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EFFECT OF HEAT TREATMENT ON METALLURGICAL AND MECHANICAL PROPERTIES OF AN ALUMINIUM 6061 HYBRID COMPOSITE

VPLIV TOPLOTNE OBDELAVE NA METALURŠKE IN MEHANSKE LASTNOSTI HIBRIDNEGA KOMPOZITA S KOVINSKO OSNOVO IZ ZLITINE Al-6061

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Aluminium hybrid metal matrix composites (Al-HMMCs) are fabricated to investigate the influence of heat treatment on metallurgical and mechanical properties. Aluminium 6061, known for its heat treatability and versatile applications is used as the base material, and it is reinforced with tungsten carbide nanoparticles and graphite. Stir casting is used for fabricating four composite samples. The prepared samples are subjected to a microscopic analysis and mechanical testing. The microscopic analysis shows segregation along the grain boundaries, which are refined upon heat treatment. FESEM images with EDX mapping reveal an even distribution of reinforcement particles without any agglomeration. Two different intermetallic phases are formed in the shapes of plates and scripts, some of which dissolve to form precipitate-resisting grain growth and dislocation movement upon heat treatment, enhancing the mechanical properties. The hardness of heat-treated composites is, on average, 33 % higher than that of non-heat-treated composites. The yield strength and ultimate tensile strength of the heat-treated Al-HMMC are improved by 43 % and 38 %, respectively, compared to the Al6061 alloy. The ductility of Al-HMMC is preserved even with the addition of hard ceramic reinforcement, as evidenced by the percentage elongation of heat-treated Al-HMMC and supported by fractographic analysis.

Keywords: hybrid metal matrix composites, tungsten carbide nanoparticles, graphite, heat treatment

Avtorji so izdelali in preiskovali hibridne kompozite z matrico iz Al zlitine vrste 6061 (Al-HMMC; angl: Aluminium Hybrid Metal Matrix Composites). Analizirali so vpliv toplotne obdelave na njihove metalurške in mehanske lastnosti. Al zlitina vrste 6061 se dobro toplotno obdeluje in uporablja za različne namene. Kovinsko osnovo iz Al 6061 so avtorji ojačali z nano delci volfram karbida (WC) in grafita z izbranim metalurškim postopkom. V ta namen so za izdelavo štirih različnih kompozitov uporabili t.i. postopek umešavanja delcev v kovinsko talino (angl.: stir casting process). Izdelane vzorce kompozitov so metalografsko pregledali pod vrstičnim elektronskim mikroskopom na emisijo polja (FESEM) in izdelali preizkušance za mehanske preiskave. Mikroskopska analiza je pokazala segregacije vzdolž mej kristalnih zrn, ki pa so se zmanjšale s toplotno obdelavo kompozitov. Pregled FESEM posnetkov z mikro-kemijsko linijsko (angl.: mapping) EDX analizo so celo odkrili področja enakomerne porazdelitve delcev ojačitve brez njihove aglomeracije. Tvorili sta se dve različni intermetalni fazi v obliki ploščic in "črk", ki so se med toplotno obdelavo delno raztopili in tvorili izločke. Ti so preprečevali rast kristalnih zrn in gibanje dislokacij ter s tem izboljšali mehanske lastnosti izdelanih kompozitov. Trdota toplotno obdelanih kompozitov je bila približno za 33 % višja kot trdota toplotno neobdelnih vzorcev. Meja plastičnosti in končna natezna trdnost izdelanih kompozitov se je izboljšala za cca 43 % oziroma 38 % v primerjavi s čisto Al 6061 zlitino. Ohranili so tudi njihovo duktilnost, razvidno tudi iz SEM prelomov preizkušancev, kljub dodatku trdih keramičnih delcev.

