

CHARACTERIZATION OF HYBRID ALUMINUM COMPOSITES REINFORCED WITH Al_2O_3 PARTICLES AND WALNUT-SHELL ASH

KARAKTERIZACIJA HIBRIDNIH KOMPOZITOV NA OSNOVI ALUMINIJA, OJAČANIH Z DELCI Al_2O_3 IN PEPELA IZ OREHOVIH LUPIN

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Hybrid aluminum composites obtained with stir casting were investigated in this study. Aluminum alloy EN AW 6061 was reinforced with particles of Al_2O_3 and walnut-shell ash (WSA). The weight fraction of Al_2O_3 was constant (5 w%) and for the walnut-shell ash, it was (1, 2 and 3) w%. The morphology of the composites and particle distribution were examined with an optical microscope and scanning electron microscope. Microstructural studies showed a uniform distribution of the reinforced particles in all the investigated samples. A chemical analysis of the reinforcing particles on the samples' surfaces was done using an energy-dispersive spectrometer. The changes in the mechanical properties including the hardness, tensile strength and elongation in relation to the increase in the weight fraction of ash were investigated. The results showed that the hardness and tensile strength increase, while the elongation decreases with an increase in the weight fraction of ash in the composites. The mechanical properties of the obtained composites showed improvement compared to the EN AW 6061 alloy.

Keywords: aluminium alloy, reinforcement, walnut shell, ash

Članek opisuje raziskavo hibridnih kompozitov na osnovi Al zlitine, izdelanih s postopkom gnetenja oziroma livarskega postopka premešavanja z gnetenjem (angl.: stir casting). Zlitino na osnovi Al z oznako EN AW 6061 so ojačali z delci Al_2O_3 in pepelom orehovit lupin (WSA; angl.: walnut shell ash). Masni delež delcev Al_2O_3 so držali konstanten (5 w%), medtem ko so bili izbrani masni deleži delcev pepela (1, 2 in 3) w%. Morfologijo izdelanih kompozitov in porazdelitev delcev so opazovali pod optičnim in vrstičnim elektronskim mikroskopom. Mikrostrukturne študije so pokazale, da je v vseh izdelanih kompozitih porazdelitev delcev ojačitve v kovinski osnovi enakomerna. Kemijska analiza delcev ojačitve je bila izvedena na površini vzorcev z energijskim disperzijskim spektrometrom. Določene so bile tudi spremembe mehanskih lastnosti, kot so trdota, natezna trdnost in raztezek, zaradi spremembe oz. povečanja deleža ojačitvene faze (pepela). Rezultati analiz so pokazali, da trdota in natezna trdnost naraščata, medtem ko se raztezek kompozita zmanjšuje s povečevanjem deleža pepela orehovit lupin v kompozitu. Očitno je bilo torej izboljšanje mehanskih lastnosti (trdote in trdnosti) izdelanih hibridnih kompozitov v primerjavi z osnovno Al zlitino EN AW 6061.

Ključne besede: zlitina na osnovi aluminija, ojačitev, orehove lupine, pepel

1 INTRODUCTION

The continuous development of science and technology has contributed to the development of new materials for the production and design of engineering products. Since the middle of the last century, special attention was focused on the research of composite materials. Composites were created with the idea of combining two or more materials to obtain new ones, using the best properties of components.¹⁻³ The research is usually conducted on metal-matrix composites – MMCs. Since the components do not mix, two phases can be clearly distinguished in the structure of a composite: the metal base (matrix) and the reinforcing phase (reinforcer). Aluminum, titanium, magnesium, copper and their alloys are the most often used metal bases for making MMCs.^{4,5}

Reinforcement materials are ceramic particles in the form of oxides (Al_2O_3 , MgO, TiO_2), carbides (SiC, TiC, B_4C), nitrides (AlN, BN, Si_3N_4) and borides (TiB_2).^{6,7} Aluminum matrix composites – AMCs, most often reinforced with Al_2O_3 and SiC particles, have the largest market share among different MMCs.⁸ Recent research has been conducted in the area of hybrid aluminium-matrix composites – HAMCs. The name implies that there are two or more reinforcements in their structure.⁹ They are produced in order to improve the characteristics of composite materials, achieve competitive prices on the market and meet the environmental-protection requirements. Three combinations of reinforcing particles can be found in HAMCs. The first combination involves two different synthetic ceramic materials, the second combines ceramic particles and industrial waste ash, and the third combines ceramic particles and agro-waste ash.^{10,11} The use of the last combination is expanding.

