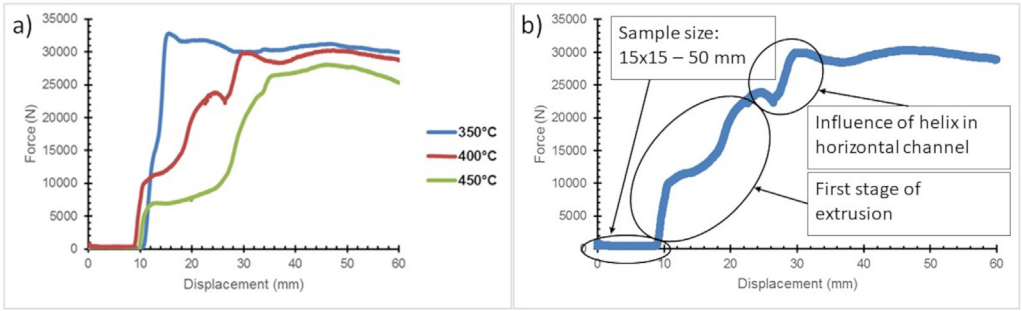

Fig. 6 – a) Graphs recorded during TCAP showing the force versus displacement of extruded sample at different temperatures and b) three characteristic stages on the curve of sample extrusion at 400 °C.

Fig. 7 – Photographs of samples after extrusion and method of cutting the sample for further microstructural investigations.

A small number of studies on Al–Si base composites in the as-cast state following by SPD processes motivated us to undertake the present studies on the near eutectic Al–Si cast alloy strengthened with SiC particles. The composite was produced using a high pressure die casting assisted by ultrasonic mixing followed by severe plastic deformation process in the modified channel with the twisted output channel (TCAP). TCAP allows to obtain the larger strain and better microstructural homogeneity of materials while the number of passes can be significantly decreased. Thus, TCAP significantly enhances the efficiency of the grain refinement process comparing to well-known ECAP. Moreover, particular attention was given to detailed microstructural studies carried out by SEM and TEM techniques in order to analyse the distribution of Si and SiC particles, their refinement and relationship with the matrix after following TCAP pass as well as its effect on mechanical properties.

2. Experimental procedure

The mixture of aluminium (99.9%) and silicon carbide powders with particles sizeof about 20–30 μm and 1000 nm, respectively, in the proportion of Al 80%vol./SiC 20% vol. was homogenized by ball milling in the argon atmosphere and then consolidated at room temperature by hydrostatic pressing with a pressure of 20 MPa. As a result, pellets with a circular cross-section were produced. The pellets were

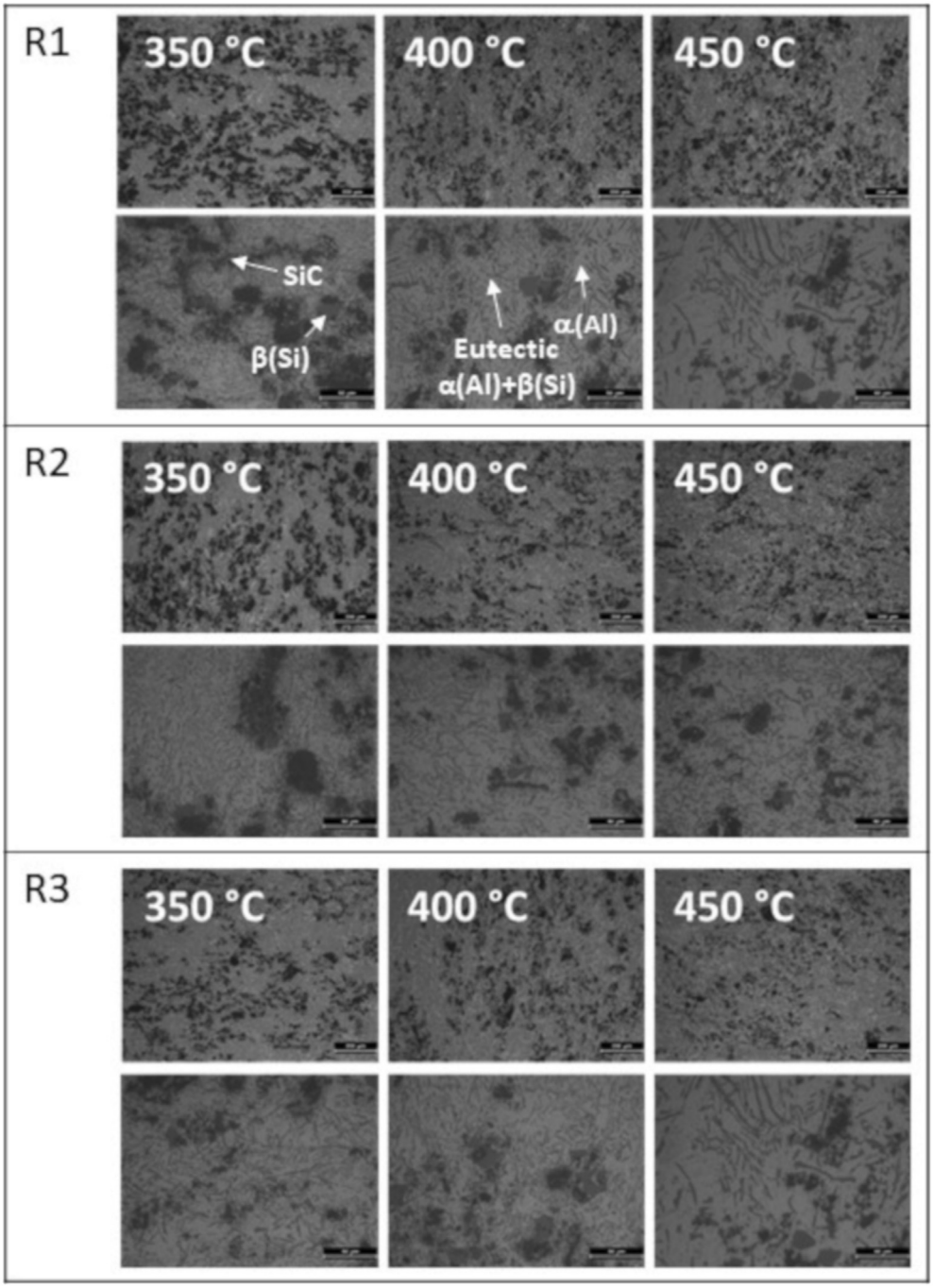

Fig. 8 – Set of LM micrographs showing the microstructure of composite in different regions (R1-R3) of deformed sample after 1 pass of TCAP process at 350, 400 and 450 °C.

