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THE EFFECT OF FRICTION STIR PROCESSING (FSP) ON THE MICROSTRUCTURE AND PROPERTIES OF AM60 MAGNESIUM ALLOY

The samples of the as-cast AM60 magnesium alloy were subjected to Friction Stir Processing (FSP). The effect of FSP on the microstructure of AM60 magnesium alloy was analyzed using optical microscopy and X-ray analysis. Besides, the investigation of selected properties, i.e. hardness and resistance to abrasion wear, were carried out. The carried out investigations showed that FSP leads to more homogeneous microstructure and significant grain refinement. The average grain size in the stirred zone (SZ) was about 6-9 μm. In the thermomechanically affected zone (TMAZ), the elongated and deformed grains distributed along flow line were observed. The structural changes caused by FSP lead to an increase in microhardness and wear resistance of AM60 alloy in comparison to their non-treated equivalents. Preliminary results show that friction stir processing is a promising and an effective grain refinement technique.

Keywords: Friction stir processing; Magnesium alloy

1. Introduction

Friction stir processing (FSP) is a promising grain refinement technique. This method comes from the FSW (Friction Stir Welding) technology developed in 1991 by Wayne Thomas from the Welding Institute (TWI Ltd.) in Cambridge. The FSW method consists in friction welding with the stirring of the material, and it is used for joining materials in a solid state [1]. In both methods the same scheme is used: the heat generated as the result of the friction between the working tool and the material surface heats up and plasticizes the material. The tool is normally made of high-speed steel, the tool steel for hot work, or polycrystalline cubic boron nitride, and also wolfram alloys. After the tool shank is put in rotation it sinks slowly into the joint area (for the FSW technology) or into the material being modified (for the FSP technology). In the first phase, only the front surface of the shank has contact with the material being modified, and then the side surface of the shank and the surface of the retaining collar. During the reaction between the tool and the material a series of complicated thermodynamic processes occur, including heating up and cooling down of the material with different rates, plastic strain, as well as physical flow of the processed material around the tool [2]. The heat plasticizes the friction stir processed material below the melting point. The thermal effect arising in the material and accompanying structural changes, including the shape and the dimensions of the zone being modified, depend on the treatment parameters (e.g. rotational speed of the tool), but also on the dimensions and construction of the working tool [3-6]. The heat generated by the friction between the shank and the material surface constitutes 20% of the heat generated during the process, the rest is the heat given off due to the friction between the material surface and the front surface of the retaining collar [7]. At this point, it is worth mentioning that during the treatment of the surface with the FSP method or the joining of materials in the FSW method the melting temperature of the materials being modified or joined is not exceeded (the temperatures occurring in the FSP and/or FSW process constitute 70-90% of the melting temperature of the material being modified), the process is of single-stage type, and because the thermal energy source is the friction process, the FSW and FSP technologies represent entirely ecological solutions [8]. The attempts to modify the structure of materials with the FSP method are carried out on aluminum alloys [9, 10], magnesium alloys [4, 9, 11-13], steels [14], and also composites with a metallic matrix [15]. The FSP method may be used also for the creation of composite structures in the surface layer of the material by the introduction of a strange phase, e.g. carbon nanotubes, SiC, Al_2O_3 and SiO_2 particles, and others [5, 6, 16-20]. An interesting research issue of the high application potential is the modification of the structure of magnesium alloys. Magnesium alloys are modern construction materials which due to advantageous mechanical properties and the lowest density when comparing with all other known technical alloys more and more effectively compete with aluminum alloys, steels, and even plastics. The decisive aspect for their application potential is low density (about 1.8 g/cm^3), a one-third lower than the density of aluminum alloys, and almost 80% lower than steel density. This places magnesium alloys in the group of the most attractive and prospective metallic materials [21-23]. Magnesium alloys are widely used in the automotive industry, aviation, electronics and electrical engineering. The changes in the structure of magnesium alloys occurring during the treatment with the FSP method, and in particular the refinement and homogenization, may contribute to the improvement of the plasticity and deformability of

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those alloys, and by this to the increase of their application attractiveness [4, 13]. Magnesium alloys are characterized by very limited ductility accompanied by brittle-like behavior at room temperature due to its intrinsic hexagonal close-packed (hcp) crystal structure and a limited number of available slip systems [11, 12].