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An addition of reinforcements to a metal matrix provides: increased strength, higher modulus of elasticity, higher operating temperature, improved wear resistance, reduced weight of structural parts, high electrical and thermal conductivity, low coefficient of thermal expansion, and dimensional stability of the product compared to metals and alloys.^{12,13} HAMCs are characterized by their low price, isotropic properties and the possibility of a production using the existing technologies.⁹ They are used in almost all branches of industry: automotive, aerospace, military, construction, medicine and others. Diversity in the application requires answers to many questions regarding the production and characterization of metal composites. HAMCs can be synthesized with various methods and this is a key factor in achieving the required material properties. However, ingot-metallurgy methods have prevailed.¹⁴⁻¹⁷ Among them, stir casting stands out due to its simplicity, economy and high productivity.⁴ The choice of parameters for this process determines the distribution of reinforcement in the composite material, which directly affects its mechanical and tribological properties.^{18,19}

Disadvantages, which can occur in HAMCs, are an inhomogeneity in the particle distribution, occurrence of particle agglomeration or weak connection between the metal base and the reinforcer. The main cause of these defects is poor wettability of the reinforcing particles with the liquid metal, which can be overcome in several ways. The most prominent ones include an addition of wettability-supporting alloying elements (magnesium is the most common), mechanical mixing which is an integral part of the stir-casting process, and heating of the reinforcing particles before adding them to the molten metal matrix.²⁰

Alaneme et al.²¹ investigated the production conditions and mechanical properties of a hybrid aluminum composite based on the AlMgSi alloy. The reinforcing phase consisted of mixed Al_2O_3 particles and rice-husk ash (RHA) in the total amount of 10 w%. The composites were obtained with the two-steps stir-casting method. Their results showed a decrease in the density and microhardness of the hybrid composites compared to the composites reinforced only with Al_2O_3 particles. Alaneme and Adewale²² studied the effect of reinforcements on the mechanical properties of AlMgSi alloy composites. Their results showed that the porosity decreased, and the tensile strength and yield strength increased with the increasing percentage of the reinforcing phase. It was also observed that the tensile strength and yield strength decreased with an increasing RHA content in the reinforcing phase. The elongation of the composite decreased with an increasing proportion of the reinforcing phase. Narasaraju and Raju²³ used RHA and fly ash in different weight ratios as the AlSi10Mg alloy reinforcers. Samples were obtained with stir casting. Hardness and tensile strength increased with a decreasing RHA and fly-ash ratio in the composite. Elongation values were higher when the fly-ash content decreased and the RHA content increased.

However, MMCs, although expanding, still have a limited market share due to the difficulties in production processes and high production costs. Recent scientific investigations have aimed to overcome these problems showing agro-waste ash particles as a promising material. Using these ashes, the total costs are significantly reduced. Rice-husk ash (RHA), bagasse ash (BA), bamboo-leaf ash (BLA), maize-stalk ash (MSA), corn-cob ash (CCA), sugarcane bagasse and many more²⁴⁻²⁶ are the most commonly used materials.

According to the available literature, it can be concluded that the influence of WSA on the mechanical properties of HAMCs has not been investigated so far. The use of this agro waste avoids the problem of its disposal, which is very important from the point of view of environmental protection. In economic terms, the use of WSA provides a competitive price of HAMCs in the market and increases their use in industry. When choosing a material for industrial use, one of the most important criteria is mechanical properties. Hybrid composites based on the EN AW 6061 alloy are increasingly used for the production of parts for cars, planes and yachts, so this research focuses on these areas.

2 EXPERIMENTAL PART

2.1. Materials

For the production of HAMC materials, aluminum alloy EN AW 6061, Al_2O_3 particles (an average size of 50 μm) with a weight fraction of 5 w% and WSA with weight fractions of (1, 2 and 3) w% were used as the starting materials.

2.2 Equipment

Samples for experimental tests were obtained with stir casting. A schematic representation of the equipment used to perform this procedure is shown in **Figure 1**.