subsequently extruded at temperature of 450 °C with a dimeter reduction equal 16. A rod-shaped samples were obtained. The bars of the extruded composite were gradually introduced into the metal bath at a temperature range of 745–790 °C in a quantity ensuring production of AlSi/2wt.%SiC composite. In order to enhance the distribution of the particles in the Al–Si alloy matrix, mechanical mixing (Vortex) and the effect of ultrasound (USG) were used. Then, the liquid composite slurry was pressure cast on a cold chamber machine in the temperature range of 680–740 °C. The aluminium-silicon commercial AlSi11 type alloy was used as a matrix. The chemical composition declared by the manufacturer is shown in Table 1. The SiC powder with the particles size of about 1000 nm (supplied by the manufacturers) were used for the production of composites. Moreover, the powders were subjected to phase and microstructural analysis. Based on the XRD analysis: SiC powders were identified as hexagonal SiC–6H (P63/mc) structure (Fig. 1).

In order to verify the average particles size of the supplied powders and determine their ability for agglomeration, SEM

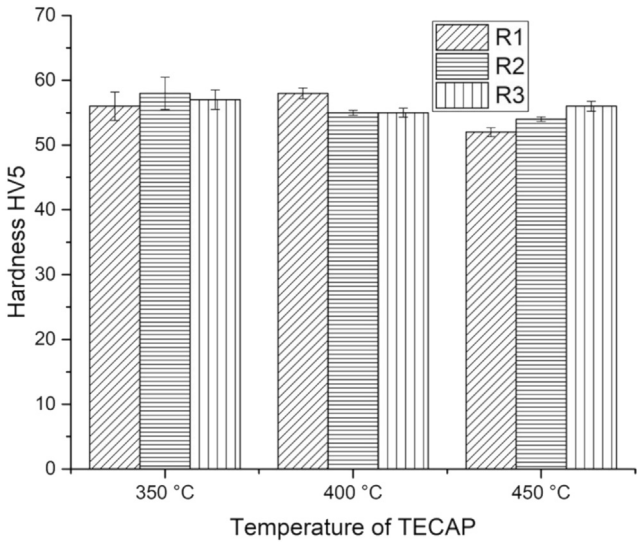

Fig. 9 – Results of micro-hardness HV5 measurements within different regions of TCAP samples at different temperatures.

microstructural observations were carried out. Based on the SE - SEM images, particle size analysis was performed. Fig. 2 presents an example of SE - SEM image of SiC particles with the size of 1000 nm declared by the manufacturer and particle size distribution histogram.

One can see that particles have mainly angular shape characteristic for SiC powders [28] due to the type of manufacturing technology. Histogram of particles size distribution possess almost normal distribution with slight positive skew while the average particle size was found to be 1002 nm with the standard deviation of 229 nm. Thus it can be stated that obtained values are close to those declared by the manufacturer.

Severe plastic deformation process of as cast AlSi/2wt.%SiC composite was realised in the modified channel with the twisted output channel (TCAP). Fig. 3 presents the geometry of a tool used for this process. The TCAP tool consisted of helical part in the horizontal area of the channel with the angle of lead $\gamma = 10^{\circ}$. The main role of helix was to simulate a back pressure and thus to increase extrusion force and local plastic deformation during one pass. The extruder, matrix and formed material are heated up to the appropriate temperature before the forming process. A lubricant of the type: Thermocup 1200 was used in the forming process.

Three optimal temperatures of TCAP process were chosen 350, 400 and 450 °C taking into the account the strength of the tool material and difficulties in plastic deformation of AlSi/2wt.%SiC composites. Microstructure and chemical composition of the composites were investigated by light microscope (LM) LeicaDM IRM, scanning electron microscope (SEM) FEI ESEM XL30 equipped with X-ray energy dispersive spectrometer EDAXGEMINI 4000 and transmission electron microscope (TEM) TecnaiG2 operating at 200 kV equipped with an Energy Dispersive X-ray (EDX) microanalyser and High Angle Annular Dark Field Detector (HAADF). In order to identify crystal structure of SiC powder and composites after TECAP process X-ray diffraction measurements (XRD) were performed by a Bruker D8 diffractometer using Co radiation. Thin foil samples for TEM observations were prepared by dimpling of 3 mm disc using Gatan dimpler and ion milling in the Lecica. The mechanical properties of the composites were tested in the compression testing machine Shimadzu equipped with an electronic measuring circuit.

3. Results and discussion

The investigations were stared with characterization of microstructure and hardness of AlSi11 base alloy chosen as a composite matrix material. Fig. 4 presents LM micrographs taken at different magnifications showing microstructure of base alloy consisting of α(Al) + β(Si) eutectic and solid solution of Si in α(Al) dendrites.

At the higher magnification also the β(Si) primary precipitates are visible. The mean hardness value is 75 HV_5. The significant change of microstructure was observed after addition of SiC powder particles into the AlSi11 base alloy. Fig. 5 presents a set of LM micrographs taken at different magnifications showing microstructure of AlSi/2wt.%SiC composite obtained by the high pressure die casting assisted by ultrasonic mixing. The refinement of α(Al) solid solution dendrites, fine eutectic and homogeneous distribution of SiC particles together with their agglomerates can be noticed. However, the matrix still possess typical cast microstructure consisting of dendrites with arms of size of about 20 μm and inter-dendritic areas filled with fine eutectic. In order to modify the microstructure towards granular with the equiaxed grains, which should improve strength and plasticity of composite, the TCAP process at elevated temperatures was applied. Fig. 6a presents the graphs recorded during TCAP processing showing the dependence of force and displacement of extruded sample after 1 pass. One can see that the required force for displacement of the sample inside the channel decreases with the temperature increase. Also, the character of the curves differs with regard of applied temperatures, whereas generally three characteristic stages on the curves can be distinguished being presented in the case of extrusion at temperature of 400 °C (Fig. 6b). At the beginning of the process, no force increase associated with a free movement of sample was observed until it has reached the angular exit of the channel. The duration of this stage depends on the length of the sample. The second stage was characterised by the intense increase of the force connected with the movement of the sample through the angular channel and increase of the flow stress. In the last stage reappearing increase of the force after its slight drop occurs. It is associated with additional plastic deformation due to helix exit in the horizontal channel. It indicates that application of such geometry of a tool allowing to obtain higher plastic deformation during one pass extrusion in comparison to standard ECAP process.

