Microstructural evaluation of Re particle reinforced composite on aluminium surface by friction stir processing

Marek St. WĘGLOWSKI
He received his Ph.D. degree in AGH in Krakow in 2010. He is currently working at the Institute of Welding University of Science and Technology in Gliwice (Poland) as Associate Professor. Author of 148 scientific publications, 6 monographs and 82 presentation (and posters). Area of Interest – surface modification technology, weldability of high strength steel, electron beam technologies.

Adriana WRONA
Doctor of Philosophy in Physics, Since 2006 – Institute of Non-Ferrous Metals in Gliwice in a position of adiunkt, including: since 2013 – Head of Powder and Composite Materials Department, from 2007 to 2012 - head of Laboratory of Metallic Powder and Composite Materials in the Department of Materials Engineering and Powder Metallurgy. She is author or coauthor of 15 inventions applied for patent protection to the Patent Office of the Republic of Poland (UPRP) and 57 scientific publications.

Ilona NEJMAN
is a PhD at the Faculty of Non-Ferrous Metals at the in Krakow. In the AGH University of Science and Technology department related from the beginning of her academic path. She is a co-author of scientific articles on Polish and international scale. Research leads in the field of surface engineering.

Maria RICHERT
is a Prof. D.Sc. Ph.D at AGH University of Science and Technology (Poland , Kraków). She is author and co-author of 168 articles, co-author of two chapters in books in English language and author of scientific book in Polish. She presented of 50 lectures at national and international conferences, and participated in 70 conferences. Prof. Maria Richert participated in 68 different scientific projects, works order by industry as manager or contractor.

Marek Stanisław WĘGLOWSKI ▪ Department of the Testing of Materials Weldability and Welded Construction, Institute of Welding ▪ marek.weglowski@is.gliwice.pl
Adriana WRONA ▪ The Department of Material Science and Powder Metallurgy, Institute of Non Ferrous Metals ▪ adrianaw@imn.gliwice.pl
Ilona NEJMAN ▪ Department of Materials Science and Non-Ferrous Metals Engineering, Faculty of Non Ferrous Metals, AGH University of Science and Technology ▪ Inejman@agh.edu.pl
Maria RICHERT ▪ Faculty of Management, AGH University of Science and Technology ▪ mricher@agh.edu.pl

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Abstract
The paper presents the results of friction stir processing (FSP) process of cast aluminium alloy AlSi9Mg. The studies included a modification of the microstructure in the grain refinement, elimination of voids and the production of composite material AlSi9Mg + Re. Rhenium is famous for its refractory character, high density as well as high hardness. It is well known as alloying element for improvement of strength, plasticity, weldability, reduction of the degree of recrystallization embrittlement mainly in W, Mo and Ni based alloys. Re is used in superalloys as a solid-solution strengthening alloying element and increasing corrosion resistance at high temperatures. Rhenium containing alloys have high electrical resistivity and refractory character. The modification was carried out single-and multiple runs. The results show significant fragmentation in the mixing microstructure, and a metallic continuity. Fragmentation in the mixing microstructure was confirmed by SEM research. The study revealed that mechanical alloying technology FSP allows to increase the hardness of the material. The ability to produce graded materials AlSi9Mg alloy matrix by frictional treatment is also indicated.
Keywords: Friction Stir Processing, rhenium, composite
Kulcsszavak: dörzshegesztési eljárás, rénium, kompozit

1. Introduction

The friction stir processing (FSP) is a solid state process, and therefore the problem related to the liquid phase processing can be eliminated [1]. Friction stir processing was developed based on the concept of friction stir welding (FSW). Conventional FSP is performed with a tool consisting of a shoulder that touches the surface of the modified material, and a smaller pin, which penetrates into the material thickness. The shoulder essentially prevents the escape of softened material as the tool is rotated and forced along the designed path. The pin, commonly threaded, pushes the surrounding material downward, assisting in the retention of material within the modified zone. The downward force applied to the tool to maintain the correct plunge depth also results in a forcible contact between the shoulder and the workpiece surface. The relative motion from the tool rotation results in significant heat generation from friction at the shoulder interface. The pin generates heat by both friction and plastic deformation. A detailed description of the FSP process was described in the previous papers [2].

In addition, the large processing strain results in microstructural refinement and homogenisation. This has led to several applications such as grain refinement for superplasticity, modification of casting microstructure as well as composite materials fabrication [3-6]. The FSP in one or multi-run technique can be carry out [7].