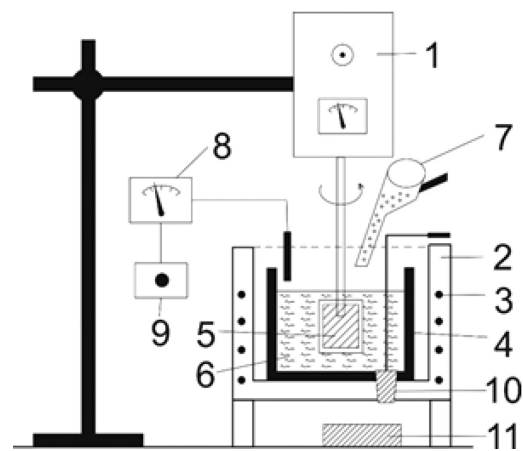


Figure 1: Scheme of the stir-casting experimental set-up: 1 – motor with speed regulation, 2 – electric furnace, 3 – heaters, 4 – graphite crucible, 5 – mixer, 6 – molten metal, 7 – funnel for dosing reinforcing particles, 8 – thermocouple, 9 – temperature regulator, 10 – stopper, 11 – metal mold

Stir casting consisted of melting the batch, stirring the melt, infiltrating the reinforcement particles and pouring the mixture into a heated graphite mold. The EN AW 6061 alloy was melted in a graphite crucible by heating it in an electric resistance furnace called Heraeus K 1150/2 to 800 °C. Magnesium in an amount of 1 w% was added to the melt to increase the wettability of the reinforcement particles. The melt was cooled to 710 °C to increase the viscosity. At this temperature, the stirring of the melt began, with a plate-shaped steel-tool stirrer that was previously heated in the furnace at 350 °C in order not to cool down the melt. Stirring with a revolution of 500 min⁻¹ was applied. After stirring for 2 min to homogenize the molten-metal matrix, Al₂O₃ and WSA particles were added. The particles were preheated in the furnace at 500 °C for 3 h to remove the moisture. After adding the total amount of reinforcements, stirring was continued for another 5 min in order to distribute them uniformly throughout the melt. The HAMC was poured through a siphon into a metal mold. HAMC ingots were obtained with dimensions of (200 × 20 × 15) mm. From the HAMC, specimens were cut for mechanical and metallographic tests. For the sake of comparison, the EN AW 6061 alloy was cast and investigated, too. The designations of the synthesized HAMC samples are shown in **Table 1**.

Table 1: Designations of synthesized HAMCs

Sample designation	Composition
AL	EN AW 6061
AL1	EN AW 6061 + 5 w% Al ₂ O ₃ + 1 w% WSA
AL2	EN AW 6061 + 5 w% Al ₂ O ₃ + 2 w% WSA
AL3	EN AW 6061 + 5 w% Al ₂ O ₃ + 3 w% WSA

2.3 Characterization

A microstructural analysis, measurement of the hardness, tensile strength and elongation were used to characterize the tested hybrid composites.