Fig. 7 shows photographs of samples after following passes of channel extrusion and a way of sample cutting, which is important for further microstructural investigations. One can see that all samples possess the network of cracks of their surfaces but the number of cracks decreases as the temperature of extrusion increases. In order to determine the degree of

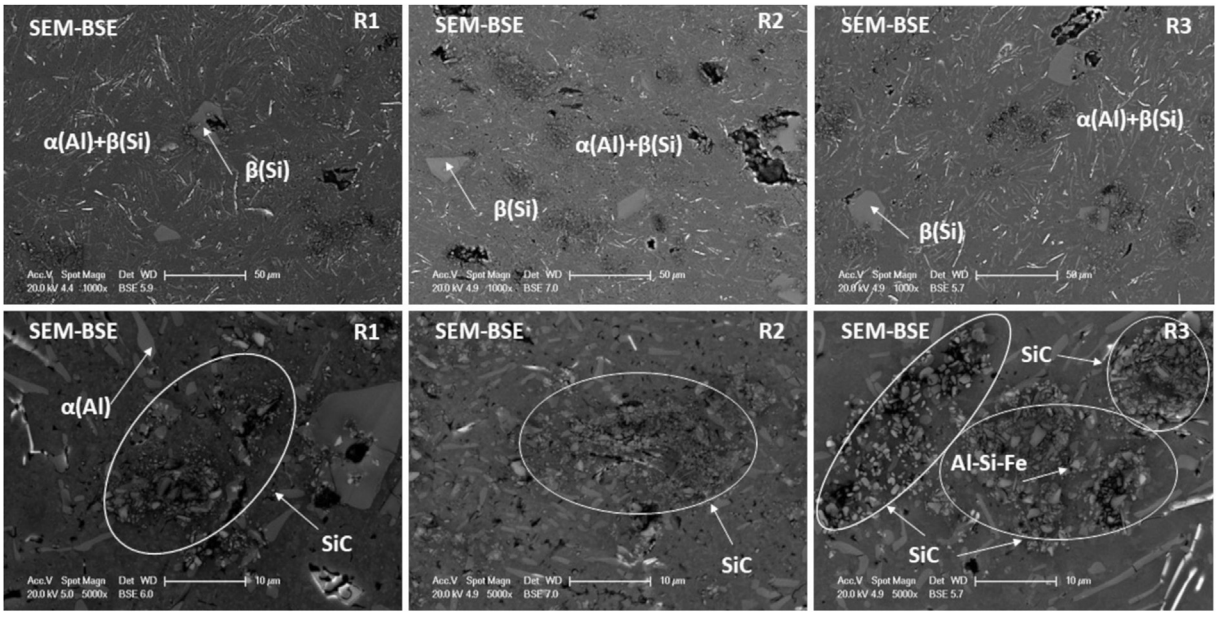

Fig. 10 – Set of SEM BSE micrographs showing the microstructure of composite in different regions of the deformed sample after 1 pass of TCAP process at 400 °C.

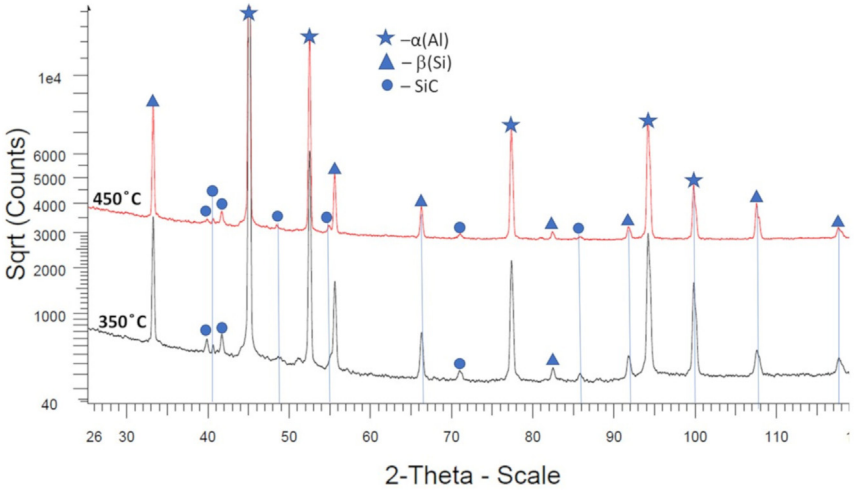

Fig. 11 – X-Ray diffraction patterns of samples TCAP-ed at temperatures of 350 and 450 °C.

homogeneity of the microstructure, the samples were cut in three places as shown in Fig. 7.

Fig. 8 presents set of LM micrographs showing the microstructure of composite after one TCAP pass at 350, 400 and 450 °C taken from different regions (R1-R3). One can see homogenous distribution of SiC particles without agglomerates observed in the as cast composite. Based on both electron and optical microscopy observations it can be noticed that the microstructure in regions R1, R2 and R3 differs. In the region R1 the α(Al)+β(Si) eutectic and α(Al) solid solution are coarser than in regions R2 and R3. Taking into the account measured chemical composition of the AlSi base alloy and the Al–Si phase diagram [29] it was assumed that its melting point is T_t ~ 580 °C. According to the temperature criteria for a hot plastic deformation process: $T_h > 0.4T_t$, in our case $T_h = 232$ °C, plastic deformation process was performed at 350, 400 and 450 °C. Additionally criteria for dynamic recrystallization during hot plastic deformation is as follows $T_{dr}>0.6T_t$ giving the T_{dr} value of 348 °C which is also lower than all applied temperatures of SPD process. Therefore, the main reason of existence of a such microstructural phenomena in the composite after TCAP process is associated with a possible various stages of the recrystallization process in the investigated regions due to a different time exposition to extrusion temperatures. The temperature of 350 °C is very close to temperature of dynamic recrystallization criteria, therefore it ensures the smallest grain growth. Moreover, higher grain refinement in the R2 region may be associated with the most uniform deformation during the process. The hypothesis of a dynamic recrystallization during the TCAP process was also confirmed by the results of micro-hardness tests presented in Fig. 9. One can see that with temperature increase the micro-hardness