To date, a few authors presented interesting results on friction stir processing with filler material. *Gandra* et al. [8] reported the study of producing the functionally graded metal matrix composite (MMC) reinforced by SiC ceramic particles with median size of 118.8, 37.4 and 12.3 µm. AA5083 aluminium alloy plates in the H111 and partially annealed conditions were processed. Several strategies for reinforcement were investigated and its influence on the particle distribution and homogeneity was studied. A square shaped groove packed with reinforcement particles of SiC was studied and it was seen that it was more effective when the groove was placed under the probe. *Mahmoud* et al. [9] revealed that the SiC particles were distributed more homogeneously in the nugget zone, of an A1050-H24 aluminium plate, by using square probe tool than other shapes. *Kurtyka* et al. [10] explained the influence of the plastic deformation generated in friction stir processing on the changing concentration and distribution of SiC reinforcement particles in the cast composite A339/SiC/p, as well as determined its mechanical properties. *Sathiskumar* et al. [11] reported that in Cu/B_4C surface composite, the SZ area with B4C particles was reduced as compared to the SZ area in FSP of copper without particles. The number of the major achievements in the field of FSP technology with additional particles is well presented in the review paper of *Sharma* [12].

Taking advantages of FSP to reinforcement and refinement the grain of alloys has recently attracted attention of researchers [12]. However, no work has been yet reported on mechanical alloying by FSP of cast aluminium alloy with rhenium particle [13].

Rhenium, is famous for its refractory character (melting point 3186°C), high density (21.02 g/cm^3), high hardness (7 in Mohs scale, up to 7850 MPa - Vickers) thermal Conductivity (71.2 W/m·K at 20°C). It is well known as alloying element for improvement of strength, plasticity, weldability, reduction of the degree of recrystallization embrittlement mainly in W, Mo and Ni based alloys. Re is used in superalloys as a solid-solution strengthening alloying element and increasing corrosion resistance at high temperatures. Rhenium containing alloys have high electrical resistivity and refractory character. Due to its properties rhenium is often used in alloys for turbines in aircraft engines or vehicle shields, heating elements, thermocouples, vacuum electrodes and in catalysts (Pt-Re) for high-octane unleaded fuels [14-15]. The second wide range of application is surface engineering application. The previous experiment revealed that the Re can improve hardness as well as wear resistance characteristic of material surface [16-21].

In the present paper, the microstructural evaluation of cast aluminium alloy AlSi9Mg modified by friction stir processing with Re particles was presented. The microstructural analysis by means of light microscopy, scanning electron microscopy, chemical microanalysis by energy dispersive spectroscopy (EDS) analysis were carried out.

2. Materials and experimental procedure

Commercial available AlSi9Mg cast plates 6 mm in thickness for friction stir processing modification and the Rhenium powder (99,8% Re, KGHM Polska Miedź) as additional particles were used. The FSP process was performed at the following parameters: rotational speed ω=560 rpm, travelling speed v=560 mm/min, tilt angle α=1.5° at the Instytut Spawalnictwa (Institute of Welding) using a welding machine built on the base of a conventional, vertical milling machine FYF 32JU2. The FSP Triflute tool (*Fig. 1*) was made of HS6-5-2 high speed steel. The tool was machined to have a shoulder diameter 20 mm, pin diameter 8 mm, and pin depth 4.5 mm. The workpiece was clamped tightly to an 8 mm thick plate made of plain carbon steel which served as a reinforcement and then fixed to the machine table. The surface of plates was not cleaned before processing. The rhenium powder was filled into a groove of 2–3 mm width and 1–2 mm depth. Double passes were applied in order to improve the homogeneity of the Re particle distribution. The first pass was conducted in such way that grove was on the advancing side. In the second pass, the tool was moved along the same line as the axis of the grove at the same travelling direction.

Examination of the microstructure for all of the samples was studied using a light microscope OLYMPUS GX5 and Scanning Electron Microscope Hitachi SU 70. The samples to light microscopy observations were polished mechanically applied Struers equipment and technique. They were grinded, than polished in diamond pastes and in suspension OPS. Microhardness of modification material was performed by using a microhardness types PMT3 at load 100 G.