Taking into account the growing interest in magnesium alloys, as well as some application limitations for those materials, in this study an attempt was made to modify the AM60 alloy structure with the FSP technique, and to assess the structural changes and the changes of selected properties of AM60 magnesium alloy generated by the treatment process.

2. Material and experimental procedures

The material used in this study was AM60 magnesium alloy. The chemical composition of the alloy is presented in Table 1. Cuboidal samples sized 70 mm x 30 mm x 10 mm machined from cast AM60 billets were subjected to FSP (processed in air). Prior to the FSP, the samples were polished and chemically cleaned with alcohol to eliminate surface contamination, and dried in air. The friction stir processing was done on a vertical CNC milling machine. All samples were processed using a tool made of H13 tool steel, consisting of a cylindrical 10 mm diameter shoulder. A pin diameter was 3 mm, and a pin length was 0.6 mm. The surface of the stir tool was smooth. The tool was linearly traversed at V=16 mm/s. The rate at which the tool was immersed perpendicularly to the surface (R) was 0.1 mm for all samples. Then, after having achieved full immersion of the tool, the main movement of the tool was performed with the welding rate V. The rotational speeds (N) were within the range of 3,500 to 4,500 rpm. Within the range of the adopted process parameters a heat plasticization of the magnesium alloy was observed, and this was the precondition for carrying out the process of modification with the FSP method. The specification of the treatment parameters is presented in Table 2. The depth of structural changes was about 600–650 μm, and this approximately corresponded to the length of the working tool shank. The FSP pattern and the examples of the macroscopic effects of the treatment obtained at 16 mm/s traverse speed, using 4,000 revolutions per minute is shown in Fig. 1.

TABLE 1

Chemical composition of the AM60 alloy, in accordance with ASTM B93-94

Content of element (wt. %)				
Al	Zn	Mn	Si	Mg
5,6-6,4	0,20 max	0,26-0,50	0,05 max	Bal.