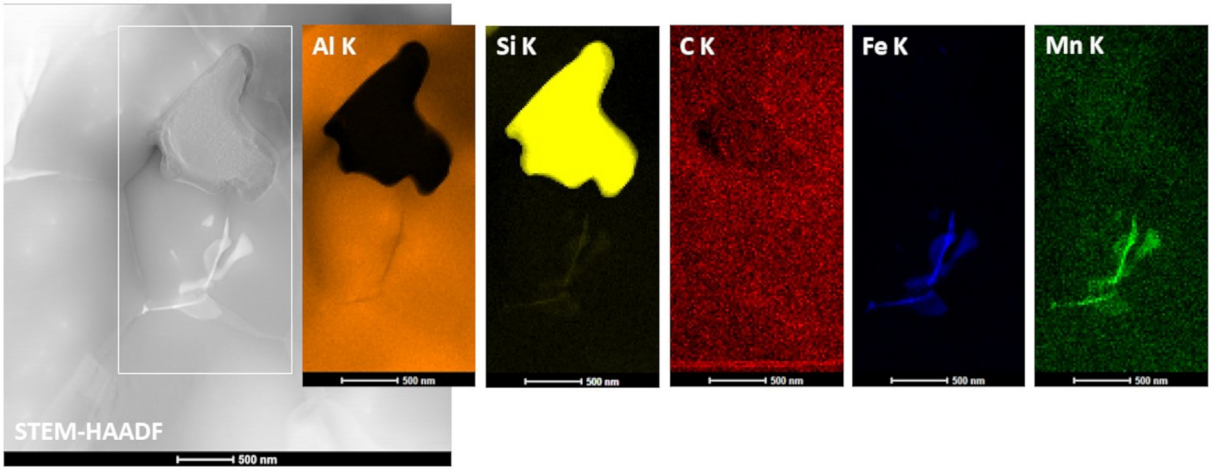

Fig. 12 – STEM-HAADF image and corresponding maps distribution of elements in the indicated area observed in the sample after 1 TCAP extrusion at 350 °C.

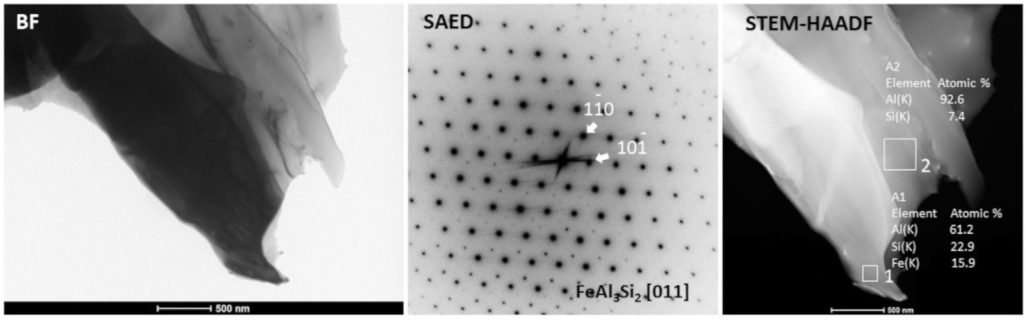

Fig. 13 – BF and STEM-HAADF microstructures, the corresponding SEAD image and the results of the EDS chemical analysis at the indicated areas in sample extruded at 350 °C.

HV_5 decreases to the lowest observed values for the sample extruded at 450 °C. Also, it is clearly visible that the hardness measurements are homogeneous in given areas and temperatures, and the influence of temperature on the standard deviation (SD) of the average hardness measurement values can be seen. The smallest SD values are presented for the TCAP-ed samples at 400 and 450 °C which proves the most homogeneous microstructure of the equiaxed grains obtained at these temperatures due to dynamic recrystallization process. Fig. 10 presents a set of SEM BSE micrographs taken from different regions of the composite after 1 TCAP pass performed at 400 °C. One can see three regions differing in microstructure containing the primary precipitates of β(Si), fine α(Al)+β(Si) eutectic and aggregations of SiC particles of size of about 20 μm in which separated SiC particles with size of about 1 μm can be distinguished. The phase composition of samples after TCAP at 350 and 450 °C was determined by X-ray diffraction presented in Fig. 11. For both temperatures, the same phases have been identified, i.e. α(Al), β(Si) and SiC carbide which means that increasing the process temperature by 100 °C does not change the phase composition. Transmission Electron Microscopy (TEM) microstructure investigations were performed for samples extruded at 350 and 450 °C in order to identify all existing phases and confirm that dynamic recrystallization has taken place. Fig. 12 presents STEM-HAADF image and corresponding maps of elements distribution in the indicated area for the sample extruded at 350 °C. The α(Al) matrix of composite contains a small amount of Mn and Fe, β(Si) particle with size of about 1 μm and another particle enriched in Fe and Mn. The C distribution map does not indicate the presence of carbides in this area, so it can be assumed that the signal comes only from contamination. The precipitates enriched with Fe and Mn were also observed in the other places of sample.

Fig. 13 shows the BF and STEM-HAADF microstructures, the corresponding SAED image and the results of the EDS chemical analysis taken form the indicated areas. Electron diffraction can be well indexed in accordance with the $FeAl_3Si_2$ phase along the [011] zone axis. The Al–Si–Fe phase was identified since the commercial aluminium-silicon alloy contains Fe impurities as was shown in Table 1. However, in this case it is visible that it has a different morphology dependently on the sample region (to compare Figs. 12 and 13) due to the application of a severe plastic deformation causing its fragmentation.

