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Effect of friction stir processing (FSP) on microstructure and hardness of AlMg10/SiC composite

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Abstract. The AlMg10 aluminum alloy reinforced with SiC particles was subjected to friction stir processing (FSP). The composite was made by mechanical mixing and gravity casting. The mass fraction of SiC particles in the composite was about 10%. Evaluation of the effects of FSP treatment was performed by means of light microscopy, scanning electron microscopy, EDS and hardness measurement. It was found that the inhomogeneous distribution of SiC particles and their agglomeration, which were observable in the cast composite, were completely eliminated after FSP modification. The treatment was also accompanied by homogenisation of the material in the mixing zone as well as fragmentation of both the matrix grain of the composite and SiC particles. In the case of SiC particles, a change in their shape was also observed. In the as-cast composite, particles with dimensions from 30 to 60 µm and a sharp-edged polyhedral shape prevailed, while in the material subjected to friction treatment, particles with dimensions from 20 to 40 µm and a more equiangular shape prevailed. Pores and other material discontinuities occurring frequently in the as-cast composite were completely eliminated after friction modification. The recorded changes in the microstructure of the material were accompanied by an increase in the hardness of the composite by nearly 35%. The conducted investigations have shown that FSP modification of the AlMg10/SiC composite made by the casting method leads to favorable microstructural changes in the surface layer and may be an alternative solution to other methods and technologies used in surface engineering.

Key words: friction stir processing, AlMg10/SiC composite, microstructure.

1. Introduction

Friction stir processing is a new solid-state surface modifying technique. This technology is derived from FSW (friction stir welding) technology, developed by Wayne Thomas from the Welding Institute (TWI Ltd) in Cambridge in 1991, but unlike it, it is not used for joining materials, but for modifying their surface layer [1]. FSP technology is characterized by a high application potential resulting from the simplicity and universality of the method in the scope of constituting the surface layers of engineering materials. FSP technology does not require dedicated, specialized equipment, machining can be carried out using ordinary milling machines. During FSP treatment, the heat that arises from the friction of the tool on the surface of the material leads to plasticization of the material and significant changes in its microstructure and phase morphology in relation to the state before processing. The tool usually has the form of a cylinder ending in a pin, the shape and dimensions of the pin and the shoulder depend on the type of modified material, target dimensions of the modified zone, and the scope and nature of the microstructural changes planned to be obtained. The tool is rotated during machining, after which it is slowly plunged into the modified material for the entire length of the pin and then moved at a predetermined speed. Both the rotational speed of the tool and its speed of movement are the main parameters of FSP processing which determine the size and nature of microstructural changes obtained in the modified material. About 80 to 90% of the heat generated during tool friction against the material surface arises as a result of the friction of the shoulder, and the rest as a result of friction of the other surfaces of the tool against the material surface [2]. An important parameter of the process is therefore the shape and dimensions of the tool. The tool not only generates a heat which leads to plasticization of the alloy, but also causes strong plastic deformation of the material. As a result of the combined effect of heat and force, dynamic recrystallization of the material takes place in the material subjected to friction modification, shaping its final microstructure. During processing, the melting temperature of the modified material is not exceeded. It is worth emphasizing that the only source of thermal energy is friction, which makes FSP technology an environment-friendly solution. Shaping the microstructure of the surface layer of engineering materials by means of FSP machining is the research topic of numerous works and scientific papers [3–5].

