Effect of ZrO₂ quantity on mechanical properties of ZrO₂reinforced aluminum composites produced by the vacuum infiltration technique

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ABSTRACT: This study aims to demonstrate the effect of ZrO_2 quantity on the Al 2024-based ZrO_2 -reinforced composite materials produced by using the vacuum infiltration technique, which is reported relatively less often in the literature. ZrO_2 was used as the reinforcing element with ratios of 5%, 10%, 15%, and 20%. Following the production process, the density of the composite materials was measured, and their microstructures were investigated under the optical microscope and scanning electron microscope (SEM). The study also dealt with the determination of the mechanical properties of the produced composite materials. To this end, hardness measurements were done and cross-breaking strength tests, as well as abrasive wear tests, were conducted. The microstructure analysis revealed that the ZrO_2 additive element was partially homogeneously distributed in the composite structure and the wetting between Al 2024 and ZrO_2 was successful. Increasing the ZrO_2 amounts gave rise to higher density, hardness and wear resistance values. However, cross-breaking strength decreased. To sum up, the results of this study revealed that the ZrO_2 reinforcement improved the mechanical properties of Al 2024.

KEYWORDS: Aluminum composite; Microstructure; Mechanical property; Vacuum infiltration; ZrO,

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RESUMEN: Efecto de la cantidad de ZrO_2 sobre las propiedades mecánicas de los compuestos de aluminio reforzados con ZrO_2 producidos por la técnica de infiltración al vacío. Este estudio tiene como objetivo demostrar el efecto de la cantidad de ZrO_2 en los materiales compuestos reforzados con ZrO_2 basados en la aleación de aluminio 2024 obtenidos mediante la técnica de infiltración al vacío, con escasa información en la bibliografía. Se utilizó el ZrO_2 como elemento de refuerzo en proporciones de 5%, 10%, 15% y 20%. Después del proceso de fabricación, se midió la densidad de los materiales compuestos y se investigó su microestructura mediante microscopía óptica y microscopía electrónica de barrido (SEM). El estudio también se ocupó de la determinación de las propiedades mecánicas de los materiales compuestos obtenidos. Para ello, se realizaron mediciones de dureza y pruebas de resistencia a la rotura cruzada, así como pruebas de desgaste mediante abrasivo. El análisis de microestructura reveló que el elemento aditivo ZrO_2 se distribuyó parcialmente de manera homogénea en la estructura compuesta y que la humectación entre Al 2024 y ZrO_2 fue adecuada. El aumento de las cantidades de ZrO_2 dio lugar a valores más altos de densidad, dureza y resistencia al desgaste. Sin embargo, la resistencia a la rotura cruzada disminuyó. En resumen, los resultados de este estudio revelaron que el refuerzo de ZrO_2 mejoró las propiedades mecánicas de la aleación Al 2024.

PALABRAS CLAVE: Compuesto de aluminio; Infiltración al vacío; ZrO2; Microestructura; Propiedades mecánicas