Microstructural analysis of α(Al) matrix and β(Si) particles in the sample extruded at 350 °C is presented in Fig. 14. In the case of α(Al) matrix, [011] zone axis was identified by electron

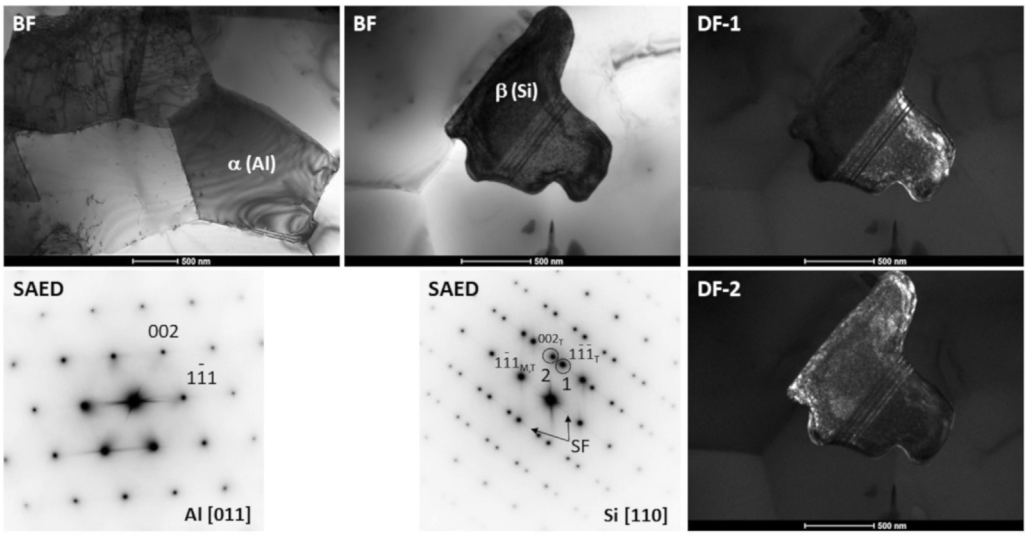

Fig. 14 – BF, DF microstructure and corresponding SAED of α(Al) matrix and β(Si) precipitates in sample extruded at 350 °C.

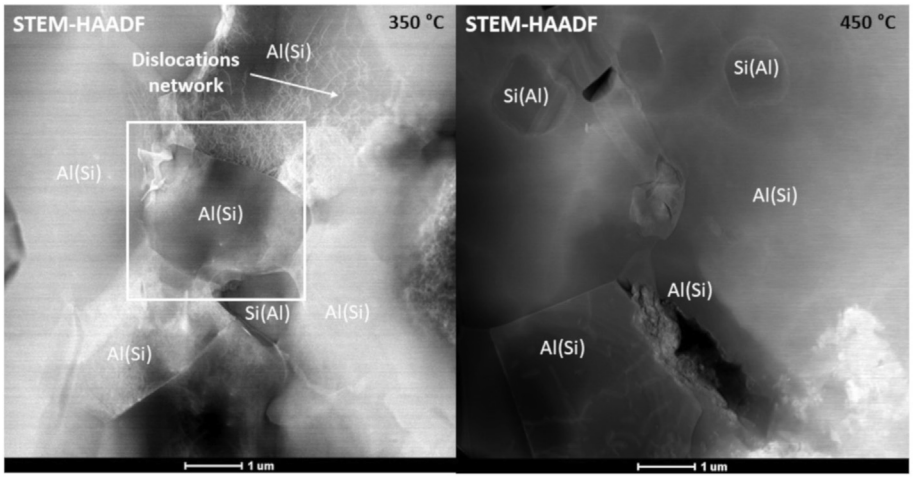

Fig. 15 – STEM-HAADF images of samples deformed by TCAP at 350 and 450 °C, respectively.

diffraction and one can see equiaxed grains of size of about 2 μm and a high density of dislocations. Whereas β(Si) particles of size of about 1 μm is characterising by the twinned microstructure confirmed by the [110] zone axis electron diffraction with the (1–11) twinning plane. The presence of both dislocations and twins in the studied sample results from a high plastic deformation during the TCAP process at elevated temperature. The twining process of eutectic Si phase in Al–Si alloy can be associated with both Si growth [30] and/or twinning deformation during the tensile deformation [31].

In this case, taking into account the morphology of the observed Si particle, the growth process seems to be more likely than plastic deformation. However, due to the occurrence of frequent stacking faults (SF) (Fig. 14) in the twinned Si particle manifested by streak diffraction reflects the plastic deformation cannot be excluded. As was mentioned before, the temperature of dynamic recrystallization for the investigated composite is around T_{dr}~348 °C, therefore significant differences in microstructure can be expected for selected samples for TEM observations. Because the SPD process is associated with generation of large amounts of microstructural faults, in particular dislocations and changes in the shape of grains, while the processes of recovery and recrystallization cause annihilation of dislocation and growth of grains, the STEM-HAADF observation technique is a very useful tool because it is sensitive to chemical composition change and structural faults. Fig. 15 shows STEM-HAADF images of samples deformed by TCAP at 350 and 450 °C, respectively. In both cases one can see equiaxed grains of α-Al(Si) and β-Si(Al) solid solutions. While the Si(Al) grains are practically the same for both cases, reaching of about 1 μm in size, the Al(Si) grains are fundamentally different. Refinement of Si particles down to 1 μm is similar like observed in Al–Si/SiC composites after ARB treatment [26]. For a lower