Ključne besede: hibridni kompozit s kovinsko matrico, nano delci volframovega karbida, grafit, toplotna obdelava

1 INTRODUCTION

Metal matrix composites (MMCs) with reinforcements of varying weight percentages, shapes, and sizes, have become indispensable materials.[1] Hybrid metal matrix composites (HMMCs) with more than one reinforcement provide superior mechanical properties and adaptability compared to single reinforcement composites.[2] The third most abundant material on the Earth, aluminium, is well-known for its light weight, strength, durability, and malleability. Alloyed with other metals, aluminium 6061 is the most widely consumed material for engineering products like bicycle frames, food containers, automotive wheels and aircraft components such as bulkheads, fuselages, stabilizers and wings. Heat-treatable aluminium alloys hold particular prominence due to their aplicability across engineering domains and aluminium alloy Al6061 stands out, often enhancing the properties of its equivalents in the Al2XXX series.

Cao Fenghong et al. reinforced aluminium 6061 with equal proportions of tungsten carbide (WC) and silicon carbide. Hardness, ultimate tensile strength, yield strength and compressive strength of the hybrid composite increase with the increase in the weight percentage of reinforcement, but percentage of elongation decreases.[3] Suresh et al. studied mechanical properties of silicon carbide and tungsten carbide reinforced Al6061 hybrid metal matrix composites. The hybrid metal matrix com-

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posites had better mechanical properties with a reduced particle size, improving the wettability and homogeneous dispersion in the matrix.[1] Ebenezer Jacob Dhas et al. examined the effect of silicon carbide and tungsten carbide reinforced aluminium metal matrix composites with graphite as the solid lubricant. Though the ductility of tungsten carbide reinforced composites is found to be inferior, its hardness, tensile and compressive strength are superior to silicon carbide reinforced composites.[4]

Swamy et al. analysed the effect of tungsten carbide reinforced metal matrix composites and found that their properties improved with an addition of 3 *w*/% of tungsten carbide, beyond which they started to deteriorate.[5] Veeresh Kumar and Pramod fabricated Al6061 composites with tungsten carbide (1, 2 and 3) *w*/% as the reinforcement using powder metallurgy. An increase in the tensile strength and hardness is observed with an addition of the reinforcement at the expense of ductility, which is found to decrease by nearly 60 % compared to the base material.[6] Swamy et al. investigated individual effects of graphite and tungsten carbide on Al6061 based metal matrix composites. The hardness, ultimate tensile strength and compressive strength of 3 *w*/% tungsten carbide reinforced MMC are found to be the best and upon further increase in the weight percentage they decrease. Percentage elongation (ductility) decreases upon an addition of tungsten carbide, but it increases with an addition of graphite. While ultimate tensile strength and compressive strength increase with the addition of graphite, the hardness decreases.[7]