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1. INTRODUCTION

There have been significant developments in materials used for engineering applications in recent years. Traditional engineering materials are not suitable for many applications as they are unmodified monolithic materials, while composite materials are useful in meeting the requirements of such applications. Composite materials differ from traditional engineering materials. Much research focuses on metal matrix composites (MMCs) today due to the advantages they provide in scientific and technical engineering applications (Madhusudhan et al., 2017). Almost 69% of the MMCs used annually for industrial purposes are Aluminum Metal Matrix composites (AlMMCs) (Idusuyi et al., 2019). Aluminum-based metal matrix composites are manufactured by various techniques such as liquid metallurgy, powder metallurgy, diffusion bonding, and spray forming. The liquid metallurgy techniques are more economical and more suitable for mass production than the powder metallurgy method (Aruna et al., 2018). The liquid metallurgy method consists of such techniques as hot pressing, stir casting, pressure casting, centrifugal casting, pressure infiltration, non-pressure infiltration, and vacuum infiltration. Vacuum infiltration is a new liquid phase technique used in the production of composite materials, which is relatively less reported in the literature. Ceramic based materials such as SiC, Al₂O₃, TiC, B₄C, MgO, TiB₂, ZrO₂ are used as reinforcing elements in the production of AlM-MCs. ZrO₂ (Zirconia), with its high strength capacity and excellent mechanical and wear properties at high temperatures, is one of the cheapest and most available (Udayashankar and Ramamurthy, 2018). Zirconium oxide (ZrO_2) is highly used in fuel cell technology, catalyst or catalyst support, protective coating for optical mirrors and filters, ceramic biomaterial production and thermal barrier coating (Şimşek, 2019). There are studies on the aluminum matrix-ZrO, particle-reinforced composites in the literature. Some of these studies employ the mixing casting technique, while others use the powder metallurgy technique (Abdizadeh and Baghchesara, 2013; Karthikevan and Jinu, 2015a; Ramachandra et al., 2015; Prasad and Rao, 2016; Rao et al., 2017; Radhika and Venkata Priyanka, 2017).

In this study, the vacuum infiltration technique was used in the production of Al 2024 composite materials reinforced with 5%, 10%, 15% and 20% of ZrO_2 ratios. Certain mechanical properties of the composites such as microstructure, hardness, fracture strength, and wear behavior were investigated.

2. MATERIALS AND METHODS

In the production of composite materials, ZrO_2 and Al 2024 powders sized $\leq 105 \mu m$ were used in composite compacts in the infiltration tubes while the alloy Al 2024 was employed as the molten matrix material. Table 1 shows the technical properties of Al 2024 and the reinforcing element ZrO_2 .

In vacuum infiltration tests, Al 2024 was used as molten matrix material and ZrO_2 powders sized \leq 105 µm as the reinforcing element. The following steps were followed in the calculation of the reinforcement-volume ratios (R-V) for the reinforcing element and matrix material to be filled into the infiltration tube (Eq. 1).

$$R-V \text{ ratio} = V_{\text{reinforcement}} / V_{\text{composite}}$$
(1)

In other words, the ratio of the actual weight of the reinforcing powders in the infiltration tube to the theoretical weight gives the R-V ratio. Al 2024 and ZrO_2 powders with 5% wt, 10% wt, 15% wt, 20% wt ratios were mixed in the rotary drum mixer for 120 min at 300 rpm. Composite mixtures were calculated according to (Eq. 2).

$$W_{c} = W_{r} \cdot V_{r} + W_{m} \cdot V_{m}$$
(2)

Where (W_c) is the weight of the composite, (W_r) is reinforcement weight, (V_r) is reinforcement volume, (W_m) is matrix weight, (V_m) is matrix volume. Then, the mixed Al 2024 and ZrO₂ powders were filled into steel infiltration tubes and compressed. The stainless filter elements were placed at the top and bottom of the composite compacts compressed in infiltration tubes. With the filters installed, Al 2024 and ZrO₂ powders in the tube were prevented from mixing into the molten metal and leaking into the vacuum line during the vacuum process. Finally, the infiltration process was initiated by immersing the test tubes into the molten Al 2024 in 750 +/-10 °C. The

	Table 1	Technical	properties of	f ZrO,	and Al 2024
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ZrO ₂									
$ZrO_2\%$	SiO ₂ %	TiO ₂ %	$Fe_2O_3\%$	Density	Hardness	Tensile Strength	Melting point		
99.5	0.10	0.007	0.002	5.0-6.15 g.cm ⁻³	1350 HV	711 MPa	2823 °C		
Al 2024									
Al %	Cu %	Mg %	Mn %	Density	Hardness	Tensile Strength	Melting point		
93.5	4.4	1.5	0.6	2.77 g.cm ⁻³	55 HB	186 MPa	638 °C		

tubes were preheated at 500 °C before immersion. This process has been done in order to remove the moisture and oxidized structures in the composite mixture and to better wet the reinforcing particles of the matrix material. Additionally, pure magnesium weighing 0.5% was added to molten aluminum to aid wetting. Infiltration procedures, which lasted for 10 min, were carried out at 650 mm Hg. The test setup used in the vacuum infiltration process is shown in Fig. 1.