TABLE 2

Treatment parameters

No.	N [rpm]	V [mm/s]	R [mm/s]
1.	3500	16	0.1
2.	4000	16	0.1
3.	4500	16	0.1

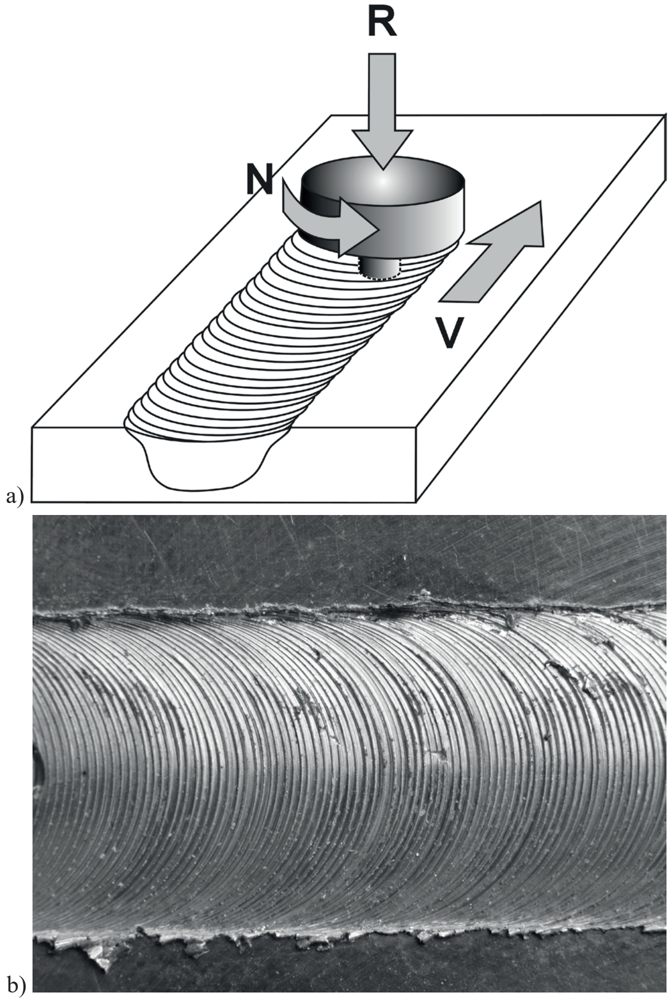

Fig. 1. Friction stir processing; a) scheme, b) sample- exemplary macroscopic effects

The structural research was carried out with the use of the Olympus GX47 light microscope. Transverse sections of the modified surfaces were mounted, polished and etched using nital solution. The phase composition of the samples was determined by X-ray diffraction (XRD) with a Seifert XRD-3003 diffractometer (Rich. Seifert & Co, Hamburg) using filtered CoKα radiation (λ=0.17902nm) operated at 30 kV and 40 mA. The used exposure time was 186 min and the analysed spectra taken from 2θ range of 20-90°. The microhardness measurements were conducted on the cross-sections of the of the friction stir processed zone using the Vickers Microindentor (Future-Tech FM-7) with the loads of 50 g applied respectively for 6 s. The tribological properties were measured using a pin-on-disc type laboratory tribometer under unlubricated sliding contact against the steel ring (HRC 58-63). The magnesium alloy samples had a form of a shank with the diameter of 4 mm. Prior to the test, the surface of the samples was polished on emery paper up to 600 grade. The tests were run at the constant normal load of 20 N and sliding velocity ~0.15 m/s (the rotational speed was 96 rpm). The total wear distance was 1,500 m. The friction force and linear material loss (displacement) were recorded automatically against time by the tester software. The friction coefficient was calculated from the ratio of the friction force to the normal load.

3. Results and discussion

3.1. Microstructure characterization

The structural investigations of the as-received AM60 alloy revealed the presence of large α-Mg grains having an average grain size of 140 μm and a network-like eutectic β-$Mg_{17}Al_{12}$ phase along the grain boundaries. The presence of the Al_8Mn_5 phase was also found in the course of the microstructural research and was confirmed by EDS investigations (not presented in this study). Fig. 2 shows the microstructure of the as-received AM60 magnesium alloy.

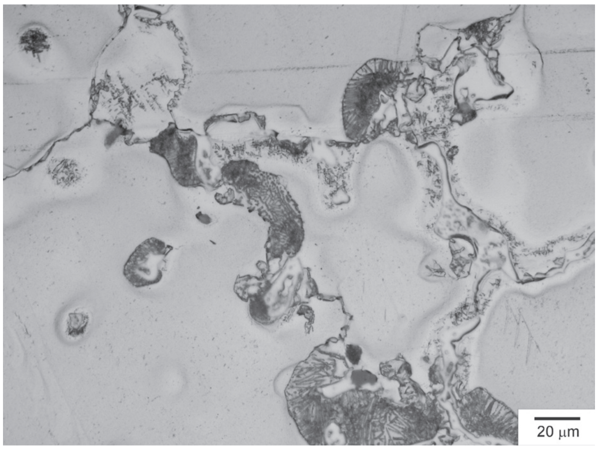

Fig. 2. The microstructure of the as-received AM60 alloy, etched micro-section, light microscope