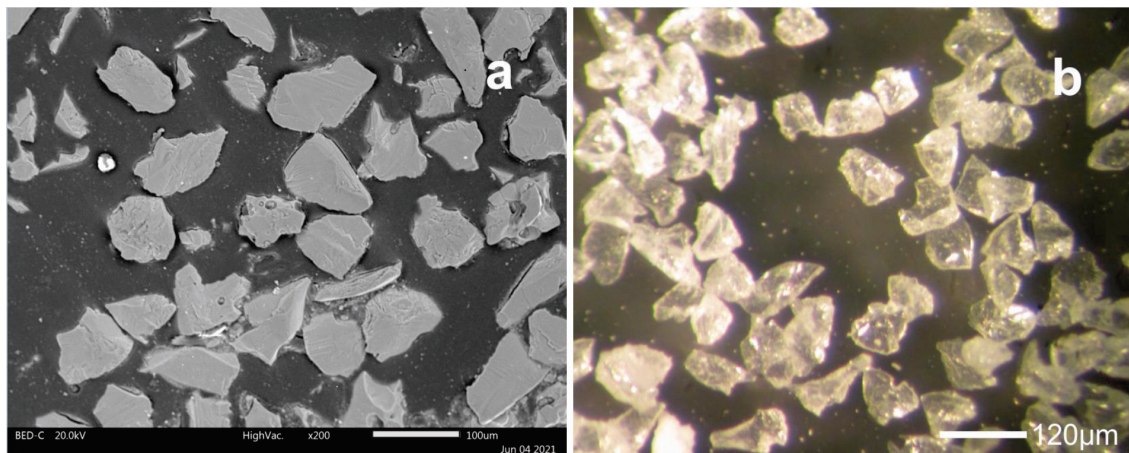


Figure 2: SEM and optical micrographs of Al₂O₃ particles

2.3.1 Microstructural analysis

A microscopic examination of the matrix alloy and HAMC was carried out using a Leica DM 2700M optical microscope (OM) and JEOL JSM IT 300LV scanning electron microscope (SEM) with an energy-dispersive spectrometer (EDS). Oxford Instruments X-Max EDS was used to determine the chemical composition of the reinforcing particles. The specimens for microstructural studies (10 × 10 × 5) mm were prepared by grinding and polishing. Wet grinding of the specimens was carried out with progressively finer SiC abrasive papers; polishing was done with a device for electrolytic polishing and etching was done with Metkon ELOPREP 102. Polishing and etching were performed with a standard solution for aluminum and its alloys (800 mL 95 % C₂H₅OH and 60 mL 65 % HClO₄).

2.3.2 Hardness test

Hardness was tested with standardized methods in accordance with ASTM E384.²⁷ Hardness was measured with a VEB Leipzig hardness tester, using the Vickers method on the samples taken from the middle of the ingots. A load of 10 kg (HV10) was used in a time interval of 10 s. Ten measurements were performed on each sample and the results were presented as the mean value of the measurements.

2.3.3 Tensile test

Tensile strength and elongation were measured on cast samples with dimensions of (8 × 8 × 150) mm. The measurements were performed on a universal Shimadzu machine with the Trapezium X software package, using standardized methods in accordance with ISO 6892-1.²⁸ The applied load speed was 100 mm/min. The measurements were performed three times for each sample and the average value was reported.

3 RESULTS AND DISCUSSION

The chemical composition of the used alloy, determined using a Niton XL3t spectrophotometer, is given in

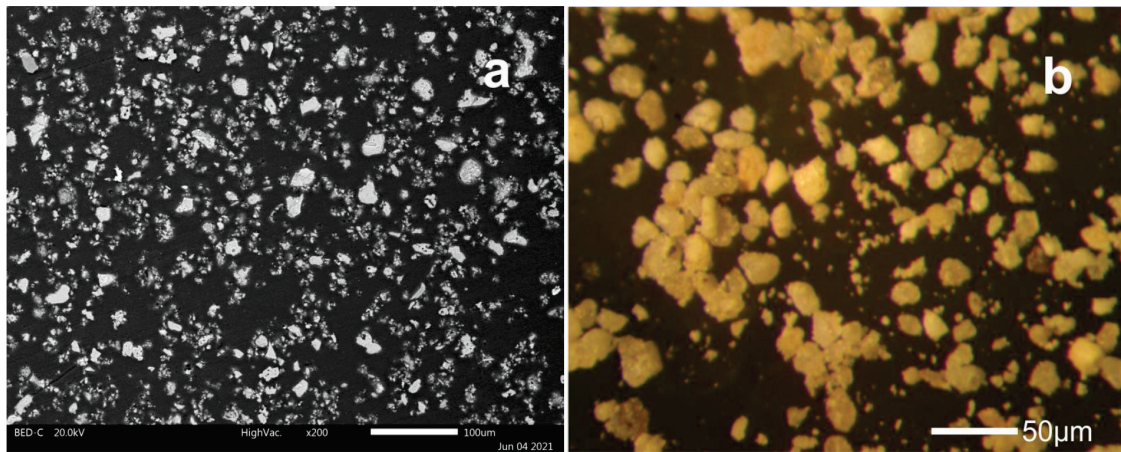


Figure 3: SEM and optical micrographs of WSA particles

Table 2. The weight contents of the elements in the alloy corresponded to the standard EN 573-3.²⁹

Table 2: Chemical composition of the EN AW 6061 alloy

Element	Al	Si	Cu	Fe	Zn	Mg	Mn	Ti	Cr	Ti + Zr
w/%	97.59	0.82	0.15	0.29	0.11	0.84	0.04	0.02	0.13	0.01

Figure 2 shows the appearance of Al₂O₃ particles, recorded by the SEM and optical microscope. It was noticed that the particles were mostly irregular in shape with sharp angles.