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FSP technology can also be used to create a composite microstructure in the surface layer of the modified material [6–9]. During processing, it is possible to introduce additional material that serves as a reinforcing phase in the final composite. The additional material is usually placed in the grooves hollowed in the surface of the material, after which the groove is closed with a tool without a pin with a flat resistance surface, and then proper modification is carried out using a tool with a pin. For example, Sharifitabar et al. (2011) used FSP to produce an Al/Al_2O_3 nanoceramic reinforced composite. The results showed that the grain size of the stir zone decreased with an increasing number of FSP pass and the composite created after four passes had a submicron mean grain size [8]. Barmouz et al. (2011) investigated the effect of traverse speed on the microstructure and microhardness of Cu/SiC MMC layers and found enhancement in the wear resistance and a higher average friction coefficient in comparison with pure copper as well as lower strength and elongation than for pure copper [9]. An alternative solution to "groove method" is the multi-chamber method [10–12]. In this method the reinforcing material is placed in separate chambers hollowed in the surface of the sample. Such a solution significantly reduces the risk of uncontrolled material movement outside the modified area when the pin goes into the material, as well as during its movement in the plasticized material. It is also worth emphasizing that the multi-chamber method makes it possible to carry out the friction process with only one tool and can be implemented in a single or two-step variant. Another solution enabling the production of a composite microstructure is the DFSP (Direct Friction Stir Processing) method, using a special tool equipped with an internal channel supplying powder directly into the friction-modified zone [13]. In all these methods, the additional material is introduced into the matrix after its plasticizing, leading to the formation of a composite microstructure in the surface layer of the material. Another case is when a material that is already a composite is subjected to FSP [14–16]. In this case, by performing FSP treatment we have the ability to cause changes in the material microstructure and its properties, while retaining the original composite material characteristics. The type and scope of microstructural changes is to a large extent dependent on the technology used to make the composite and the primary microstructure of the material. For example Kurtyka et al. [14] studied the effect of FSP on the mechanical properties as well as the concentration and distribution of SiC reinforcement particles in the cast A339/SiC/p composite. The authors observed changes in the concentration and distribution of SiC particles and they found significant improvement in the distribution of the reinforcing phase particles in the A339/SiC/p composite. The determined anisotropy coefficient value of SiC particle distribution varied from the level of 1.8 for the starting material to 0.2 for the modified material. An increase of about 40% for the compressive strength and 30% for hardness was observed. Amirizad et al. [17] examined the microstructure and mechanical properties in the friction stir welded A356 + 15%SiCp cast composite and found that there was a more homogenous distribution of SiC particles as well as fragmentation of SiC particles and silicon needles in the eutectic phase. The consequence of the changes in the composite microstructure was improved mechanical properties such as the modulus of elasticity, yield and ultimate strength, elongation, as well as hardness in the weld zone compared to that of the base composite. Bauri et al. [15] investigated the effect of friction stir processing on the microstructure and properties of the Al–TiC in situ composite. The material was subjected to single and double FSP passes. The authors found that a single FSP pass was enough to prevent particle segregation from the grain boundaries and improve their distribution, but two FSP passes resulted in complete homogenization of the material and eliminated structural defects of the cast. After each FSP pass, refining of the grain size and significant improvement in the mechanical properties of the composite were observed.

In this study, friction stir processing of the AlMg10/SiC cast composite was carried out. The microstructure and hardness of the modified surface layer was analyzed and discussed.

2. Experimental procedure

Friction modification was applied to a composite composed of an AlMg10 aluminum alloy matrix reinforced with SiC particles. SiC technical powder with an average particle size of about 43 μm and a polyhedral shape was used. The composite was made by mechanical mixing using a propeller stirrer, after which the resulting composite suspension was gravitationally cast into a metal mold. A rod with a diameter of 40 mm was obtained in this way. The mass fraction of the SiC phase in the composite was about 10%. Plates with a thickness of 10 mm were machined from the cast billets and subjected to friction stir processing. The samples were chemically cleaned with alcohol prior to treatment to degrease the surface and eliminate surface contamination. Friction processing was performed using a vertical CNC milling machine, which allowed the sample to be shifted in the XYZ planes and the tool to be tilted. The tool used in the treatment was equipped with a pin with a threaded side surface and length of 4.5 mm. The diameter of the shoulder was 18 mm. The tool was made of X37CrMoV5–1 hot work tool steel. Frictional processing was carried out using the following parameters: linear velocity of the tool $V = 30$ mm/min, and plunge rate $R = 6$ mm/min. The rotational tool speed (N) was 250 rpm. The tool during surface layer modification was deflected by an angle of 2 degrees in relation to the axis perpendicular to the modified surface. The stand for friction modification of the AlMg10/SiC composite is shown in Fig. 1.

The sample in the initial state and the one after FSP modification were subjected to comparative microstructural investigations using light microscopy and scanning electron microscopy. EDS studies of micro-areas were also carried out. Optical microscopy studies were performed using an Olympus GX41 optical microscope, and SEM investigations using a JEOL JSM-6610LV scanning electron microscope. The chemical composition of the micro-areas was analyzed using an EDS analyzer. Hardness measurements were conducted on cross-sections of the friction stir processed zone using a Shimadzu HMV-G20 microhardness gauge with loads of 980.7 mN applied for 10 s.