The microscopic observations of the samples after the friction modification revealed significant changes in the structure of the analyzed material. In the magnesium alloy structure, the presence of a clear stirred zone (SZ), thermomechanically affected zone (TMAZ) with a characteristic – for that zone – structure of strained grains and poorly noticeable zone of the heat impact – HAZ (heat affected zone) adjoining the base material (BM) was found. Optical micrographs of the friction stir processed zone at rotational speeds of 3,500 rpm are shown in Fig. 3. The presence of equiaxed and fine grains of 2-10 μm in the stirred zones was observed (Fig. 3c). The grain refinement microstructure of the stirred zone indicates the occurrence of dynamic recrystallization due to concurrent intense plastic deformation and frictional heat. This also means that the temperature during the modification was higher than the temperature of dynamic recrystallization of the magnesium alloy. The analysis of the grain size versus the applied treatment parameters showed that the largest grain occurs in the samples subjected to modification with the use of the tool rotational speed of 4,500 rpm, whereas the smallest grain was found in the samples modified with the use of the tool rotational speed of 3,500 rpm. The average grain sizes of the friction stir processed zone were about 6 μm and 9 μm in the 3,500 rpm and 4,500 rpm friction stir processing trials, respectively. The found regularities should be linked with the temperature the material reached during treatment. This temperature increases as the rotational speed of the tool increases, and this results in a lower refinement of the structure. These results are in agreement with the results