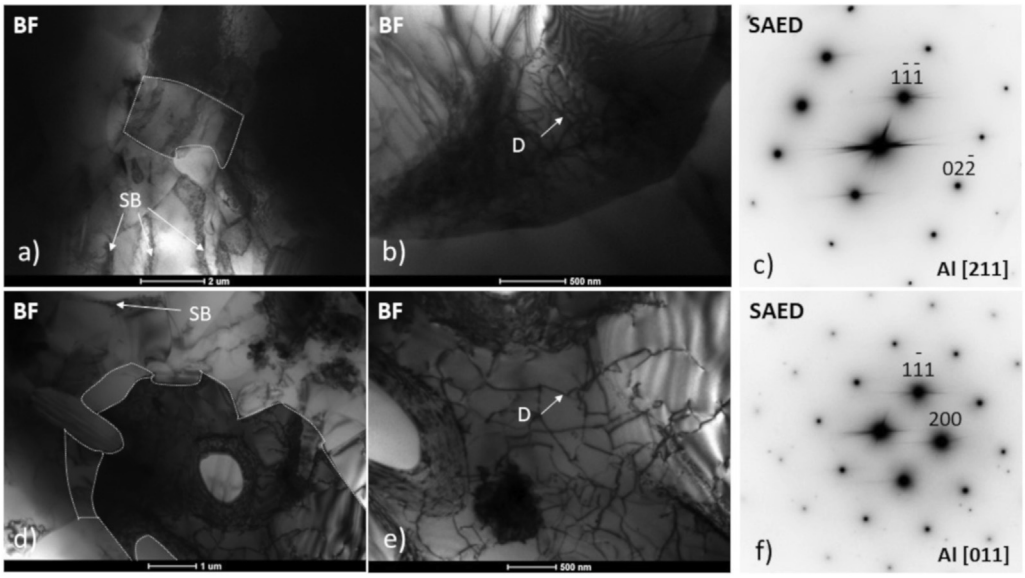

Fig. 16 – Set of BF microstructures taken at different magnifications and corresponding SAED from the a solid solution of composite extruded by TECAP at 350 °C (a), (b), (c) and 450 °C (d), (e) and (f).

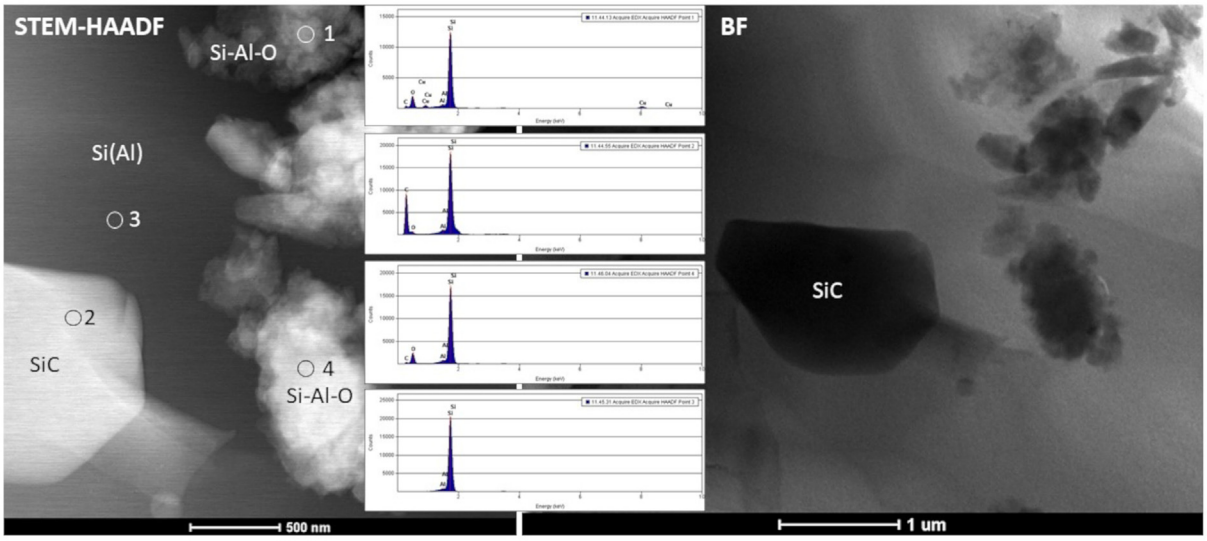

Fig. 17 – STEM-HAADF image and results of EDS point analyses and corresponding BF microstructure.

temperature of TCAP, smaller grains decorated with a large amount of dislocations forming a typical network microstructure characteristic for large plastic deformation can be observed. In the case of higher temperature, the grains of Al(Si) are significantly larger, with lower amount of dislocations. Thus it can stated that during extrusion at 450 °C or even 350 °C recrystallization and grain growth processes occurred. This was also confirmed by observations performed in a bright field (BF) and selected area electron diffraction (SAED). Fig. 16 presents the set of BF microstructures taken at different magnifications and corresponding SAED patterns of composite extruded by TCAP at 350 and 450 °C. One can see the deformed Al(Si) grains with [211] and [011] zone axis for composite extruded at 350 and 450 °C, respectively. The substantial differences in grains size is visible at lower magnifications and in densities of dislocations at higher one. In both cases well-developed subgrain boundaries (SB), built from dislocations arranged in nets at subgrain boundaries as result of mechanical shearing during TCAP can be observed. Number of such subgrains is significantly larger for sample TCAP-ed at 350 than 450 °C. Simultaneously, in both cases the recrystallized grains with boundaries without dislocations are presented with dislocation (D) networks forming of walls and cells in interior of grains. This type of microstructure proves that recrystallization occurring during TCAP of SiC/AlSiC composite at 350 and 450 °C. Fig. 17 shows STEM-HAADF image and EDS point analyses results and corresponding BF microstructure showing the SiC particle with a size of about 1 μm. The thin foil in this area is not enough thin for

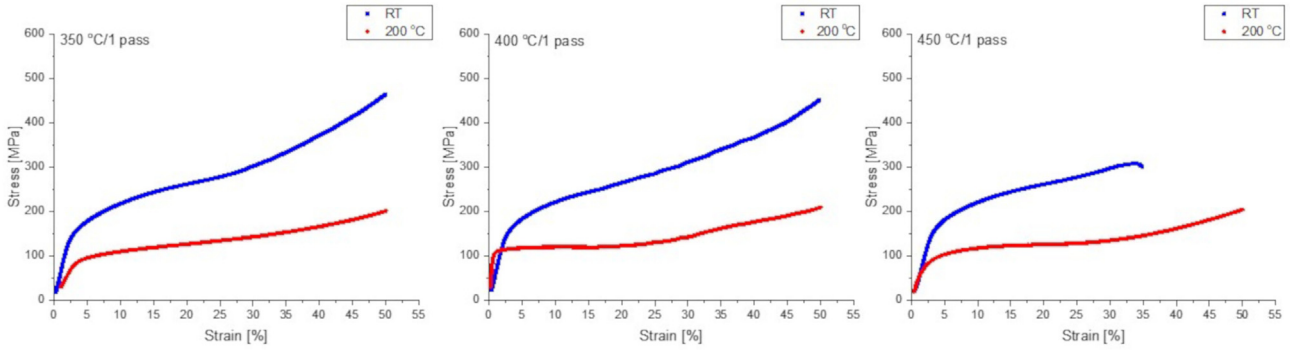

Fig. 18 – Set of stress–strain curves obtained for AlSi/SiC composites after 1 pass at 450 °C of TCAP recorded at RT and 200 °C.