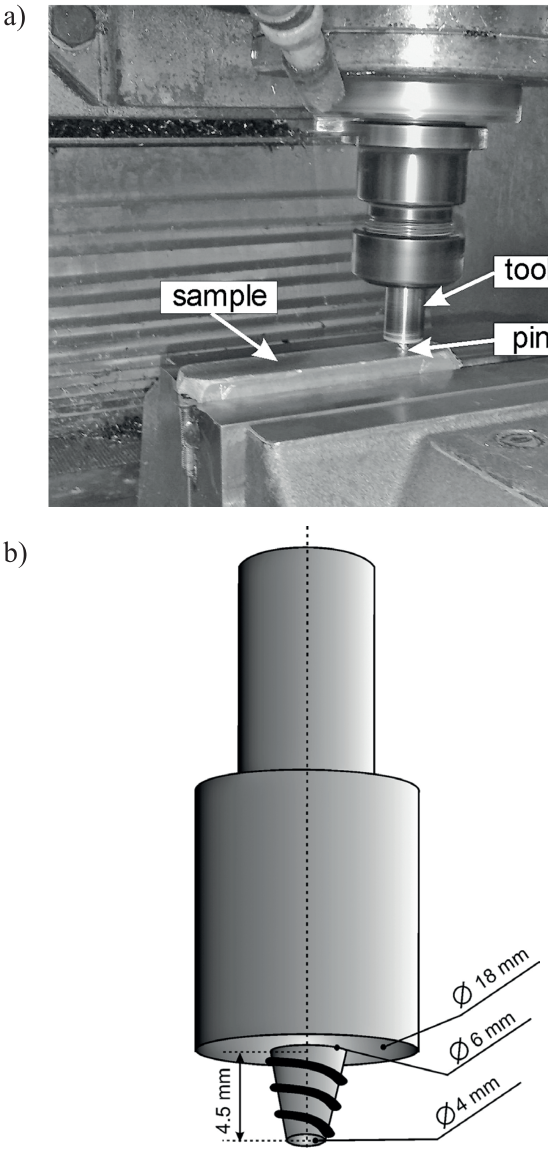

Fig. 1. Friction modification stand (a), tool (b)

3. Results

3.1. Microstructural research. The microstructure of the AlMg10/SiC composite in the as-cast condition is shown in Fig. 2a-d. Three phases were present in the microstructure of the material, namely the α solid solution, secondary β phase (Al_3Mg_2) and SiC particles. During the microstructural investigations, it was found that the SiC particles were partially or completely surrounded by the β phase. This effect can be explained by the fact that in the final phase of crystallization, the liquid enriched in magnesium crystallized as the β phase, which in combination with the phenomenon of pushing SiC particles through the moving crystallization front resulted in the formation of SiC/Al_3Mg_2 type separation surfaces [18].

Analysis of the composite microstructure revealed irregularity of the reinforcement phase distribution in the AlMg10

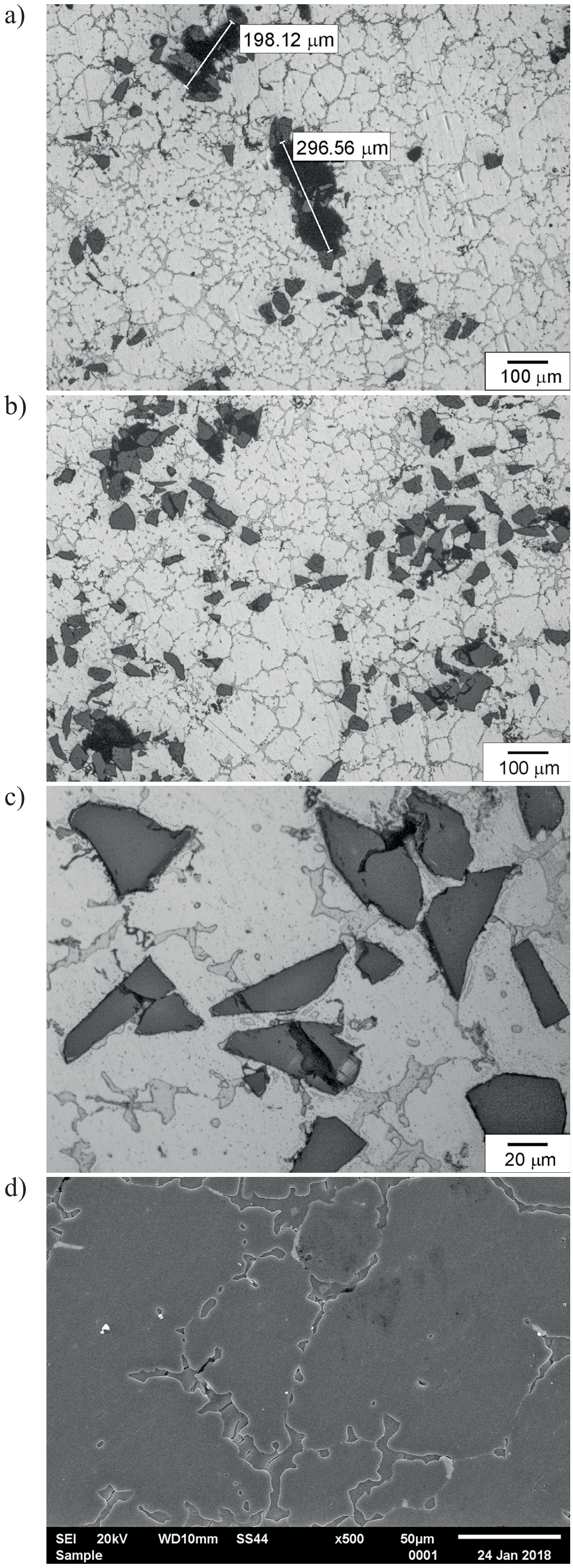

Fig. 2. Microstructure of AlMg10/SiC composite in as-cast condition, a) material discontinuities in composite, b) inhomogeneous distribution of SiC particles, c) direct contact between SiC particles, d) exemplary area without reinforcement. Optical microscopy (a–c), SEM (d). Etched