The ash, used in the production of HAMCs, was obtained by burning walnut shells. The walnut shells were first thoroughly washed with water and soaked in a 1 % formaldehyde solution to remove all impurities. The shells were then thoroughly washed with distilled water and dried in an oven for 24 h. Drying was followed by

burning in the furnace. The burning lasted for 60 min. The resulting ash was ground, sieved and annealed in an electric furnace at 700 °C for 3 h to remove all carbon. After cooling, sieving of the ash was performed in order to determine the particle-size distribution, which is given in Table 3.

Table 3: Particle-size distribution of WSA

Sieve size (µm)	%
> 100	0.20
≤ 100 > 90	35.40
≤ 90 > 80	29.70
≤ 80 > 70	23.00
≤ 70 > 60	8.80
≤ 60 > 50	0.40
≤ 50 > 45	2.1
≤ 45	0.4
	Σ 100

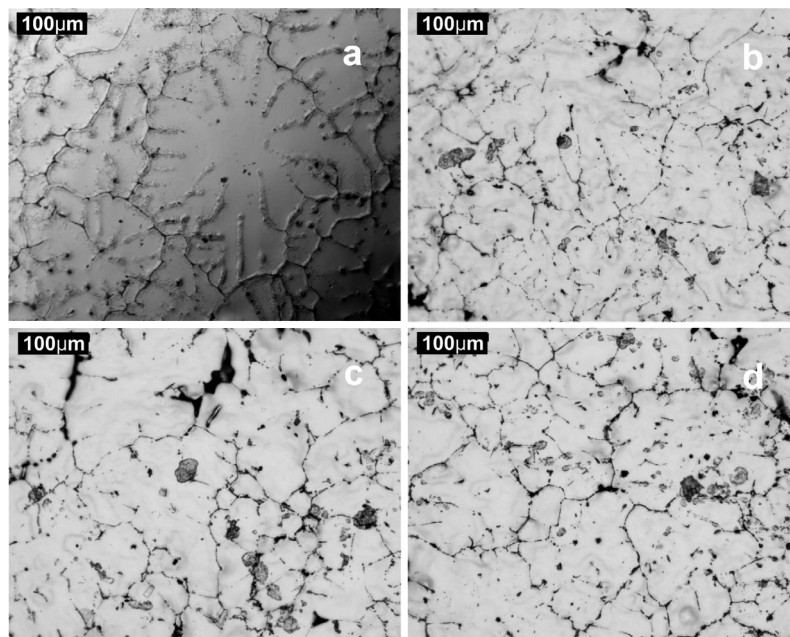


Figure 4: Optical micrographs of the metal matrix and synthesized HAMC samples: a) AL, b) AL1, c) AL2, d) AL3

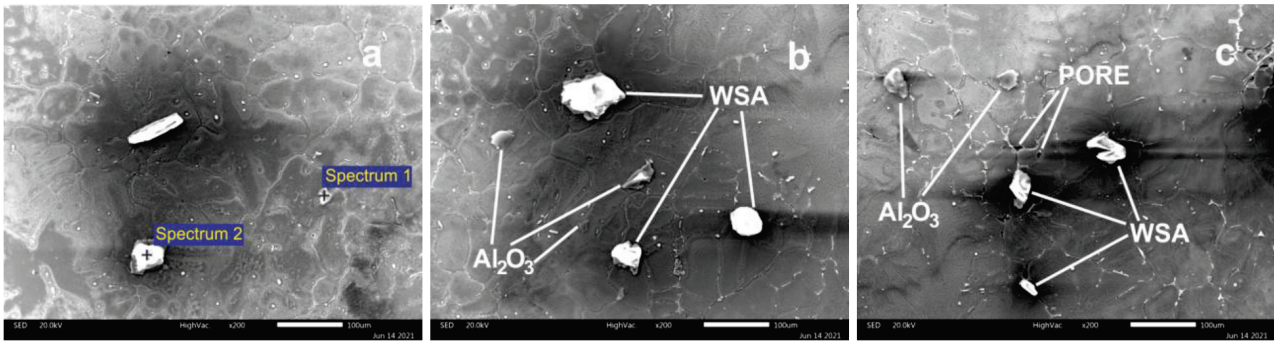


Figure 5: SEM micrographs of synthesized HAMC samples: a) AL1, b) AL2, c) AL3

Figure 3 shows the appearance of WSA particles, taken with the SEM and optical microscope. It was noticed that the particles were mostly irregular in shape and of different sizes.