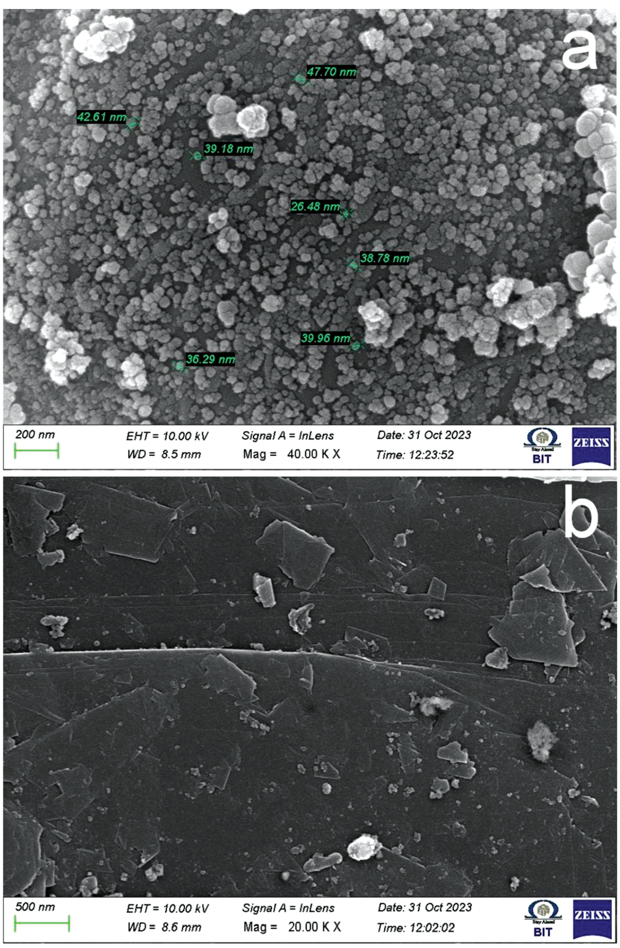

Figure 1: FESEM images of: a) tungsten carbide nanoparticles, b) graphite

Shubhajit Das et al. investigated the effect of nano reinforcement on the properties (physical and mechanical) of hybrid metal matrix composites by fabricating composites with nano and micro reinforcements. It was found that there was no significant change in the density and porosity among the prepared composites. A significant improvement in the hardness, tensile and impact strengths were observed in nano-particle reinforced composites compared to micro reinforcements.[8] Better hardness and tensile strength were achieved with an addition of 1.5 *w*/% of nano Al_2O_3 reinforcement to Al7075 aluminium alloy, beyond which they started decreasing.[9] Vignesh Kumar et al. investigated the mechanical properties of aluminium (AA7075) composites prepared by stir casting with tungsten carbide nanoparticles and molybdenum disulphide (MoS_2) as the reinforcements. Nano-sized particles allowed better mechanical properties at a lower percentage of reinforcement compared to micron-size particles. Beyond 1.5 *w*/% of tungsten carbide nano-particles, tensile, compressive strength and hardness were reduced considerably.[10] A compromise is necessary when selecting the percentage of reinforcement to be added for enhancing the mechanical properties of the composite without sacrificing its ductility.

2 EXPERIMENTAL DETAILS

2.1 Specimen fabrication

Table 1: Compositions and weight percentages of cast specimens

Specimen designation	Al6061 (*w*/%)	Graphite (*w*/%)	Nano WC (*w*/%)
Al6061	100	0	0
Al6061 + Gr	98	2	0
Al6061 + WC	95.5	0	1.5
Al-HMMC	93.5	2	1.5

Aluminum alloy 6061 is the base material; tungsten carbide nanoparticles with an average particle size of 30 nm and graphite particles are used as the reinforcement. FESEM images of the reinforcements are shown in **Figure 1**. Four different samples including re-melted and as-cast Al6061 alloy with the weight percentages specified in **Table 1** were fabricated using stir casting. In the fabrication process, the reinforcing particles were mixed with the molten metal using a stirrer operating at 400 min^{-1} for up to 6 min at two-thirds depth from the molten metal surface.[11,12] Additionally, 1 *w*/% magnesium and 1 *w*/% degassing powder were introduced to the molten matrix to enhance the wettability and remove gases from the melt.[13] Using the bottom pouring mechanism, the molten metal was transferred to a circular mould with a diameter of 25 mm. The cast specimens were machined to obtain a near-net shape to meet the ASTM standards for hardness and tensile testing.

2.2 Heat treatment

To enhance their properties, a T-6 heat treatment process was applied to 50 % of the composite specimens fabricated.[14] This involved the initial solution heat treatment, where the samples were heated up to 530 °C and maintained for 8 h. The heated samples were then quenched in water. Subsequently, solution-treated samples were subjected to artificial aging at 165 °C for 6 h, followed by cooling in air.

2.3 Testing methods

A Carl Zeiss Axiotech 100HD-3D microscope was used to record the optical microstructures of the cast composites. Sample preparation included initial polishing with 600-grit sheets, followed by finish polishing with 6 μm diamond paste on a disc polisher, and etching with Keller's etchant. A field emission scanning electron microscope, Carl Zeiss Sigma 300, was utilized to record surface morphology, reinforcement particle size, and conduct fractographic analysis. A Rockwell hardness tester was used to measure the hardness on the B scale as per ASTM E18 standards. A computerised tensile testing machine with a loading rate of 0.5 mm/min and linear displacement resolution of 0.01 mm was used for tensile tests as per ASTM B577M standards.