alloy matrix and the presence of locations where the SiC particles were not separated from each other by the matrix and as a result there was direct contact between them (Fig. 2a-c). In the case where the particles are not separated from each other by the matrix, they are then less strongly connected with the matrix and tend to crumble out of the matrix during exploitation of the composite. Such particles are also less involved in load transfer by the composite. In the microstructure of the composite, it was also found that material discontinuities are usually located at SiC agglomeration sites, but also at the SiC/Al_3Mg_2 phase separation interface (Fig. 2a, b). Voids and other material discontinuities had a length of up to 0.4 mm. Material discontinuities are an obvious threat to the durability of the product and its functional properties. Defects found in the casting microstructure prove both the too low cooling rates of the material during material crystallisation and insufficient mixing of the composite during its production. During the microstructural investigations, it was also found that there were numerous areas showing a negligible share of the reinforcing phase in the alloy matrix, well below the average for the material (Fig. 2d).

The next phase of the research concerned analysis of the microstructure of the material subjected to friction modification. As a result of friction treatment, a zone of microstructural changes with a width of about 5 mm and a depth of about 5.5 mm was obtained. The dimensions of this zone corresponded approximately to the dimensions of the tool pin. The area of microstructural changes in the friction modified zone is shown in Fig. 3.

Microscopic examination showed significant changes in the microstructure of the material in relation to the state before processing. First of all, there was marked fragmentation of the matrix grain, partial dissolution of β phase precipitates in the α solid solution and more even distribution of SiC particles in the aluminum alloy matrix combined with a simultaneous disappearance of SiC particle agglomerates (Fig. 4a, b). An important feature of the FSP treated material was also the high SiC particle fragmentation and shape change (Fig. 5a, b).

In the as-cast composite, particles with dimensions from 30 to 60 μm and a sharp-edged polyhedral shape prevailed, while in the material subjected to friction treatment, particles with dimensions from 20 to 40 μm and a more equiangular shape

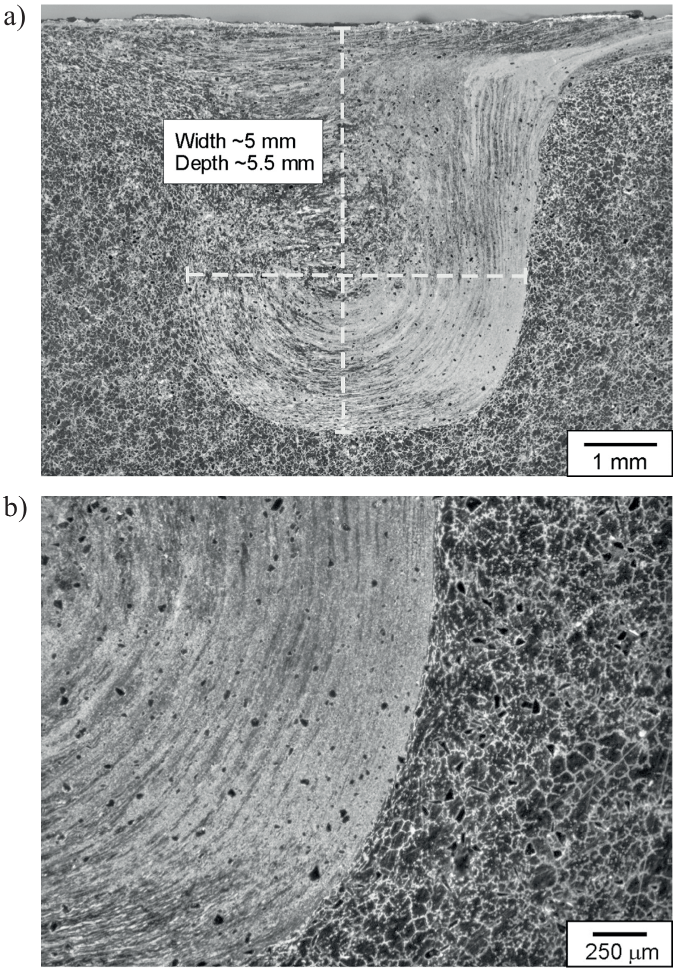

Fig. 3. Friction modified zone, a) approximate dimensions of modified zone, b) boundary zone between modified zone and core material. Optical microscopy, etched