Fig. 1. FSP Triflute tool
1. ábra FSP Triflute szerszám

3. Results and discussion

Firstly, the microscopic examination of parent material cast aluminium alloy AlSi9Mg were performed by SEM and light microscopies. *Fig. 2* shows LM and SEM micrographs of AlSi9Mg samples in the as-cast condition. The microstructure is mainly composed of α-Al dendrites and Si particle concentrated Al-Si eutectic regions. This type Si particles segregation in the interdendritic regions is the effect of the natural solidification sequence of this alloy, the sizes and morphologies vary depending on the melt chemistry and the cooling rate. The porosity occurs in the base metal (*Fig. 3*). The distribution of Si, Mg particles is not uniform throughout the aluminium matrix [22].

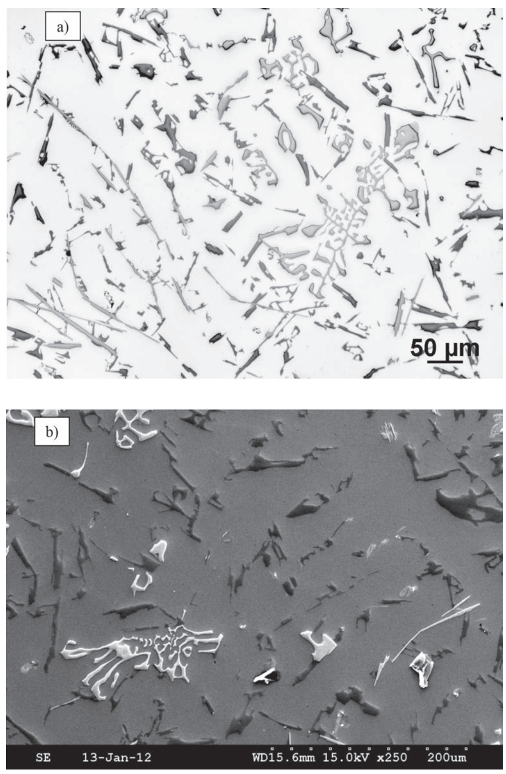

Fig. 2. Microstructure of cast aluminium alloy AlSi9Mg grade, a) light microscopy, b) SEM
2. ábra AlSi9Mg minőségű alumínium ötvözet mikroszerkezete, a) fénymikroszkópia, b) SEM

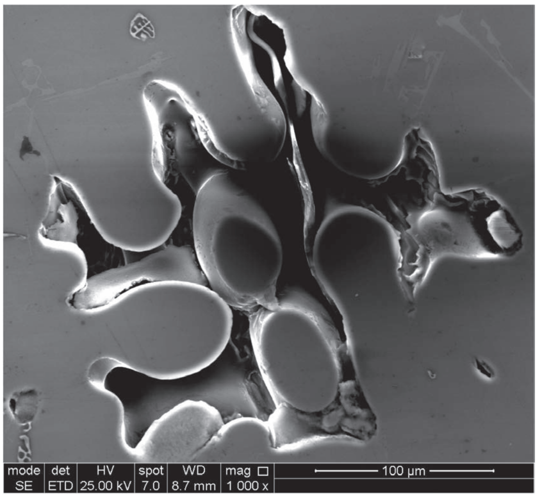

Fig. 3. The porosity of cast aluminium alloy AlSi9Mg grade
3. ábra AlSi9Mg minőségű alumínium ötvözet porozitása

Secondly, the macrostructure and microstructure after FSP process with rhenium were analysed. Particles of Re of 73 µm median diameter were used as the reinforcement as shown in *Figs. 4* and *5*. The single grain of Re powder has no uniform shape and dimension.