3 RESULTS

3.1 Microstructural analysis of the composites

The optical microstructures of the as-cast Al6061 alloy **(Figure 2a)** and Al-HMMC **(Figure 2b)** show equiaxed grains and segregations of secondary phase particles along the grain boundaries. The reinforcements added to the composites are evenly distributed and no agglomeration is observed inside the grains. After the T6 heat treatment, optical micrographs of the as-cast Al6061 alloy **(Figure 2c)** and Al-HMMC **(Figure 2d)** show discontinuous grain boundaries. Undesirable secondary phase segregations along the grain boundaries dissolve into the matrix, resulting in discontinuous grain boundaries. Dissolved atoms diffuse to form precipitates throughout the matrix due to the aging process. Fine precipitates resist the grain growth and movement of dislocations, resulting in better properties compared to the non-heat-treated samples.[15]

3.2 FESEM and EDX analysis of the composites

Figure 3 shows FESEM and EDX mapping of tungsten carbide and graphite reinforced Al-HMMC before heat treatment. Intermetallic phases are formed due to the interaction between the alloying elements of Al6061 like magnesium (Mg), iron (Fe) and silica (Si). Intermetallic phases in two different shapes are observed: a) plate-like ones, most of which dissolve upon heat treatment, and b) script-like ones, which remain undisturbed after heat treatment. Intermetallic phases with Fe-Al-Si and Mg-Si are formed during casting along the grain boundaries and inside the grains, respectively.[16,17] The Fe-Al-Si intermetallic phase is undesirable and becomes dissolved during heat treatment, leading to discontinuous grain boundaries. Dissolved atoms diffuse to form more Mg-Si precipitates both along the grain boundaries and within the grains, resisting the grain growth and dislocation movement. The reinforcements (graphite and tungsten carbide) are evenly distributed throughout the matrix and do not react during the formation of the intermetallic due to their thermal stability.

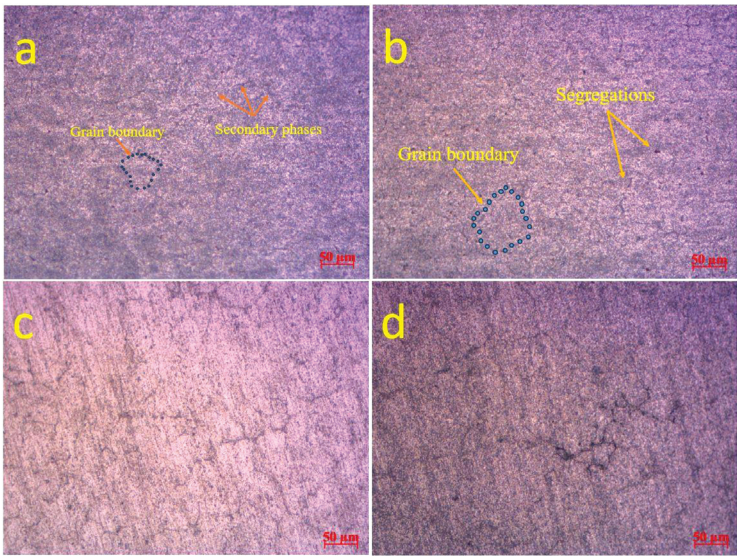

Figure 2: Microscopic images of a) as-cast Al6061 before heat treatment, b) Al-HMMC before heat treatment, c) as-cast Al6061 after heat treatment, d) Al-HMMC after heat treatment