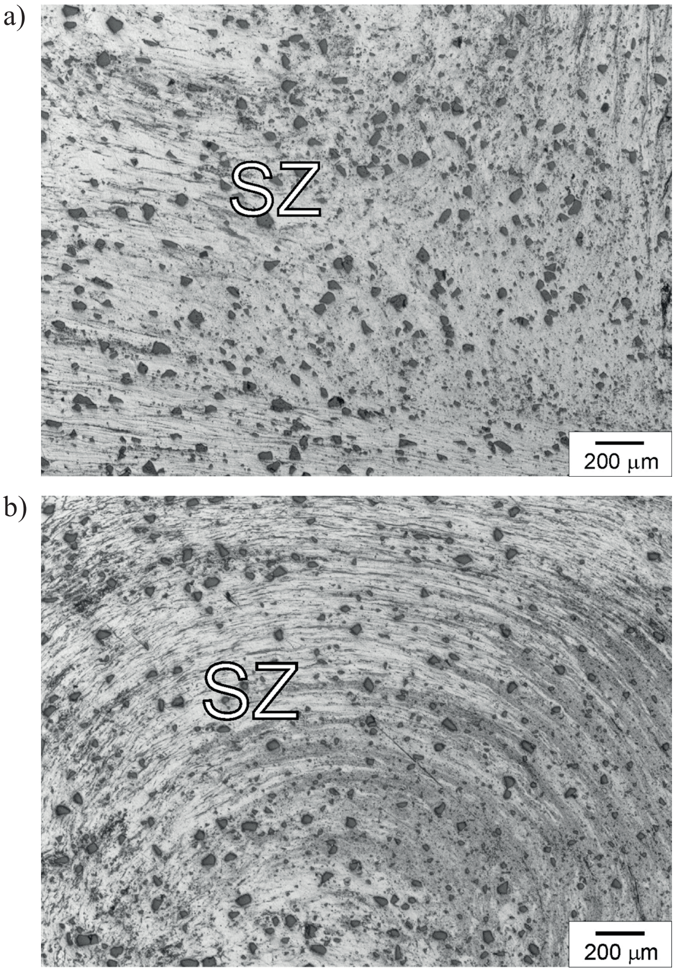

Fig. 4. Microstructure of AlMg10/SiC composite in stirring zone about 1 mm below surface (a) and about 3 mm below surface (b), optical microscopy, etched

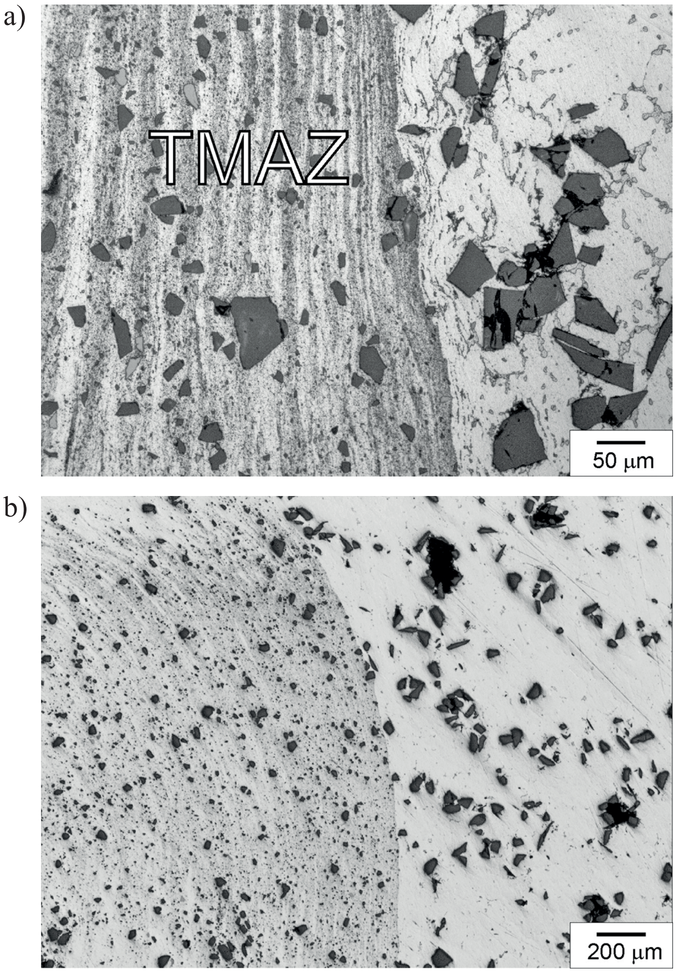

Fig. 5. Composite microstructure on border of modified zone and core material, optical microscopy, etched (a), non-etched (b)