Figures 4 and 5 represent optical and SEM micrographs, respectively. Figure 4a shows the microstructure of the metal matrix for comparison with the HAMC microstructures, shown in Figures 4b, 4c and 4d. The microstructural analysis showed a relatively uniform distribution of reinforcement particles in the composite materials, which was one of the conditions for increasing the value of the mechanical properties of the obtained composites. Similar conclusions were presented in the paper by Kumar et al.³⁰ The unreinforced EN AW 6061 alloy showed coarser grains in the microstructure than those in the HAMCs.

Figure 5 presents SEM micrographs of the tested HAMCs. A uniform distribution of the reinforcing particles without agglomeration was observed. EDS results of the chemical composition of the particles from Figure 5a are shown in Table 4. The EDS analysis showed the presence of Al₂O₃ and WSA particles.

Table 4: EDS results for the chemical composition Al₂O₃ x/% and WSA particles from Figure 5a

Spectrum	O	Al	Si	Ca	K	Mg	P	S
Spectrum 1	58.13	41.87						
Spectrum 2	46.40	5.93	3.58	14.73	23.88	3.45	0.60	1.43

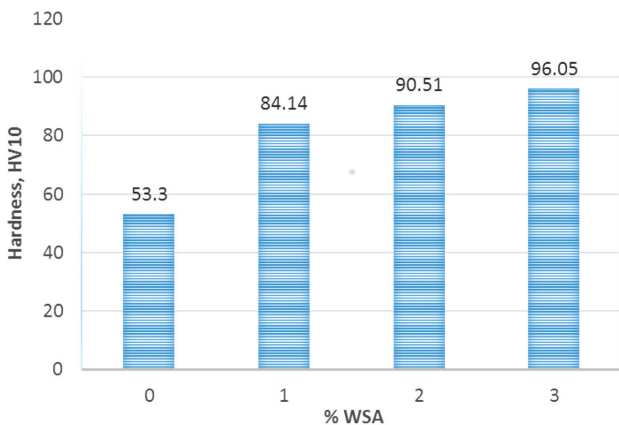


Figure 6: Variation in the hardness with the weight fraction of WSA

Figures 6, 7 and 8 show the results of the mechanical tests. Figure 6 shows the graphical dependence of the HAMC hardness on the WSA weight fraction. It can be observed that the HAMC hardness increased with the increasing weight fraction of WSA. It can also be concluded that the hardness of the unreinforced EN AW 6061 alloy was significantly lower compared to the HAMC.

Figure 7 shows the dependence of tensile strength on the weight fraction of WSA in HAMC. The tensile strength was observed to increase with the increasing WSA content. Compared to the non-reinforced EN AW 6061 alloy, the tensile strength of HAMC also increased.



Figure 7: Variation in the tensile strength with the weight fraction of WSA



Figure 8: Variation in the elongation with the weight fraction of WSA

Table 5: Changes in the mechanical characteristics for the HAMC in relation to the matrix EN AW 6061 alloy

Sample	Hardness (HV 10)	Improvement in hardness (%)	Tensile strength (N/mm ²)	Improvement in tensile strength (%)	Elongation (%)	Reduction in elongation (%)
AL	53.30	–	146	–	15.7	–
AL1	84.14	57.86	185	26.71	10.8	31.29
AL2	90.51	69.81	189	29.45	9.6	38.85
AL3	96.05	80.20	193	32.19	8.9	43.31

Figure 8 depicts the results of the HAMC elongation depending on the WSA content. The results showed that the HAMC elongation decreased compared to the unreinforced EN AW 6061 alloy. The HAMC elongation decreased with an increasing weight fraction of the reinforcing particles in the composites.