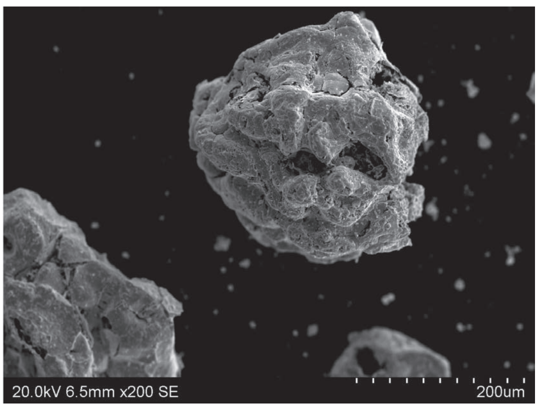

Fig. 4. SEM observation of Re particles
4. ábra Re szemcsék SEM felvétele

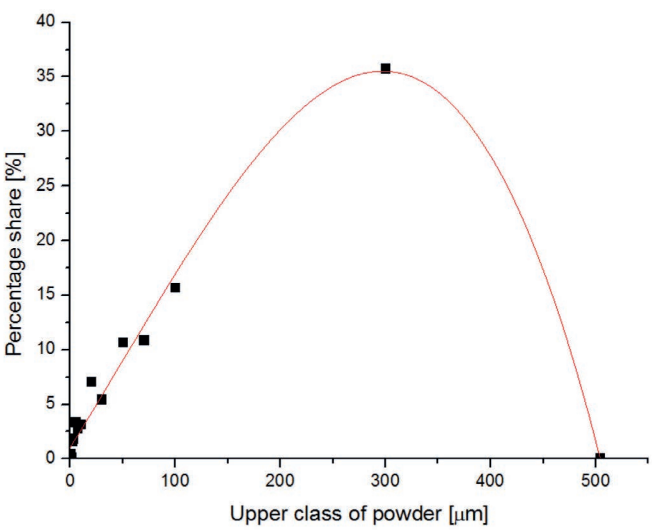

Fig. 5. Percentage share of upper class of powder
5. ábra A por felső osztályának százalékos aránya

Transverse section of the as-received AlSi9Mg after FSP modification on *Fig. 6* is presented. Severe plastic deformation and material flow caused by the stirring action of the tool together with increased temperatures due to friction force are responsible for the grain refinement and dynamic recrystallization in the processed area. FSP also brings about the closure of casting pores and a general homogenization of the microstructure. As a result of FSP the as-cast material is converted into a near-wrought condition. The microstructure of the area subjected to the FSP process with the specific parameters shows a classic asymmetry and macroscopic inhomogeneity. On the strip on the advanced side, where the microstructure is altered by reinforcement, there is a local segregation at the interface between the material flow direction.

Moreover, the FSP resulted in a significant refinement of large Si particles and subsequent uniform distribution in the aluminium matrix as well as porosity in the as cast AlSi9Mg was nearly eliminated by FSP. The structures and analysis of AlSi9Mg alloy after the introduction of rhenium by Friction Stir Processing are compared in *Figs. 7-8*. The chemical analysis by EDS has indicated the presence of rhenium in both the surface layer and the interior of the examined material. On the whole surface of the test material, rhenium was disposed in the form of white "spots" (*Fig. 7a*). On the cross-section of the test material, Vickers microhardness was measured under a load of 100 g. The obtained values differed quite considerably. The places on the sample surface and in the interior where the presence of rhenium has been identified showed an increase of microhardness up to about 100 µHV, while in the remaining areas the values of microhardness ranged from 68 - 75 µHV.

4. Conclusions

The present study has examined the FSP process after modified AlSi9Mg cast aluminium alloy with rhenium. The results were as follows:

- the microstructure of the area subjected to the FSP process shows a classical asymmetry and macroscopic inhomogeneity,
- FSP of the AlSi9Mg aluminum alloy resulted in a significant breakup of coarse acicular Si particles and primary aluminum dendrites, created a homogeneous distribution of Si particles in the aluminum matrix, and nearly eliminated all casting porosity,
- on the whole surface of the test material, rhenium was disposed in the form of white „spots"
- an increase of microhardness up to about 100 µHV is observed while in the remaining areas the values of microhardness ranged from 68 - 75 µHV,
- SEM-EDS analysis revealed concentration of rhenium in the modified material.

5. Acknowledgements

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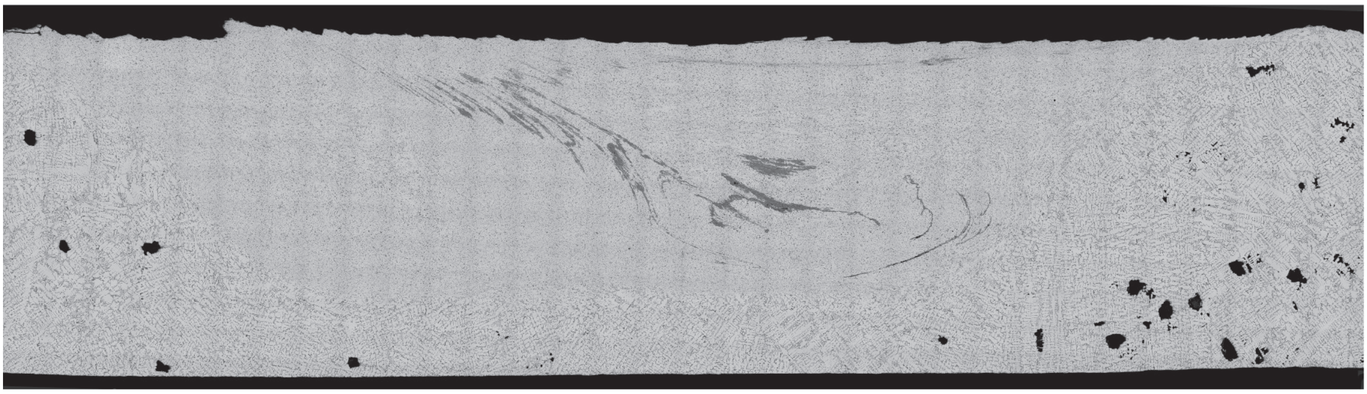