The manufactured composite materials were removed from the steel test tubes to prepare the test samples. Composite materials measuring 20 mm in diameter and 60 mm in length were removed from the test tubes. Then the composite materials were machined to the sample size required for each test. First, images of the composite materials were taken under an optical microscope. Then, density values were measured according to Archimedes Principle in order to determine the density and porosity of the composite structure. Density measurements were repeated three times on each composite sample. Arithmetic means of the values found were calculated. Finally, the tests to determine such mechanical properties of the composite materials as hardness, cross-breaking strength, and abrasive wear were conducted. While the Brinell Method was employed for hardness measurements, the three-point bending technique for cross-breaking tests, pin-on-disk method for wear tests were used. The hardness measurements were made from 5 different regions of composite samples. The arithmetic average of the obtained values are calculated and transferred to the graphics. Cross-breaking tests were performed on three samples of composite in each reinforcement volume ratio. The arithmetic average of the calculated values are calculated and transferred to the graphics. The wear tests were carried out with 1200 mesh, 600 mesh, and 280 mesh grain-sized abrasives. The tests were conducted under 10 N, 20 N and 40 N loads, with a sliding speed of $0.5 \text{ m} \cdot \text{s}^{-1}$ and a wear distance of 40 meters. The data obtained from the tests were evaluated in graphs. The wear test set up is shown in Fig. 2.

3. RESULTS

3.1. Evaluation of microstructures

Optical microscope images of vacuum-infiltrated Al 2024 composites reinforced with 5%, 10%, 15%, and 20% of ZrO₂ ratios are given in Fig. 3.

The microstructure images in Fig. 3 shows the distribution of ZrO₂ reinforcement particles in the composite structure. The images suggest that the increase in the ZrO₂ reinforcement ratio improved the homogeneity in the reinforcement distribution. However, increasing the ZrO, ratio caused agglomeration of the reinforcing element. Similar results were reported in several studies in which ZrO₂-reinforced composites were produced by the mixing casting method (Jebaraj and Chennakesava Reddy, 2000; Hajizamani and Baharvandi, 2011; Ramachandra et al., 2015; Udayashankar and Ramamurthy, 2018). It appears that the displacement of the ZrO₂ particles during the vacuum infiltration process gives way to agglomeration in certain regions in the molten Al 2024 matrix material. Figure 3, displaying the microstructure of composite material reinforced with 20% ZrO2, suggests that melting the matrix material in the infiltration tube caused the ZrO₂ particles to move towards the top of the tube in the vacuum direction.

It is frequently reported in the literature that sufficient wetting between the matrix and reinforcing particles in such composite structures would not occur. However, this study, in which the vacuum infiltration method was used, indicated that the wetting between the matrix material and the



FIGURE 1. (a) Vacuum infiltration test tube interior detail and (b) infiltration test setup.

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FIGURE 2. (a) Abrasive wear test setup and (b) test detail.



FIGURE 3. Microstructures of ZrO₂-reinforced Al 2024 composites.

reinforcement was sufficient and there was no significant porosity in the composite structure. Addition of the 0.5% of magnesium to the molten aluminum during the infiltration process might also have contributed to wetting. The pre-heating process before immersion of the infiltration tubes in the molten metal could also have affected the wetting positively. A similar result is reported in a study in the literature (Hemanth, 2011). Therefore, it can be concluded that the parameters for the infiltration process used in this study (750 °C temperature of the molten metal matrix, 10 min of infiltration time and 650 mm Hg of vacuum value) were suitable. A major problem encountered in the production of such particle-reinforced aluminum composites is the formation of the porous composite structure. It is reported in the literature that the porosity increases with the increase of the reinforcing particles in the composite structure (Harish et al., 2016). The vacuum infiltration technique used in this study seems to have led to a more successful result in matrix-reinforcement compatibility than other production methods. Figure 3 (20% ZrO₂-enlarged image) shows more clearly the interface of the ZrO₂ reinforcing element and the Al 2024 matrix material. This microstructure images indicate that there is no porous structure between the aluminum matrix (Al 2024) and the ZrO₂ reinforcing particle and the bonding of the two phases is good. This suggests that this good wetting will increase the mechanical strength of the composite material, which also suggests that the vacuum infiltration technique used in the production of composites in this study is successful.