The obtained results for the HAMCs were compared with each other and with the results obtained for the unreinforced EN AW 6061 alloy. This way, the influence of the reinforcers on the values of the mechanical characteristics of the HAMC was determined. It was concluded that the hardness and tensile strength increased, while elongation decreased with an increase in the weight fraction of the reinforcements in the HAMC. It could also be concluded that the values of hardness and tensile strength were higher, and the values of elongation were lower compared to the values for the EN AW 6061 matrix alloy. The obtained results of hardness and tensile strength were in accordance with the research by Kulkarni et al.³¹ They investigated the influence of Al₂O₃ and fly ash as the reinforcing phases in the A356 aluminum alloy on the mechanical properties of the obtained composite. It was concluded that the joint action of the two reinforcements had a significant impact on the increase of the mechanical properties of the composite such as hardness, tensile strength and compressive strength in relation to the unreinforced A356 alloy.

Table 5 presents the percentage of improvement in the hardness and tensile strength and percentage of reduction in the elongation for the HAMC relative to the EN AW 6061 matrix alloy.

The HAMC hardness was 84.14 HV10 for AL1, 90.51 HV10 for AL2 and 96.05 HV10 for AL3. It was 57.86 % higher for AL1 HAMC compared to the unreinforced EN AW 6061 alloy, 69.81 % for AL2 HAMC, and 80.20 % for AL3 HAMC.

The HAMC tensile strength values were 185 N/mm² for AL1, 198 N/mm² for AL2 and 193 N/mm² for AL3, while the value for the non-reinforced EN AW 6061 alloy was 146 N/mm². Compared to the non-reinforced alloy, the values of the HAMCs were higher by 26.71 % for AL1, 29.45 % for AL2 and 32.19 % for AL3.

The increase in the hardness and tensile strength most likely occurred due to the actions of several mechanisms described in the literature, i.e., the obstructed movement of dislocations and load transfer from the matrix alloy to the reinforcement.³² The EN AW 6061 alloy and reinforcing particles had different coefficients of

thermal expansion. Therefore, during the crystallization of the composite, residual stresses were created, causing a disturbed movement of dislocations and their accumulation, which was a condition for the increase in the hardness.³³ When it comes to tensile strength, the interaction of the reinforcing particles and dislocation limited the crack protection under the action of the tensile load. A significant increase in the hardness and tensile strength of the HAMC was also a consequence of a decrease in the crystal grain size, strong bond between the matrix and amplifier, uniform particle distribution and low level of porosity.^{34,35} The increase in the values of mechanical properties can be explained with the transfer of load from the metal matrix to the reinforcing particles.³¹ The results obtained during this investigation were in agreement with the results found in the literature.^{14,15,36,37} The higher the weight fraction of the WSA, the greater was the effect of the described factors that caused the increase in mechanical properties.

The elongation for the unreinforced AL alloy was 15.7 %; it was 10.8 % for AL1, 9.6 % for AL2 and 8.9 % for AL3. Compared to the unreinforced EN AW 6061 alloy, the elongation decreased by 31.29 % for AL1, by 38.85 % for AL2 and by 43.31 % for AL3 HAMC. The decrease in the elongation with the increasing weight fraction of WSA in the HAMC occurred due to the increase in the brittleness of the material, which was in accordance with the research found in the literature.³⁸

4 CONCLUSIONS

The influence of Al₂O₃ and walnut-shell ash as additional reinforcements in an HAMC produced with stir casting was analyzed. It was concluded that an HAMC with an AW EN 6061 matrix reinforced with Al₂O₃ particles and walnut-shell ash can be successfully synthesized with the applied method. Properly selected parameters of the stir-casting procedure provided a uniform distribution of the reinforcements over the entire volume of the obtained HAMC, as shown with the metallographic analysis. A uniform distribution of reinforcements is an important factor influencing the increase in the hardness and tensile strength of the HAMC. The metallographic analysis also showed that the addition of reinforcements had a significant role in reducing the crystal grain size, which significantly influenced the strengthening of the HAMC. An increase in the weight fraction of reinforcement caused an increase in the values of hardness and

tensile strength and a decrease in the value of elongation in relation to the AW EN 6061 matrix alloy.

The use of WSA in the production of HAMCs turns agro-waste into an industrial asset. This way, the need for the storage of this waste is reduced, which, from the point of view of ecology, solves an important issue. WSA is a promising material used as an additional reinforcing phase in the production of HAMCs.

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