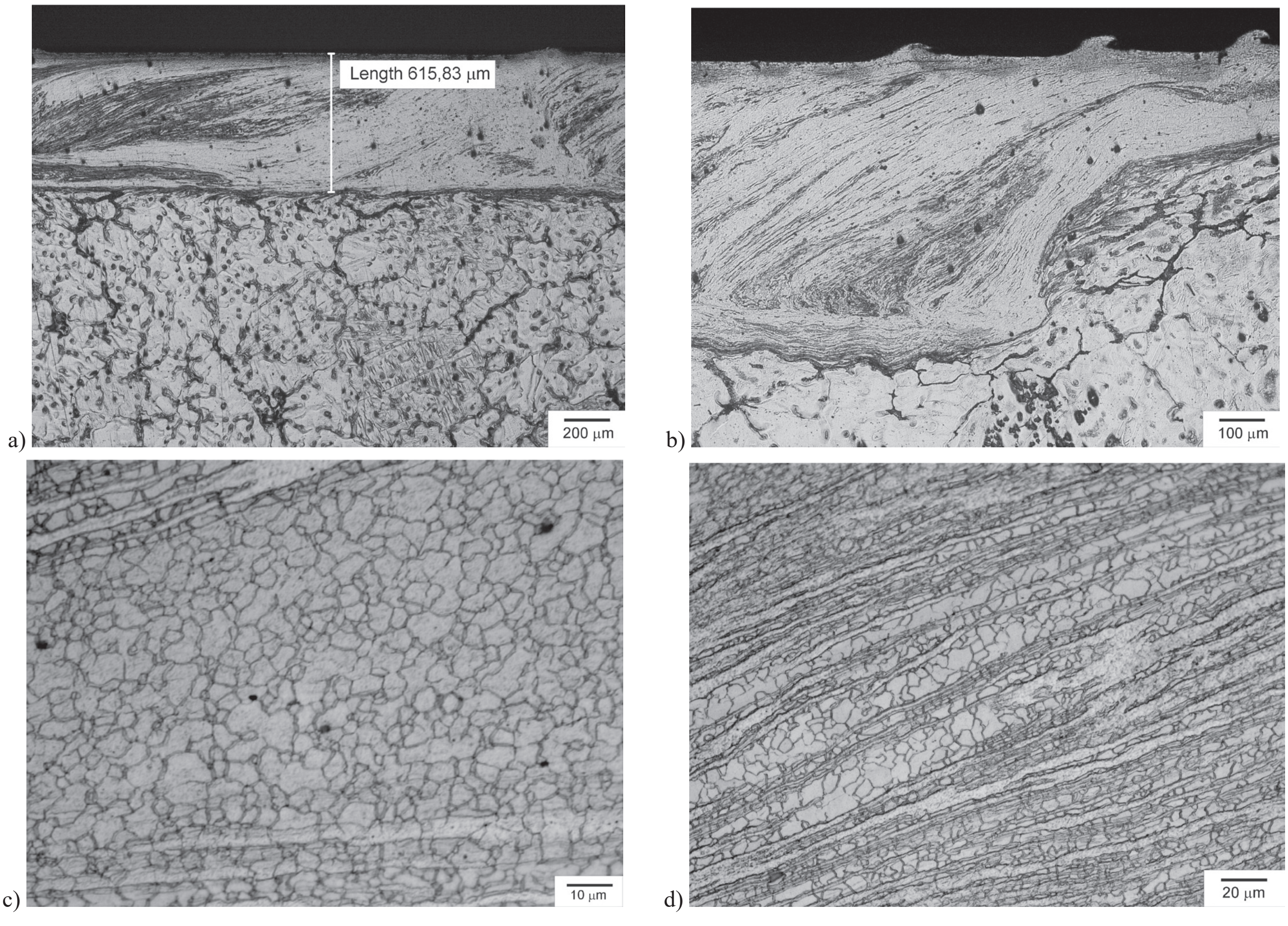

Fig. 3. The microstructure of the AM60 alloy after modification, etched micro-section, light microscope

reported by M. Barmouz et al. [20] and Y.C. Chen et al. [14]. They found, that an increase in traverse speed and a decrease in rotational speed cause a reduction in the grain size of the stir zone. In contrast to the as-cast conditions the microstructure of the friction stir processed alloy was significantly more homogenous. More homogeneous structure was observed in all friction stir processed samples. The significant β-phase dissolution was observed too. In the TMAZ, the highly elongated and deformed grains distributed along flow line were observed, and it appears that the dynamic recrystallization did not occur due to insufficient deformation strain and thermal exposure (Fig. 3d).

3.2. X-ray structural investigations

The X-ray analysis of the phase composition carried out on the material in both its initial state and after friction stir processing indicated a lack of relevant changes in the phase compositions of the investigated materials, which acknowledges the phase stability in the conditions accompanying the surface treatment (Fig. 4). The investigation carried out revealed the presence of solid solution α-Mg and inter-metallic phase β-$Mg_{17}Al_{12}$. No diffraction reflections from the Al_8Mn5 phase were observed which proves that the content of this phase in the analyzed samples was very small, below the detectability threshold of the X-ray method. However, the presence of reflections originated from magnesium oxide was found. The occurrence of oxide phases is the consequence of a strong affinity of magnesium for oxygen and the lack of gas shield during the treatment. Besides, the investigation of the stirred material revealed an above-average intensification of the reflections from certain plane lattices in comparison with the initial condition and the JCPDS 35-821 standard. The above average intensification of diffraction reflections (in particular from the plane (0002)) may be caused by the texture which was formed in the material. The specificity of the FSP process causes that the material is formed in the conditions of the increased temperature effects and the pressure of the tool by which structural changes are generated; they reflect the conditions that the material was formed in, and may lead to the occurrence of preferential material orientation.