Fig. 6. Macrostructure of the cross-sectional area after FSP modification, advancing side on the left
6. ábra Keresztmetszet makroszerkezete a dörzshegesztést követően, előre oldal a baloldalon

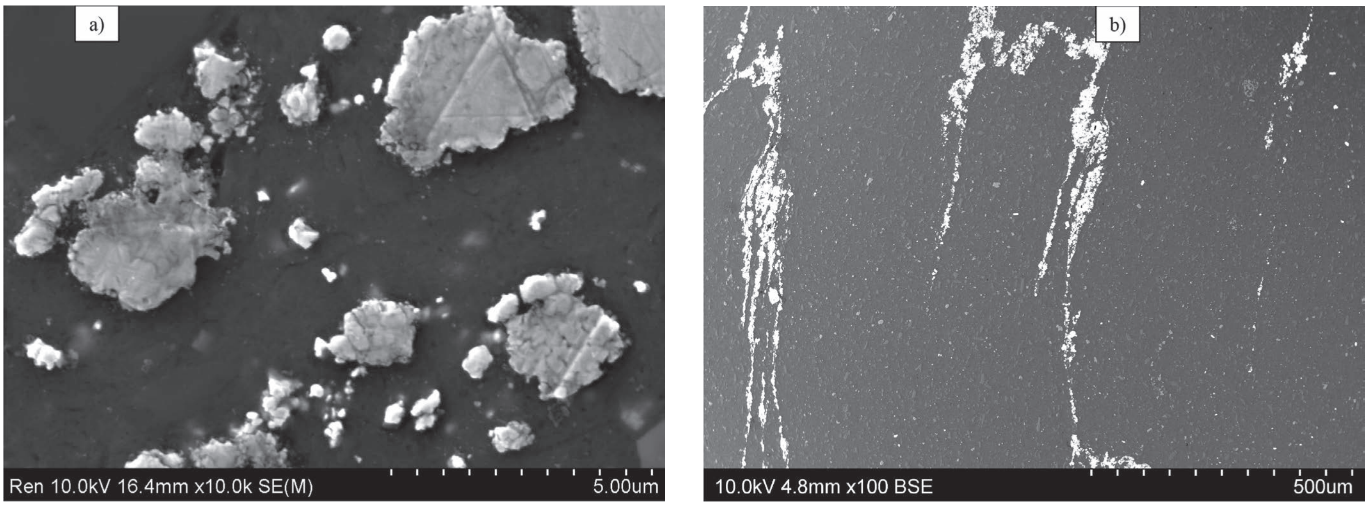

Fig. 7. Macrostructure of the cross-sectional area after FSP modification: a) the upper surface, b) the middle part tested material (SEM)
7. ábra Keresztmetszet makroszerkezete a dörzshegesztést követően: a) felső felület, b) a középső területen vizsgált anyag (SEM)