electron diffraction conditions, thus the existence of the SiC particles is confirmed only by EDS analyse. Moreover, identifications of SiC particles in AlSi/SiC composite is difficult since Si(Al) solid solution after TCAP process is in a form of regular particles (Fig. 12) of size close to SiC (Fig. 2) initially introduced into the aluminium matrix. Applying EDS analyse in STEM-HAADF mode always show a small amount of C in a spectrum due to contamination of the sample caused by interaction with the electron beam, therefore it is difficult to distinguish which particle we are dealing with. Nevertheless, taking into the account EDS spectra one can see the significant increase of intensity of C peak in point 2 corresponding to SiC particle compared to the point 1 corresponding to Si(Al) solid solution indicating the existence of SiC particle.

Due to the small amount of samples obtained after TCAP processes the mechanical properties were determined using a compression tests at two temperatures i.e. room temperature (RT) and 200 °C. Fig. 18 shows the set of stress–strain curves obtained for AlSi/SiC composites after 1 pass of TCAP recorded at RT and 200 °C. In all cases a similar character of stress–strain curves is visible. For larger deformations (over 25%), a change in the slope of the strengthening curve is visible, which may be associated with achieving maximum deformation by the compressed samples and the contribution of the anvils of the testing machine to further deformation and increase in stress. The maximum plastic deformation and compressive strength of the tested samples were determined by the tangent method at the inflection point of the curves. It can be seen that at RT the maximum plastic deformation is about 35% and the compressive strength about 300 MPa, while values are similar for all investigated samples. Results are alike observed for ARB deformed AlSi/SiC composites [26], however the strength after TCAP corresponds to a few ARB passes due to a higher degree of deformation in TCAP. At 200 °C, there is essentially no change in toughness, only a decrease in strength between 100 and 150 MPa. Lower strength values show samples after the TCAP process performed at higher temperatures. It is worth noting that the obtained mechanical properties of composites after the TCAP process are definitely better than the properties of hypoeutectic AK11 alloys subjected to cryo-rolling [32], as well as of AK11/Al_2O_3 composite [33].

4. Conclusions

The obtained results and their interpretation allows to drawn the following conclusions:

1. The addition of SiC powder particles into the AlSi11 base alloy by a high pressure die casting assisted by ultrasonic mixing caused the refinement of α(Al) solid solution dendrites arms surrounded by a very fine eutectic. Nearly homogeneous distribution of SiC particles and their agglomerates were observed.
2. Three regions in the TCAP passed sample were distinguished in the primary introduction, medium rectangular and flattened exit part. They contained primary precipitates of β(Si) with similar size, fine α(Al)+β(Si) eutectic and aggregations of SiC particles of size of about 20 μm and single separated SiC particles with size of about 1 μm.
3. TCAP process was performed at 3 different temperatures 350 °C, 400 °C and 450 °C. Different regions show slightly different hardness, however generally with increasing process temperature the medium hardness decreased from 60 HV_5 for 350 °C down to 54 HV_5 for the 450 °C. These differences were caused by larger grain size after TCAP at 450 °C near 10 μm as compared to 2 μm at 350 °C accompanied by a higher dislocation density due to incomplete recrystallization. Si particles showed a similar size after processing at 350 and 450 °C.
4. TEM studies allowed to identify twins within silicon particles with (111) twinning plane. In addition, $FeAl_3Si_2$ intermetallic phase was identified using electron diffraction. EDS analysis allowed to identify Al–SiO oxides forming during high temperature processing.
5. Compression tests performed at RT indicated the maximum plastic deformation of about 35% and the

compressive strength near 300 MPa. These values are similar for all samples deformed at temperatures 350–450 °C. At 200 °C, there are essentially similar strength results and a decrease in strength between 100 and 150 MPa occurs in comparison to RT.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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REFERENCES

[1] Amirkhanlou S. A review on high stiffness aluminium-based composites and bimetallics. S. Crit Rev Solid State Mater Sci 2020;45:1. Ji

[2] Ravisankar Balasubramanian. Equal-Channel Angular pressing (ECAP). In: Aliofkhazraei Mahmood, editor. Chapter 13 in handbook of mechanical nanostructuring; 2015. https://doi.org/10.1002/9783527674947. ch13, Handbook of Mechanical Nanostructuring, First Edition. Edited by Mahmood Aliofkhazraei 2015Wiley-VCH Verlag GmbH & Co. KGaA. Published 2015 byWiley-VCH Verlag GmbH & Co. KGaA.

[3] Xu Q, Ma A, Saleh B, Li Y, Yuan Y, Jiang J, et al. Enhancement of strength and ductility of SiCp/AZ91 composites by RD-ECAP processing. Mater Sci Eng, A 2020;771:138579.

[4] Qiao XG, Ying T, Zheng MY, Wei ED, Wu K, Hu XS, et al. Microstructure evolution and mechanical properties of nano-SiCp/AZ91 composite processed by extrusion and equal channel angular pressing (ECAP). Mater Char 2016;121:222–30.

[5] Casati R, Fabrizi A, Tuissi A, Xia K, Vedani M. ECAP consolidation of Al matrix composites reinforced with in-situ γ-Al2O3 nanoparticles. Mater Sci Eng, A 2015;648:113–22.

[6] Rezaei MR, Shabestari SG, Razavi SH. Effect of ECAP consolidation temperature on the microstructure and mechanical properties of Al-Cu-Ti metallic glass reinforced aluminium matrix composite. J Mater Sci Technol 2017;33:1031–8.

[7] Mani B, Paydar MH. Application of forward extrusion-equal channel angular pressing (FE-ECAP) in fabrication of aluminium metal matrix composites. J Alloys Compd 2010;492:116–212.

[8] Ramu G, Bauri Ranjit. Effect of equal channel angular pressing (ECAP) on microstructure and properties of Al–SiCp composites. Mater Des 2009;30:3554–9.