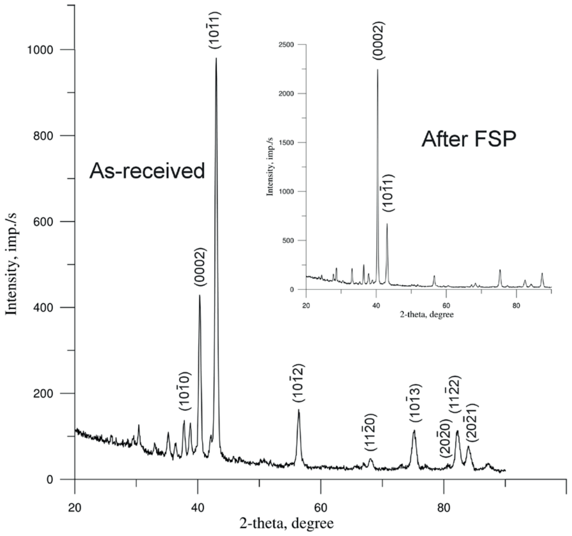

Fig. 4. Results of the X-ray analysis of the AM60 magnesium alloy

3.3. The hardness measurements and wear resistance investigations

The effect of friction stir processing on the hardness distribution versus the distance to the surface is shown in Fig. 5a. The average hardness of the as-received and friction stir processed zone were ~58±5 HV0.05 and 77±10 HV0.05, respectively. By analyzing the obtained results, it can be stated that the hardness of the friction stir processed samples increased by 20-40% for the AM60 magnesium alloy. The increase of the hardness in the modified layer is due to the microstructural refinement and homogenization of the material. The highest microhardness was observed when a tool rotational speed of 3,500 rpm was used, the smallest one when a tool rotational speed was 4,500 rpm. The effect of rotational speed on the hardness is shown in Fig. 5b. On the basis of the obtained results a conclusion can be drawn that increasing the rotational speed leads to a decrease of the hardness. This fact should be linked with the differences in the grain sizes caused by various rotational speeds of the tool. The analysis of the investigation results led to noticing one more regularity, namely the highest hardness was obtained at some distance from the surface, different for individual samples. This fact should be linked with the temperature of the material during the friction modification. The temperature is highest at the surface, which leads to an increase in grain growth. A decrease in hardness in the heat affected zone was observed too. One can assume that this is due to the lack of mechanical deformation in this zone.

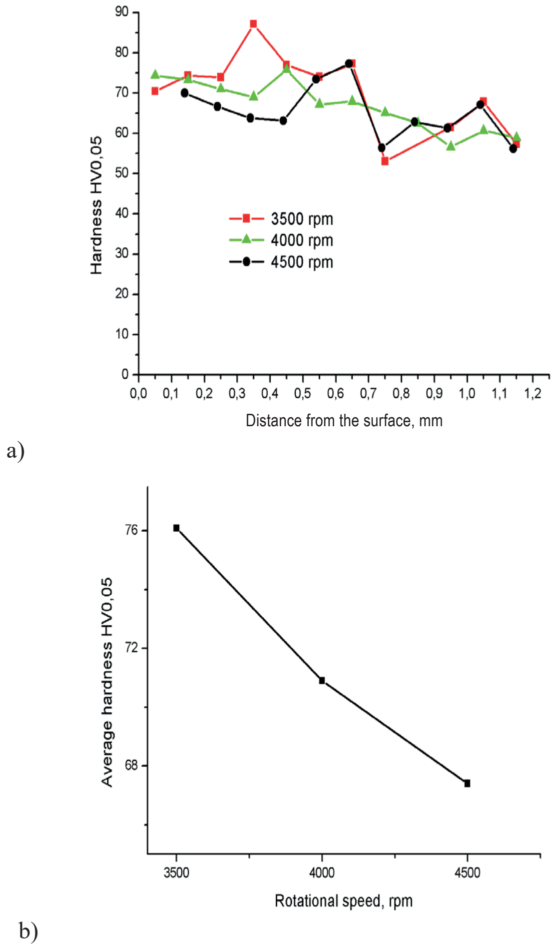

Fig. 5. Hardness measurement results

Equally positive changes in the material properties were also observed during tribological investigations. In Fig. 6 and in Table 3, a linear loss of the material recorded during tribological tests versus time is shown. All surfaces after FSP treatment showed lower friction coefficients with the load of 20 N in comparison with the as-received material, but those differences were not significant. The average value of the friction coefficient for the samples after the friction stir processing was about 0.8, and for the as-received material it was about 0.9.

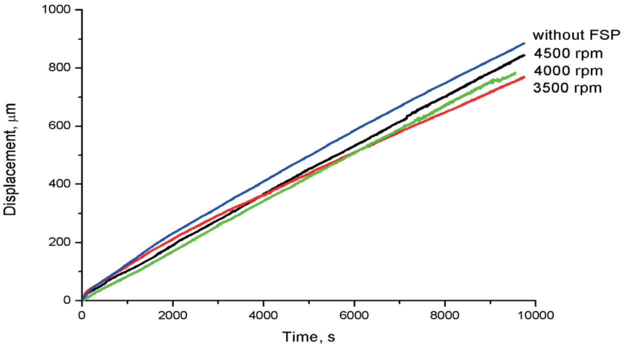

Fig. 6. The material loss (displacement) for surface layer of AM60 magnesium alloy during pin on disk test at load 20 N