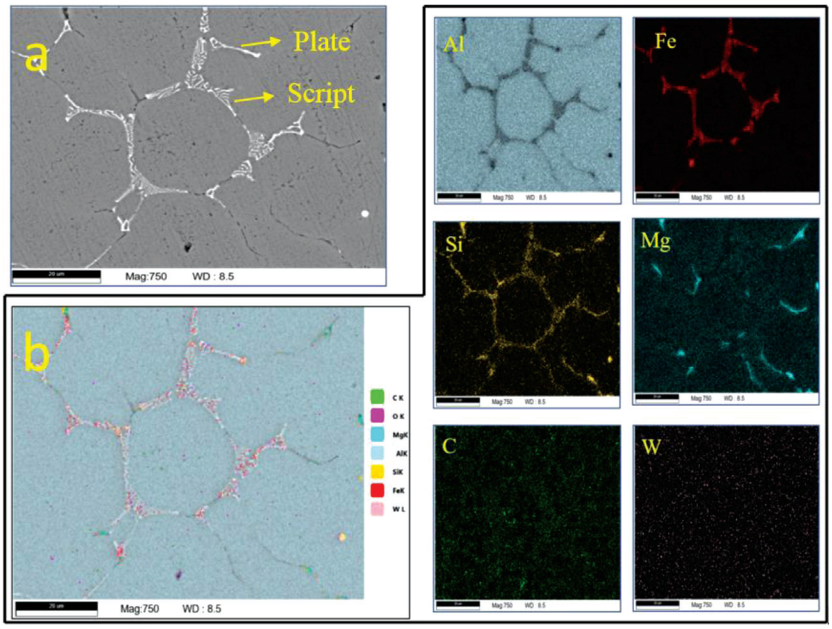

Figure 3: Al-HMMC before heat treatment: a) FESEM image, b) EDX mapping of elements

3.3 Effect of the reinforcements and heat treatment on the hardness

Variation in the hardness of the composites is shown in **Figure 4.** The addition of graphite reduced the hardness of the base alloy and the same effect is observed with Al-HMMC, when adding graphite to the tungsten carbide reinforced Al6061 aluminium alloy. This may be due to the reduced density of graphite in comparison to the base Al6061 alloy. On the other hand, the addition of tungsten carbide nanoparticles improved the hardness significantly due to their higher density and nano-particle size, even when added in a very small percentage (1.5 *w*/%).[7] The increase in the hardness may be attributed to multiple factors, such as the inclusion of hard ceramic particles, or the constraining effect provided by the hard reinforcement particles and intermetallic phases. The hardness of heat treated samples is found to be 33 % higher on average, compared to non-heat treated samples. The hardness of Al-HMMC (heat treated and non-heat treated) is found to be 45 % higher when compared to the base Al6061 alloy.

3.4 Effect of the reinforcements and heat treatment on the tensile strength

Figure 5 shows the tensile strength (yield and ultimate) of composites before and after heat treatment. The uniform distribution of reinforcing particles (WC) reinforces the metal matrix alloy by impeding the plastic flow, resulting in an overall boost in the tensile strength. The rise in the ultimate tensile strength and yield strength is attributed to the strong interfacial bonding between the soft aluminium matrix and tough reinforcement particles. Heat treated composites exhibit better strength compared to non-heat treated composites due to the differences between thermal expansion coefficients and improved interfacial bonding between the alloy and particles.[18] These strengthening mechanisms impede dislocation movement, thereby increasing the strength of the composite material.[19–20]