[9] Zare H, Jahedi M, Toroghinejad MR, Meratian M, Knezevic M. Microstructure and mechanical properties of carbon nanotubes reinforced aluminium matrix composites synthesized via equal-channel angular pressing. Mater Sci Eng, A 2016;670:205–16.

[10] Qian CH, Ping L, Xue KM. Influence of deformation passes on interface of SiCp/Al composites consolidated by equal channel angular pressing and torsion. T. Nonferr. Metal Soc. 2015;25:1376–82.

[11] Ping L, Zhang X, Xue KM, Xiao L. Effect of equal channel angular pressing and torsion on SiC-particle distribution of SiCp-Al composites. T Nonferr Metal Soc 2012;22:s402–7.

[12] Saravanan M, Pillai R, Ravi K, Pai B, Brahmakumar M. Development of ultrafine grain aluminium-graphite metal matrix composite by equal channel angular pressing. Compos Sci Technol 2007;67:1275–9.

[13] Ravi K, Saravanan M, Pillai R, Mandal A, Murty B, Chakraborty M, et al. Equal channel angular pressing of Al-5 wt% TiB_2 in situ composite. J Alloys Compd 2008;459:239–43.

[14] Xu Q, Ma AM, Wang JJ, Sun JP, Jiang JH, Li YH, et al. Development of high-performance SiCp/Al-Si composites by equal channel angular pressing. Metals 2018;8:738.

[15] Han Y, Li J, Huang G, Lv Y, Shao X, Lu W, et al. Effect of ECAP numbers on microstructure and properties of titanium matrix composite. Mater Des 2015;75:113–9.

[16] Maziarz W, Wójcik A, Bobrowski P, Bigos A, Szymański Ł, Kurtyka P, et al. SEM and TEM studies on in-situ cast Al-TiC composites. Mater Trans 2019;60(5):714–7.

[17] Maziarz W, Bobrowski P, Wójcik A, Bigos A, Szymański Ł, Kurtyka P, et al. Microstructure and mechanical properties of in situ cast aluminium based composites reinforced with TiC nano-particles. Mater Sci Forum 2020;985:211–7.

[18] Wojcik A, Olejnik E, Bigos A, Chulist R, Bobrowski P, Kurtyka P, et al. Microstructural characterization and mechanical properties of in situ cast nanocomposites Al/TiC type. J Mater Res Technol 2020;9(6):12707.

[19] Nishanth B, Guruchannabasavaiah NG, Auradi Virupaxi, Bharath V, Boppana Satish Babu, Nagaral Madeva. Microstructure evaluation of Si_3N_4 reinforced Al6082 composites subjected to severe plastic deformation. In: R & D centre, department of IOP conf. Series: materials science and engineering. vol. 1013; 2021, 012019. https://doi.org/10.1088/1757-899X/1013/1/012019. IOP Publishing.

[20] Sabbar Huda M, Leman Zulkiflle, Shamsudin Shazarel B, Mohd Tahir Suraya, Aiza Jaafar Che N, Azmah Hanim Mohamed A, et al. AA7075-ZrO2 nanocomposites produced by the consecutive solid-state process: a review of characterisation and potential applications. Metals 2021;11:805.

[21] Kawasaki Megumi, Langdon Terence G. Using severe plastic deformation to fabricate strong metal matrix composites. Mater Res 2017;20(Suppl. 1):46–52. 2017.

[22] Musa Mateja Šnajdar, Schauperl Zdravko. ECAP – new consolidation method for production of aluminium matrix composites with ceramic reinforcement. Processing and Application of Ceramics 2013;7:63–8.

[23] Shehata Farouk, ElMahallawy Nahed, Mohamed Arab, Mohamed Agwa. Equal Channel Angular pressing of Al-SiC composites fabricated by stir casting. Open J Met 2013;3:26–33.

[24] Arab MS, El Mahallawy N, Shehata F, Agwa MA. Refining SiCp in reinforced Al–SiC composites using equal-channel angular pressing. Mater Des 2014:280–6.

[25] Xu Qiong, Ma Aibin, Wang Junjie, Sun Jiapeng, Jiang Jinghua, Li Yuhua, et al. Development of high-performance SiC_p/Al-Si composites by equal Channel Angular pressing. Metals 2018;8(10):738. https://doi.org/10.3390/met8100738.

[26] Amirkhanlou Sajjad, Jamaati Roohollah, Niroumand Behzad, Reza Toroghinejad Mohammad. Using ARB process as a solution for dilemma of Si and SiCp distribution in cast Al–Si/SiCp composites. J Mater Process Technol 2011;211:1159–65.

[27] Bryła K, Dutkiewicz J, Litynska-Dobrzynska L, Rokhlin LL, Kurtyka P. Influence of number of ECAP passes on microstructure and mechanical properties of AZ31 magnesium alloy. Arch Metall Mater 2012;57(3):711–7.

[28] Anggraini Lydia, Ameyama Kei. Effect of particle morphology on sinterability of SiC-ZrO_2 in microwave. J Nanomater 2012;2012:8. https://doi.org/10.1155/2012/741214. Article ID 741214.
[29] Massalski TB, editor. Binary alloy phase diagrams, vol. 1. Metals Park, OH: ASM; 1986. p. 165.
[30] Liu Xiaorui, Zhang Yudong, Beausir Benoît, Liu Fang, Claude Esling, Yu Fuxiao, et al. Twin-controlled growth of eutectic Si in unmodified and Sr-modified Al–12.7%Si alloys investigated by SEM/EBSD. Acta Mater 2015;97:338–47.
[31] Chen Jiqiang, Liu Chao, Wen Feng, Zhou Qiongyu, Zhao Hongjin, Guan Renguo. Effect of microalloying and tensile deformation on the internal structures of eutectic Si phase in Al-Si alloy. J Mater Res Technol 2020;9(3):4682–91.
[32] Immanuel RJ, Panigrahi SK. Influence of cryorolling on microstructure and mechanical properties of a cast hypoeutectic Al–Si alloy. Mater Sci Eng, A 2015;640:424–35.
[33] Chabera P, Boczkowska A, Zych J, Oziębło A, Kurzydłowski KJ. Effect of specific surface fraction of interphase boundaries on mechanical properties of ceramic-metal composites, obtained by pressure infiltration. Kompozyty 2011;11(3):202–7.

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