TABLE 3

Material loss after pin on disk test

No.	N [rpm]	Normal load [N]	Material loss (displacement) after 1,500 m [mm]
As-received	-	20	893
No. 1	3500		779
No. 2	4000		784
No. 3	4500		844

4. Summary

The research showed that the treatment of AM60 magnesium alloy with the FSP method leads to the formation of characteristic zones in the material structure, i.e. the stirring zone, the thermomechanically affected zone, and the heat affected zone. In the stirring zone, very fine and equiaxed grains dominate; their presence is the consequence of recrystallization triggered by the plastic strain and frictional heat that accompany the FSP. The average grain sizes in the stir zone were about 6 µm and 9 µm in the 3,500 rpm and 4,500 rpm friction stir processing trials, respectively. These differences result from the maximum temperatures to which the material heats up during the treatment process. This temperature increases with the rotational speed of the tool, which finally leads to the occurrence of a lower refinement of material structure. In the TMAZ, highly elongated grains distributed along flow line dominate. It appears that the dynamic recrystallization did not occur in this zone. The consequence of the changes in the structure of the material subjected to the FSP is the increase of the hardness and the resistance to abrasive wear of magnesium alloy. The analysis of the hardness results combined with FSP parameters showed that increasing the rotational speed leads to the decrease of hardness. This fact should be linked with the differences in the grain size caused by the different rotational speeds of the tool, and the occurrence of different temperatures in the material during the friction modification. The temperature is highest at the surface, which leads to an increase in grain growth. The highest microhardness was observed at the tool rotational speed of 3,500 rpm, the smallest one when the tool rotational speed was 4,500 rpm. Besides, the material subjected to the friction modification is characterized by a lower friction coefficient and it shows a lower linear loss of the material in the tribological test. The presence of oxide phases found during X-ray tests is the consequence of a strong affinity of magnesium for oxygen and the lack of gas shield during the treatment process. To avoid the phases referred to above, the oxides should be removed from the metal surfaces directly before the FSP modification process, and in some cases a gas shield should be applied using an inert gas. To sum up, the friction stir processing is an effective method for refining and homogenizing microstructures. The FSP method may constitute an alternative and a competitive solution compared to other methods of the magnesium alloy structure modification.

REFERENCES

[1] W.M. Thomas, E.D. Nicholas, J.C. Needham, M.G. Church, P. Templesmith, C.J. Dawes, Friction stir butt welding, The Welding Institute, TWI, International Patent Application No. PCT/GB92/02203, GB Patent Application No. 9125978.8 (1991) and U.S. Patent No. 5460317 (1995).

[2] I. Kalemba, C. Hamilton, S. Dymek, Natural aging in friction stir welded 7136-T76 aluminium alloy, Materials and Design **60**, 295-301, (2014).

[3] P. Lacki, Z. Kucharczyk, R.E. Sliwa, T. Gałaczynski, Effect of tool shape on temperature field in friction stir spot welding, Archives of Metallurgy and Materials **58/2**, 595-599, (2013).

[4] G. Venkateswarlu, D. Devaraju, M.J. Davidson, B. Kotiveerachari, G.R.N. Tagore, Effect of overlapping ratio on mechanical properties and formability of friction stir processed Mg AZ31B alloy, Materials and Design **45**, 480-486, (2013).

[5] M. Bahrami, M. Kazem Besharati Givi, K. Dehghani, N. Parvin, On the role of pin geometry in microstructure and mechanical properties of AA7075/SiC nano-composite fabricated by friction stir welding technique, Materials and Design **53**, 519-527 (2014).

[6] P. Vijayavel, V. Balasubramanian, S. Sundaram, Effect of shoulder diameter to pin diameter (D/d) ratio on tensile strength and ductility of friction stir processes LM25AA-5% SiCp metal matrix composites, Materials and Design **57**, 1-9 (2014).