prevailed. In the initial material, there was no presence of particles with linear dimensions below 10 μm, whereas in the material subjected to frictional treatment there were many such particles, and the smallest of the disclosed particles had a linear dimension of 1–2 μm. The largest particles with dimensions of approximately 60 μm were also present in the friction-treated material, but were considerably less numerous than in the starting material. The observations showed that the amount of particles per unit area in the case of the friction-modified material was more than twice as high as in the starting material, which should be attributed to their intense fragmentation. A total of 6 areas with an approximate area of 0.25 mm^2 each were subjected to quantitative analysis, three of these areas were located in the central part of the stirring zone, and three in the core material. In the stirring zone, the average amount of SiC particles per analyzed area was about 162, while in the core the average amount of SiC particles was 57. It was also found that as a result of the friction treatment, material defects arising during the casting process, i.e. voids and other material discontinuities, were eliminated.

In the friction-modified zone, zones characteristic for materials treated with FSP were clearly visible, i.e. a clearly dominant stirring zone (SZ) and a narrow thermo-mechanically affected zone (TMAZ) separating the stirring zone from the core material. The grains in the stirring zone were mostly equiaxial, whereas in the thermo-mechanically affected zone grains with a slightly elongated shape prevailed. The grain morphology in the stirring zone proves that there was dynamic recrystallization of the material at this place.

In the case of composite materials, achieving a coherent connection of both components of the composite is very important issue, hence this aspect was the subject of microstructural analysis. In relation to the state before the treatment, significant differences were found at the reinforcement-matrix interface. In the case of the starting material, the effective contact area between the matrix and SiC particles was clearly smaller than after the friction treatment. These differences can be seen, for example in Figs. 6a and 6b. The better joining of SiC particles with the matrix translates, among others into a lower tendency for particle chipping during work, which is worth emphasizing.

3.2. EDS investigations. EDS investigations were carried out to assess the variability of the chemical composition in the friction-modified zone as a function of the distance from the

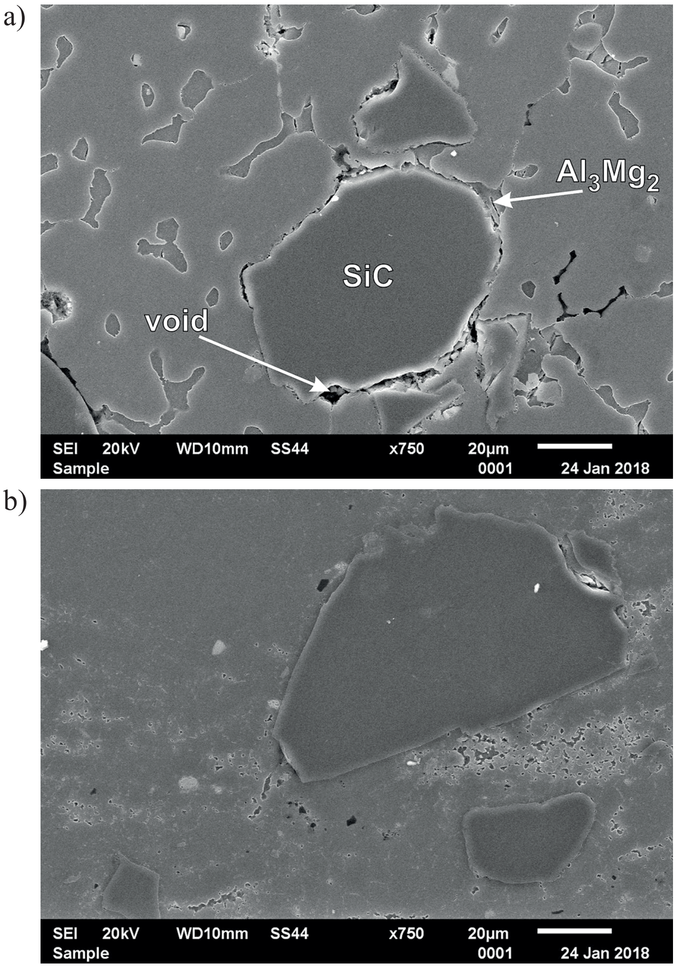

Fig. 6. Microstructure of AlMg10/SiC composite before (a) and after friction modification (b), SEM

sample surface. The EDS investigations were carried out on micro-areas located in three different places of the modified zone, i.e. in the lower part of the stirring zone, in its central part and in the near-surface zone. All the analyzed micro-areas were located centrally in the axis of the stirring zone. The EDS results are shown in Fig. 7 and summarized in Table 1. As can be seen, along with the distance from the surface of the band, the contents of Si and C decrease, which means that a higher concentration of SiC particles occurs in the upper part of the stirring zone. Another characteristic feature is the occurrence