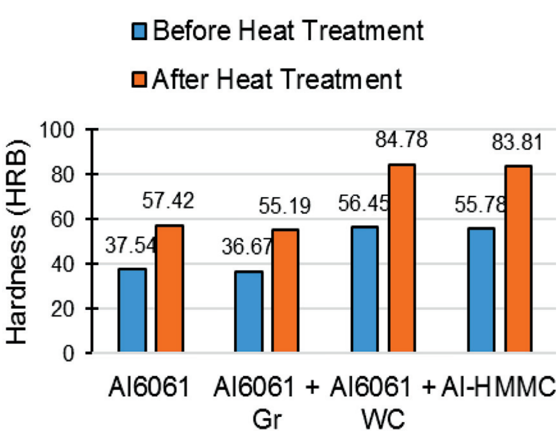

Figure 4: Hardness of Al6061 and Al-HMMCs

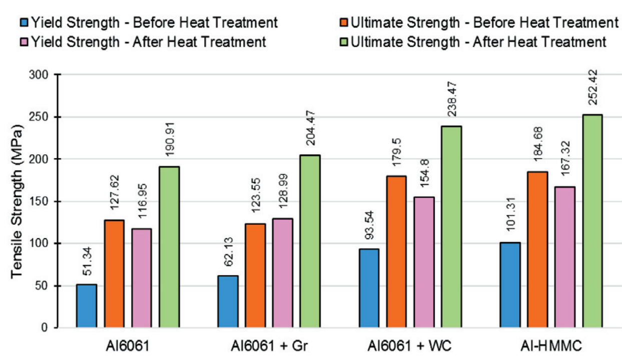

Figure 5: Tensile strength of Al6061 and Al-HMMCs

The yield strength and ultimate tensile strength of Al-HMMC improved by 65 % and 36 %, respectively, upon heat treatment. For the heat treated Al-HMMC, they were found to be 43 % and 38 % higher than those of the heat treated Al6061 aluminium base alloy.

3.5 Effect of the reinforcement and heat treatment on percentage elongation

Table 2: Percentage elongation of Al6061 and Al-HMMCs

Specimen	Before heat treatment	After heat treatment
Al6061	11.62	11.78
Al6061 + Gr	11.35	11.47
Al6061 + WC	10.72	10.85
Al-HMMC	10.60	10.68

The Al6061 base alloy is ductile in nature, exhibiting a percentage elongation of 11.62 % during the tensile test. The addition of hard ceramic tungsten carbide nanoparticles reduced the percentage elongation to a large extent as shown in **Table 2**, compared to the addition of graphite. Heat treated samples respond better to heat and exhibit better percentage elongation than that of non-heat treated samples. Even though percentage elongation is reduced after the reinforcement addition, the ductile nature is maintained as evidenced by the lowest percentage elongation of 10.60 % for the non-heat treated Al-HMMC. The addition of a small amount of reinforcement (1.5 *w*/% WC) in the nano form preserves the percentage elongation of Al-HMMC, which differs from what is reported in the literature.[5–7,21–22]

3.6 Fracture surface analysis of the Al-HMMC tensile specimen

The fracture surface of heat treated Al-HMMC subjected to uniaxial tensile testing is shown in **Figure 6**. The fractograph consists of dimples, tearing edges and small voids, indicating that the fracture of the tensile specimen is ductile in nature.[19] Even though the addition of the hard ceramic reinforcement reduced the percentage elongation (10.68 % for heat treated Al-HMMC) compared to that of the base Al6061 alloy (11.78 % for heat treated Al6061), the ductility of Al-HMMC is not lost as evidenced by the fractograph analysis.

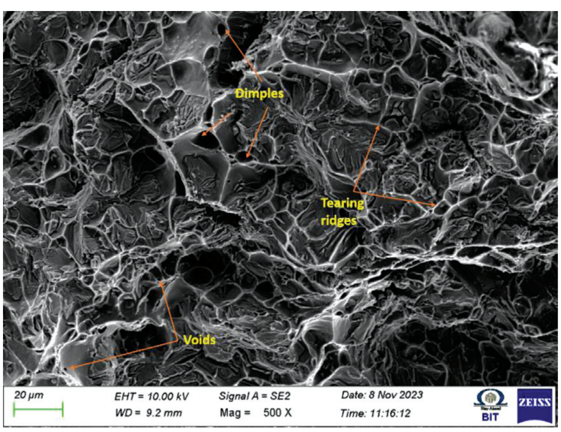

Figure 6: Fractograph of the heat treated Al-HMMC tensile specimen

4 CONCLUSIONS

Composite specimens are fabricated using stir casting and half of the samples are heat treated. The effects of heat treatment and reinforcement addition are investigated and the major conclusions are as follows:

- The microstructure analysis revealed segregations along the grain boundaries and equiaxed grains, which were refined upon heat treatment.
- The FESEM analysis shows the formation of intermetallic phases (Fe-Al-Si and Mg-Si), some of which dissolve upon heat treatment throughout the matrix, improving the mechanical properties.
- The hardness increases with the addition of reinforcement, which is mainly attributed to the addition of tungsten carbide, rather than graphite that is found to reduce hardness due to its lower density. Heat treated samples are found to have better hardness (a 33-% increase) due to the heat treatability of the Al6061 alloy.
- The tensile strength of the reinforced and heat treated composites is better than that of the base aluminium alloy. The yield strength and ultimate tensile strength of Al-HMMC are improved by 65 % and 36 %, respectively, upon heat treatment due to various strengthening mechanisms and reinforcement additions.
- The ductility of Al-HMMC is preserved as evidenced by percentage elongation and fractograph analysis. This may be attributed to the optimal percentage of reinforcement improving the strength without losing the ductility.

5 REFERENCES

[1] N. Suresh, L. Balamurugan, D. Jayabalakrishnan, Influence of SiC and WC reinforcements on the mechanical characteristics of AA6061 hybrid metal matrix composites, Proceedings of the Institution of Mechanical Engineers, Part E: Journal of Process Mechanical Engineering, 237 (**2023**) 3, 955–970, doi:10.1177/09544089221141353

[2] P. Rohatgi, R. Guo, D. Golden, Utilization of fly ash in metallic composites, Proceedings of the American Power Conference, 58 (**1996**) 1, 767

[3] C. Fenghong, C. Chang, W. Zhenyu, T. Muthuramalingam, G. Anbuchezhiyan, Effects of Silicon Carbide and Tungsten Carbide in Aluminium Metal Matrix Composites, Silicon, 11 (**2019**), 2625–2632, doi:10.1007/s12633-018-0051-6

[4] D. S. Ebenezer Jacob Dhas, C. Velmurugan, K. Leo Dev Wins, K. P. BoopathiRaja, Effect of tungsten carbide, silicon carbide and graphite particulates on the mechanical and microstructural characteristics of AA 5052 hybrid composites, Ceramics International, 45 (**2019**) 1, 614–621, doi:10.1016/j.ceramint.2018.09.216

[5] A. R. K. Swamy, A. Ramesha, J. N. Prakash, G. B. Veeresh Kumar, Mechanical and tribological properties of as-cast Al6061-tungsten

carbide metal matrix composites, Material Science Research India, 7 (**2010**) 2, 355–368, doi:10.13005/msri/070205

[6] G. B. Veeresh Kumar, R. Pramod, Influence of WC Particulate Reinforcement on the Mechanical Properties and Sliding Wear of Al6061 Alloys, Applied Mechanics and Materials, 813–814 (**2015**), 67–73, doi:10.4028/www.scientific.net/amm.813-814.67

[7] A. R. K. Swamy, A. Ramesha, G. B. Veeresh Kumar, J. N. Prakash, Effect of Particulate Reinforcements on the Mechanical Properties of Al6061-WC and Al6061-Gr MMCs, Journal of Minerals and Materials Characterization and Engineering, 10 (**2011**) 12, 1141–1152, doi:10.4236/jmmce.2011.1012087

[8] Shubhajit Das, M. Chandrasekaran, S. Samanta, Comparison of mechanical properties of AA6061 reinforced with (SiC/B_4C) micro/nano ceramic particle reinforcements, Materials Today: Proceedings, 5 (**2018**) 9 (3), 18110–18119, doi:10.1016/j.matpr.2018.06.146

[9] W. Ruirui, Y. Zheng, L. Qiushu, Microstructure and mechanical properties of 7075 Al alloy based composites with Al_2O_3 nanoparticles, International Journal of Cast Metals Research, 30 (**2017**) 6, 337–340, doi:10.1080/13640461.2017.1316954