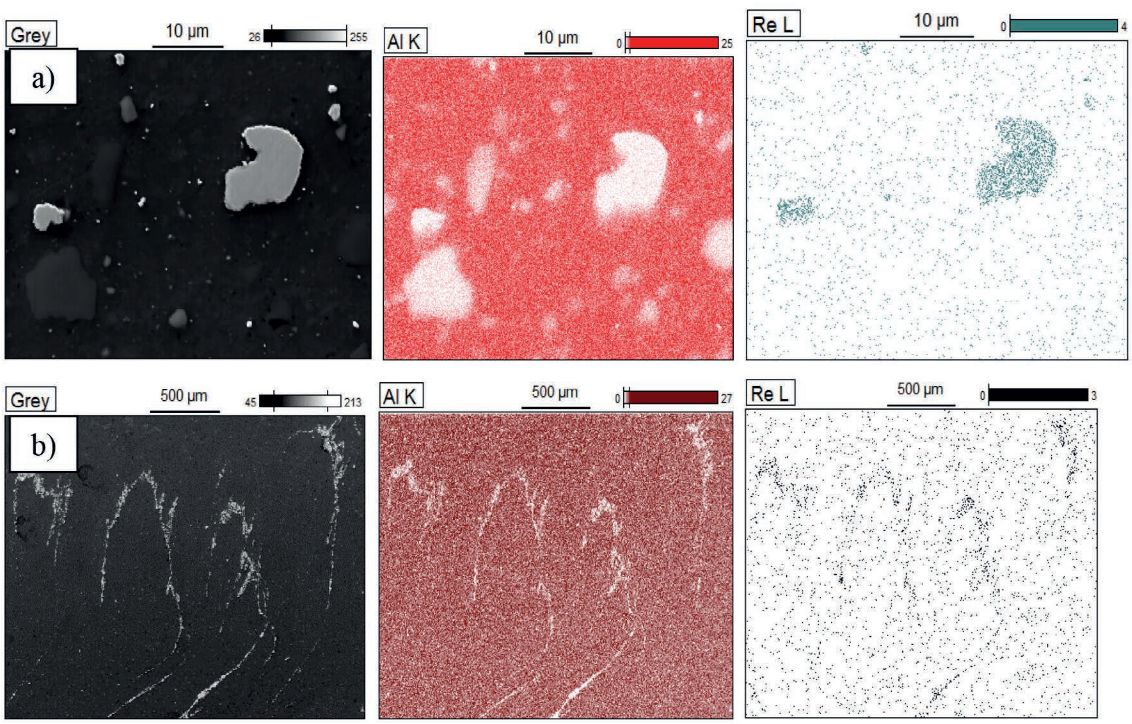

Fig. 8. SEM analysis with corresponding EDS spectra: a) the upper surface, b) the middle part tested material
8. ábra SEM analízis, összetartozó EDS spektrumokkal: a) felső felület, b) a középső területen vizsgált anyag

References

[1] Mahmoud, E. R. I. – Ikeuchi, K. – Takahashi, M. (2008): Fabrication of SiC particle reinforced composite on aluminium surface by friction stir processing. *Science and Technology of Welding and Joining.* Vol. 13, No 7, pp. 607-618, https://doi.org/10.1179/136217108X333327

[2] Węglowski, M. St. – Dymek, S. (2013): Relationship between Friction Stir Processing parameters and torque, temperature and the penetration depth of the tool. *Archives of Civil and Mechanical Engineering.* Vol. 13, No 2, pp. 186-191, https://doi.org/10.1016/j.acme.2013.01.003

[3] Weglowski, M. St. – Pietras, A. – Dymek, S. – Hamilton C. (2012): Characterization of Friction Modified Processing – a Novel Tool for Enhancing Surface Properties in Cast Aluminium Alloys. *Key Engineering Materials*. Vol. 504-506, pp. 1231-1236, https://doi.org/10.4028/www.scientific.net/kem.504-506.1231

[4] Hamilton, C. – Weglowski, M. St. – Dymek, S. (2015): A Simulation of Friction-Stir Processing for Temperature and Material Flow. *Metallurgical and Materials Transactions B. Vol. 46*, No 3, pp. 1409-1418, https://doi.org/10.1007/s11663-015-0340-z

[5] Węglowski, M. St. – Dymek, S. (2012): Microstructural modification of cast aluminium alloy AlSi9Mg via Friction Modified Processing. *Archives of Metallurgy and Materials.* Vol. 57, No 1, pp. 71-78, https://doi.org/10.2478/v10172-011-0155-0

[6] Shafiei-Zarghani, A. – Kashani-Bozorg, S. F. – Zarei-Hanzaki, A. (2009): Microstructures and mechanical properties of Al/Al2O3 surface nano-composite layer produced by friction stir processing. *Materials Science and Engineering A.* Vol. 500, No 1-2, pp. 84-91. https://doi.org/10.1016/j.msea.2008.09.064