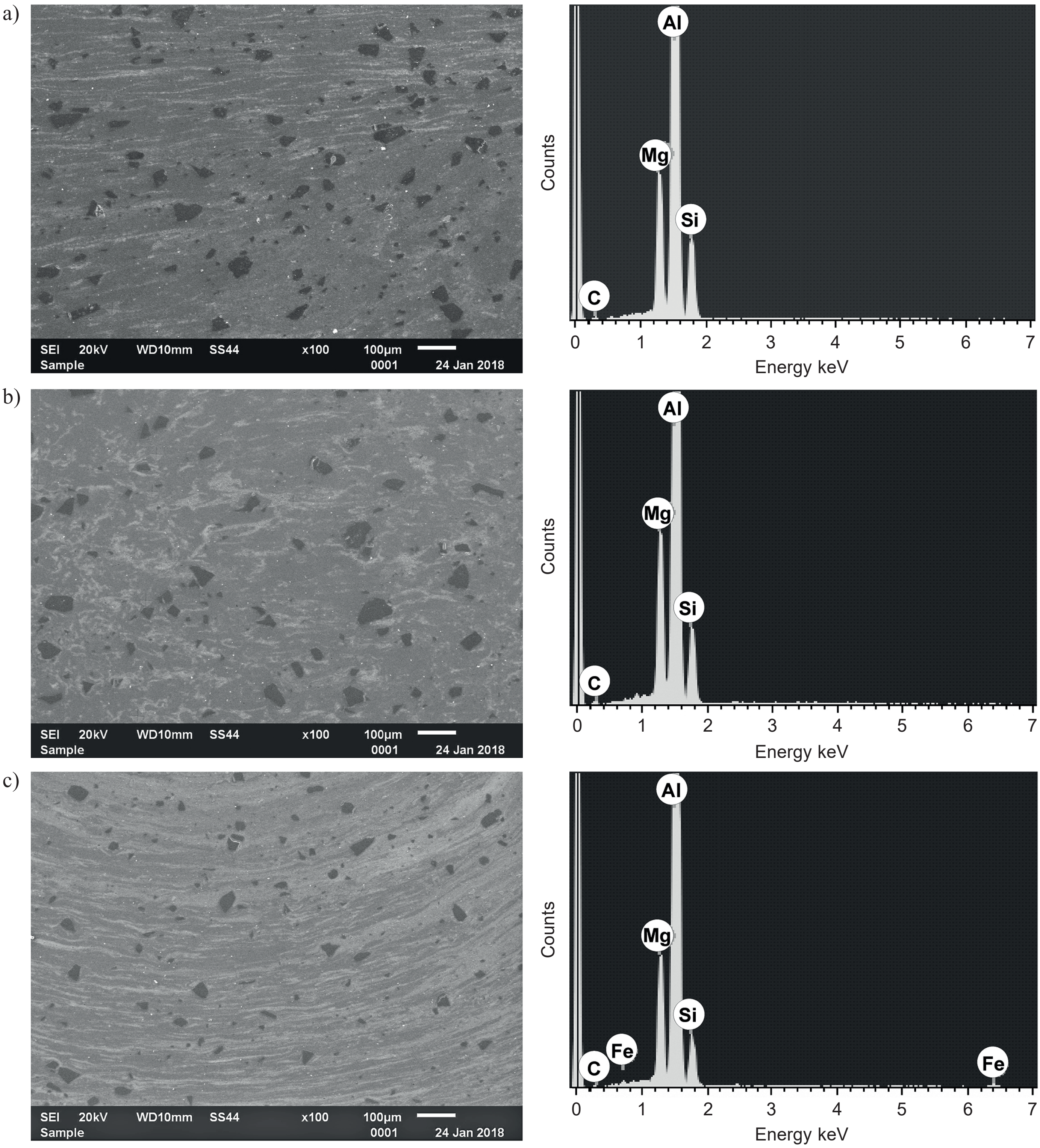

Fig. 7. Results of EDS analysis, upper part of modified zone (a), middle part of modified zone (b), bottom part of modified zone (c)

Table 1
Results of EDS analysis

Element	**Atomic and weight share**	**Localization of micro-areas**		
		Upper part	*Middle part*	*Bottom part*
Al	Weight %	70.18	72.38	74.45
	Atomic %	64.16	66.05	69.40
Mg	Weight %	9.25	9.28	9.15
	Atomic %	9.39	9.40	9.47
Si	Weight %	13.44	11.12	9.30
	Atomic %	11.80	9.75	8.33
C	Weight %	7.13	7.22	5.85
	Atomic %	14.65	14.80	12.25
Fe	Weight %	–	–	1.25
	Atomic %	–	–	0.56

of iron in the lower part of the stirring zone. The presence of iron in the friction-modified zone is a consequence of the wear of the pin surface during FSP treatment, the presence of SiC hard particles in the aluminum alloy undoubtedly intensified this process, thus contributing to contamination of modified material with the material of the pin.