[10] D. Vignesh Kumar, S. Arulselvan, A. Arul Marcel Moshi, C. Veera Ajay, P. Balamurugan, Mechanical characterization and frictional wear behavior analysis on nano tungsten carbide and molybdenum disulfide particles reinforced aluminium 7075 composites, Proceedings of the Institution of Mechanical Engineers, Part E: Journal of Process Mechanical Engineering, (**2023**), doi:10.1177/09544089221150726

[11] G. G. Sozhamannan, S. Balasivanandha Prabu, V. S. K. Venkatagalapathy, Effect of Processing Parameters on Metal Matrix Composites: Stir Casting Process, Journal of Surface Engineered Materials and Advanced Technology, 2 (**2012**) 1, 11–15, doi:10.4236/jsemat.2012.21002

[12] S. Balasivanandha Prabu, L. Karunamoorthy, S. Kathiresan, B. Mohan, Influence of stirring speed and stirring time on distribution of particles in cast metal matrix composite, Journal of Materials Processing Technology, 171 (**2006**) 2, 268–273, doi:10.1016/j.jmatprotec.2005.06.071

[13] P. Vijay, K. V. Brahma Raju, K. Ramji, S. Kamaluddin, Effect of tungsten carbide on Al6061/SiC hybrid metal matrix composites, Composites Theory and Practice, 21 (**2021**) 4, 169–180

[14] S. Das, S. V. Prasad, T. R. Ramachandran, Microstructure and wear of cast (Al-Si alloy)-graphite composites, Wear, 133 (**1989**) 1, 173–187, doi:10.1016/0043-1648(89)90122-1

[15] T. Muthuramalingam, A. Ramamurthy, K. Sridharan, S. Ashwin, Analysis of surface performance measures on WEDM processed titanium alloy with coated electrodes, Materials Research Express, 5 (**2018**) 12, doi:10.1088/2053-1591/aade70

[16] L. Quan, W. Junsheng, L. Xinxiu, W. Bing, Minimizing detrimental impacts of β-Fe in Al-Mg-Si alloy by combining thermal and compression processes, Materials Characterization, 198 (**2023**), 112752, doi:10.1016/j.matchar.2023.112752

[17] N. Srivastava, G. P. Chaudhari, Microstructural evolution and mechanical behavior of ultrasonically synthesized Al6061-nano alumina composites, Materials Science and Engineering: A, 724 (**2018**), 199–207, doi:10.1016/j.msea.2018.03.092

[18] I. Dinaharan, R. Nelson, S. J. Vijay, E. T. Akinlabi, Microstructure and wear characterization of aluminum matrix composites reinforced with industrial waste fly ash particulates synthesized by friction stir processing, Materials Characterization, 118 (**2016**), 149–158, doi:10.1016/j.matchar.2016.05.017

[19] G. Huang, W. Hou, Y. Shen, Evaluation of the microstructure and mechanical properties of WC particle reinforced aluminum matrix composites fabricated by friction stir processing, Materials Characterization, 138 (**2018**), 26–37, doi:10.1016/j.matchar.2018.01.053

[20] N. Ashok, P. Shanmughasundaram, Effect of particle size on the mechanical properties of SiC-reinforced aluminium 8011 composites, Mater. Tehnol., 51 (**2017**) 4, 667–672, doi:10.17222/mit.2016.252

[21] R. Manikandan, T. V. Arjunan, R. Akhil, O. P. Nath, Studies on micro structural characteristics, mechanical and tribological behaviours of boron carbide and cow dung ash reinforced aluminium (Al 7075) hybrid metal matrix composite, Composites Part B: Engineering, 183 (**2020**), 107668, doi:10.1016/j.compositesb.2019.107668

[22] P. Subramanya Reddy, R. Kesavan, B. Vijaya Ramnath, Investigation of Mechanical Properties of Aluminium 6061-Silicon Carbide, Boron Carbide Metal Matrix Composite, Silicon, 10 (**2018**), 495–502, doi:10.1007/s12633-016-9479-8