[7] Kopyscianski, M. – Weglowski, M. St. – Dziadosz, M. – Malyszko, M. – Dymek, S. (2015): Electron microscopy investigation of a cast AlSi9Mg aluminum alloy subjected to friction stir processing with overlapping passes. *International Journal of Materials Research*. Vol. 106, No 7, pp. 813-817, https://doi.org/10.3139/146.111203

[8] Gandra, J. – Miranda, R. et al. (2011): Functionally graded materials produced by friction stir processing. *Journal of Materials Processing Technology*. Vol. 211, No 11, pp. 1659-1668. https://doi.org/10.1016/j.jmatprotec.2011.04.016

[9] Mahmoud, E. R. I. – Takahashi, M., et al. (2009): Effect of friction stir processing tool probe on fabrication of SiC particle reinforced composite on aluminum surface. *Science and Technology of Welding and Joining*. Vol. 14 No, pp. 413÷425, https://doi.org/10.1179/136217109X406974

[10] Kurtyka, P. – Rylko, N. – Tokarski, T., et al. (2015): Cast aluminium matrix composites modified with using FSP process - Changing of the structure and mechanical properties. *Composite Structures*. Vol. 133, pp. 959-967, https://doi.org/10.1016/j.compstruct.2015.07.122

[11] Sathiskumar, R. – Murugan, N., et al. (2013): Characterization of boron carbide particulate reinforced in situ copper surface composites synthesized using friction stir processing. *Materials Characterization*. Vol. 84, pp. 16–27, https://doi.org/10.1016/j.matchar.2013.07.001

[12] Sharma, V. – Prakash, U. – Kumar, B. V. M. (2015): Surface composites by friction stir processing: A review. *Journal of Materials Processing Technology*. Vol. 224, pp. 117–134, https://doi.org/10.1016/j.jmatprotec.2015.04.019

[13] Węglowski, M. St., (2018): Friction Stir Processing – state of the art. *Archives of Civil and Mechanical Engineering*. Vol. 18, No 1, pp. 114-129, https://doi.org/10.1016/j.acme.2017.06.002

[14] Davis, J. R. (1997): ASM specialty handbook: Heat-resistant materials, *ASM International*, 379-380.

[15] Poole, Jr. Ch. P. (2004): Encyclopedic dictionary of condensed matter physics, Vol. 1 A-M, *Elsevier Inc.,* San Diego, pp. 1142-1143.

[16] Huang, L. – Sun, X. F. – Guan, H. R. – Hu, Z. Q. (2006): Effect of rhenium addition on isothermal oxidation behavior of single-crystal Ni-based superalloy. *Surface and Coatings Technology*. Vol. 200, No 24, pp. 6863-6870, https://doi.org/10.1016/j.surfcoat.2005.10.037

[17] Kolmakov, A. G. – Geminov, V. N., et al. (1995): Effect of rhenium coatings on the mechanical properties of molybdenum wires. *Surface and Coatings Technology*. Vol. 72, No 1-2, pp. 43-50,5 https://doi.org/10.1016/0257-8972(94)02329-O

[18] Chrzanowska, J. – Hoffman, J. (2015): The effect of process parameters on rhenium diboride films deposited by PLD. *Surface and Coatings Technology*. Vol. 277, pp. 15–22, https://doi.org/10.1016/j.surfcoat.2015.07.024

[19] Chmielewski, M. – Piatkowska, A. (2015): Effect of Rhenium Addition on Wear Behavior of Cr-Al2O3 Metal Matrix Composites. *Journal of Materials Engineering and Performance*. Vol. 24, No 5, pp. 1871-188, https://doi.org/10.1007/s11665-015-1462-9

[20] Chmielewski, M. – Pietrzak, K. – Strojny-Nedza, A. – Dubiel, B. – Czyrska-Filemonowicz, A. (2014): Effect of Rhenium Addition on the Strengthening of Chromium-Alumina Composite Materials. *International Journal of Materials Research*. Vol. 105, No 2, pp. 200–207, https://doi.org/10.3139/146.111002

[21] Kurzynowski, T. – Smolina, I., et al. (2017): Wear and corrosion behaviour of Inconel 718 laser surface alloyed with rhenium. Materials & Desing, Vol. 132, pp. 349-359, https://doi.org/10.1016/j.matdes.2017.07.024

[22] Weglowski, M. St., (2014): Microstructural characterisation of friction stir processed cast AlSi9Mg aluminium alloy. *Archives of Foundry Engineering*. Vol. 14, No 3, pp.75-78.

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