3.2. Effect of ZrO₂ ratio on the density

Figure 4 shows the density values according to Archimedes Principle of Al 2024 composites reinforced with 5%, 10%, 15% and 20% of ZrO, produced by vacuum infiltration. Figure 4 reveals that the density gradually increases due to the increase in the ZrO₂ reinforcement ratio. The increase in density can be attributed to the density of the reinforcing element, which is higher than the density of the matrix material. Similar results are reported in the literature (Ravi Kumar et al., 2018; Govindan and Gowthami, 2019; Parveen et al., 2019). However, the increase in density was not as high as the increase in the ZrO₂ reinforcement ratio. The increase in the ZrO₂ reinforcement ratio caused reinforcement agglomeration in the composite structure. Porous regions caused by the agglomeration in the composite led to a decrease in the density value. Theoretical densities were calculated mathematically by considering the matrix and reinforcement material densities. It is reported in the literature that the density values measured in different studies are slightly lower than the theoretical density values (Govindan et al., 2017).

3.3. Effect of ZrO₂ ratio on the hardness

Figure 5 shows the hardness values of the vacuum-infiltrated Al 2024 composites reinforced with 5 %, 10 %, 15 %, and 20 % of ZrO₂. Figure 5 reveals that the hardness value gradually increases due to the increase in the ZrO₂ reinforcement ratio. Similar results are reported in the literature (Karthikeyan and Jinu, 2015b; Madhusudhan *et al.*, 2016; Madhusudhan *et al.*, 2017; Rao *et al.*, 2017; Mirjavadi *et al.*, 2017; Pandiyarajan *et al.*, 2017; Aruna *et al.*, 2018). The major reason for this increase in the hardness is the hard phase ZrO₂ reinforcement particles in the composite structure. It can be stated that good wetting between the matrix material Al 2024 of the composite and the ZrO₂ reinforcing particles increases the mechanical strength of the structure as well as



FIGURE 4. Densities of ZrO₂-reinforced Al 2024 composites

the hardness. The 55 HB hardness value of pure Al 2024 alloy without ZrO, reinforcement gradually increased depending on the ZrO₂ reinforcement ratio. The highest hardness value was found to be 71.7 HB in the composite material with 20% of ZrO, reinforcement, which increased the hardness of the Al 2024 alloy by 30%. Another study in the literature reported that the increase of hard reinforcement particles, in addition to increasing the hardness of the composites, caused an increase in the formation of dislocation, which prevents plastic deformation. It can be stated that the fluidity of the molten matrix, the density of the reinforcing particles, the rate of solidification and the distribution of the reinforcing particles are the main factors affecting the hardness of the composites (Ravi et al., 2018). As a result, it was observed that there was a significant increase in the hardness values of ZrO₂ reinforced composites compared to pure Al 2024 alloy.

3.4. Effect of ZrO_2 ratio on the cross-breaking strength

Figure 6 shows the cross-breaking strength values of the vacuum-infiltrated Al 2024 composites reinforced with 5%, 10%, 15% and 20% of ZrO, obtained from the three-point bending tests. Figure 6 reveals that an increase in the ZrO₂ ratio leads to lower breaking strength and lower breaking force. The reason for this might be that the hard phase ZrO₂ reinforcing particles increased the brittleness of the composite structure by reducing its ductility. Similar results were reported in a study in the literature (Hemanth, 2011). The ZrO, reinforcement particles also might have caused the notch effect in the composite structure. The sharp-cornered ZrO₂ reinforcing particles caused the notch effect that initiated the breaking during the bending test. These notches eased the breaking. It is generally thought that the increase in hardness causes a decrease in breaking strength. Hardness means reduced flexibility, which naturally causes direct breakings of the composite materials during the three-point bend-