[7] K. Kudła, K. Wojsyk, Z. Kucharczyk, Własności zakładkowych złączy spajanych metodą zgrzewania tarciowego z przemieszaniem (FSW - Friction Stir Welding), Obróbka Plastyczna Metali **24/3**, 179-191 (2013).

[8] P. Lacki, Z. Kucharczyk, R.E. Śliwa, T. Gałaczyński, Wpływ wybranych parametrów procesu zgrzewania tarciowego z przemieszaniem na pole temperatury, Rudy Metale **R57/8**,

524-532 (2012).

[9] Z.Y. Ma, A.L. Pilchak, M.C. Juhas, J.C. Williams, Microstructural refinement and property enhancement of cast light alloys via friction stir processing, Scripta Materialia **58**, 361-366 (2008).

[10] M.L. Santella, T. Engstrom, D. Storjohann, T.Y. Pan, Effects of friction stir processing on mechanical properties of the cast aluminum alloys A319 and A356, Scripta Materialia **53**, 201-206 (2005).

[11] B.M. Darras. M.K. Khraisheh, F.K. Abu-Farha, M.A. Omar, Friction stir processing of commercial AZ31 magnesium alloy, Journal of Materials Processing Technology **191**, 77-81 (2007).

[12] F. Chai, D. Zhang, Y. Li, W. Zhang, High strain rate superplasticity of a fine-grained AZ91 magnesium alloy prepared by submerged friction stir processing, Materials Science & Engineering, **A568**, 40-48 (2013).

[13] B. Darras, E. Kishta, Submerged friction stir processing of AZ31 magnesium alloy, Materials and Design **47**, 133-137 (2013).

[14] Y.C. Chen, K. Nakata, Evaluation of microstructure and mechanical properties in friction stir processed SKD61 tool steel, Materials Characterization **50**, 1471-1475 (2009).

[15] H. Nami, H. Adgi, M. Sharifitabar, H. Shamabadi, Microstructure and mechanical properties of friction stir welded Al/Mg2Si metal matrix cast composite, Materials and Design **32**, 976-983 (2011).

[16] E.R.I. Mahmoud, M. Takahashi, T. Shibayanagi, K. Ikeuchi, Wear Characteristics of surface-hybrid-MMCs layer fabricated on aluminium plate by friction stir processing, Wear **268**, 1111-1121 (2010).

[17] Y. Morisada, H. Fujii, T. Nagaoka, M. Fukusumi, Effect of friction stir processing with SiC particles on microstructure and hardness of AZ31, Materials Science and Engineering A **433**, 50-54 (2006).

[18] C.J. Lee, J.C. Huang, P.J. Hsieh, Mg based nano-composities fabricated by friction stir processing, Scripta Materialia **54**, 1415-1420 (2006).

[19] Y. Huang, T. Wang, W. Guo, L. Wan, S. Lv, Microstructure and surface mechanical property of AZ31 Mg/SiCp surface composite fabricated by Direct Friction Stir processing, Materials and Design **59**, 274-278 (2014).

[20] M. Barmouz, M. Kazem Besharati Givi, J. Seyfi, On the role of processing parameters in production Cu/SiC metal matrix composities via friction stir processing: Investigating microstructure, microhardness, wear and tensile behavior, Materials Characterization **62**, 108-117 (2011).

[21] A. Munitz, C. Cotler, A. Stern, G. Kohn, Mechanical properties and microstructure of gas tungsten arc welded magnesium AZ91D plates, Materials Science and Engineering A302, 68-73 (2001).

[22] K.N. Braszczyńska-Malik, Odlewnicze tworzywa magnezowe. Postępy Teorii i Praktyki Odlewniczej, Monografia pod redakcją Jana Szajnara, Katowice-Gliwice 2009.

[23] L.A. Dobrzański, T. Tański, L. Čižek, Z. Brytan, Structure and properties of magnesium cast alloys, Journal of Materials Processing Technology **192-193**, 567-574 (2007).