In the course of EDS research, attention was also paid to the possible presence of other phases with a different chemical composition than those present in the starting material. During the production of the composite, there may be a reaction between liquid aluminum and SiC, leading to the formation of Al_4C_3 carbide at the interface. However, the EDS investigations did not reveal the presence of sites where Al and C would be in quantitative relations corresponding to the Al_4C_3 phase, neither in the starting material nor in the material subjected to friction modification.

3.3. Hardness testing. In order to evaluate the effect of changes in the composite microstructure caused by friction modification, hardness measurement was performed. An exemplary distribution of hardness as a function of distance from the surface is shown in Fig. 8a, and hardness distribution across the modified zone is shown in Fig. 8b. The tests showed a significant increase in the recorded hardness values in relation to the hardness of the starting material. The average hardness of the composite before friction modification was about 120 HV0.1, while after surface treatment the average hardness was about 160 HV0.1, therefore the hardness increased by about 35%.

The standard deviation of the starting material hardness was 9.84. In case of the modified material hardness the standard deviation was 7.98. Coefficients of variation were about 8.2% and 5% respectively for the starting material and for the modified material.

The increase in hardness is primarily a consequence of the more even distribution of SiC particles in the AlMg10 alloy matrix, as well as the strong fragmentation of the matrix grain and its homogenization.

4. Conclusions

1. As a result of friction modification homogenization and densification of the material occurs as well as a more even distribution of SiC particles in the AlMg10 alloy matrix.
2. Friction stir processing leads to a reduction in casting defects present in the as-cast composite and a consistent connection of the individual components of the composite.
3. A consequence of the advantageous changes in the microstructure of friction modified AlMg10/SiC composite is higher hardness compared with the as-cast material.
4. Friction stir processing is a promising method enabling improvement the composite microstructure and enhancement of its hardness.

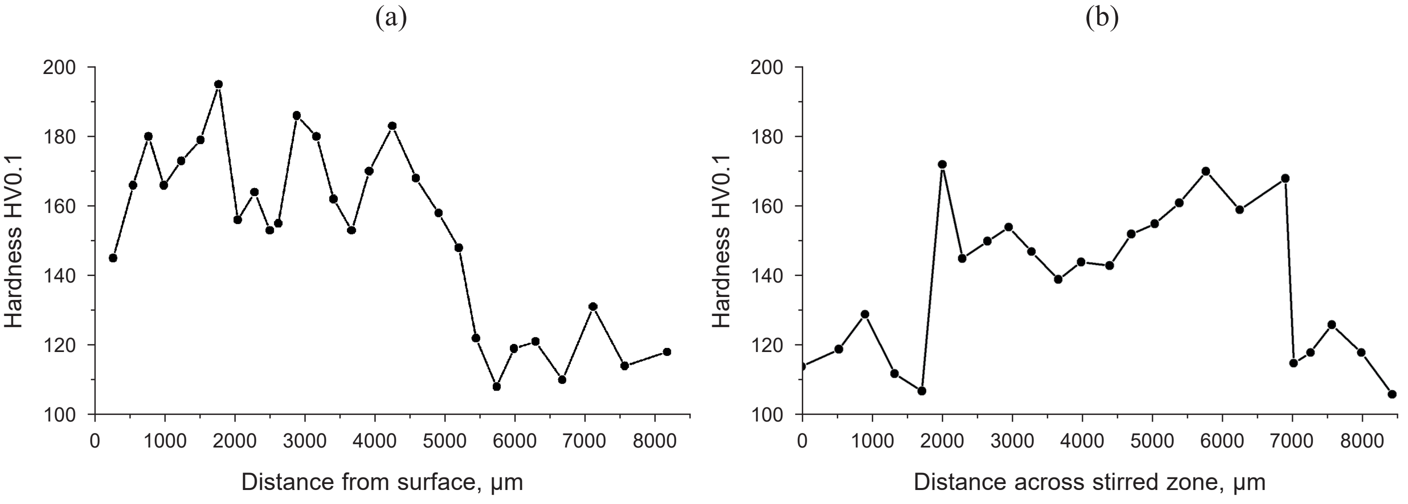
(a)
(b)

Fig. 8. Hardness measurement results, exemplary distribution of hardness as a function of distance from surface (a), hardness distribution across modified zone (b)

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