FIGURE 5. Hardness of ZrO₂-reinforced Al 2024 composites



FIGURE 6. Cross-breaking values of ZrO_2 -reinforced Al 2024 composites.

ing test. Therefore, higher hardness increased the brittleness of the composite structure and reduced the breaking strength. This generally occurs in the bending tests of ceramic-based particle-reinforced aluminum matrix composites. Figure 7 shows the SEM and BSE (Back-Scattered Electron microscopy) images of the broken surfaces in detail for the evaluation of the breaking behavior of composite materials.

Figure 7 shows the effect of the brittle breaking mechanism on the broken surfaces. The ZrO_2 reinforcing particles increased the hardness of the composite structure as well as its brittleness. Figure 7a reflects the SEM images taken from the broken sample surface and the BSE images of the same surface. The BES images reflect the ZrO₂ particles in the

matrix more clearly. The BSE images indicate that partial agglomeration occurred with the increase in the ZrO₂ ratio, causing a porous structure. During cross-breaking tests, the mechanical strength of the composite structure might have decreased and this might have facilitated the breaking in areas where agglomeration and pores occurred. There are studies in the literature reporting similar results (Chong et al., 1993; Pul, 2019). It was previously stated that the ZrO₂ reinforcing element increased the mechanical strength of the composite structure under normal conditions. Fig. 7f also gives an enlarged view showing the wetting on the interface of Al 2024 and ZrO, particles better. The image shows that wetting is good and a strong bond is formed between the matrix and reinforcement. However, as the ZrO₂ ratio increases in the composite structure, agglomeration occurs, leading to some porosity. It is also known that the angular and amorphous structure of ZrO₂ particles creates a notch effect. Therefore, the increase of the ZrO₂ ratio in the composite structure helped the breaking factors be more effective. Lowered breaking strength is a common result of increasing the reinforcing element ratios in the composite structure. A similar result has been reported in the literature (Chong et al., 1993).

The images in Fig. 7 (b,c,d,e) also reveal that the breaking of the composite samples did not deform the ZrO_2 particles in the structure so badly. The breakings generally occurred within the Al 2024 matrix material and the ZrO₂ particles remained



FIGURE 7. Broken surface SEM images of (a) 0%, (b) 5%, (c) 10%, (d) 15%, (e) 20% and (f) ZrO₂-reinforced Al 2024 composites and their wetting interface detail.

embedded in the Al 2024 matrix material. Pores expected to have been caused by the breaking of ZrO_2 particles were not available, which suggests that the bond between the matrix and reinforcement was strong due to successful wetting. All in all, the results suggest that the most important parameter affecting the cross-breaking test was the amount of reinforcing elements in the composite structure.

3.5. Effect of ZrO₂ ratio on the wear

Figure 8 shows the wear losses obtained from the abrasive wear tests of the vacuum-infiltrated Al 2024 composites reinforced with 5%, 10%, 15%, and 20% of ZrO₂ using the pin-on-disc method. Figure 8 shows that the wear resistance of the composite material increased while the wear losses decreased due to the increase in the ZrO, reinforcement ratio in the composite structure. The highest wear losses occurred at 40 N with the highest abrasion load. The findings also suggest that the abrasive paper was influential on the wear loss The highest wear losses in all composite samples occurred in tests with 280 mesh abrasive paper. Abrasive Al2O3 particles behaved as a multiple cutting tool. Therefore, the highest material loss was caused by 280 mesh abrasive with the largest particle size.

I already mentioned in the evaluation of Fig. 5 that the structure was getting harder with the increase of the ZrO, reinforcement ratio. The increase in the hardness in the composite material was the major effect on the reduction of material losses in

abrasive wear tests. In addition, the slippery ZrO, reinforcing particles helped the composite test samples slide more easily on the abrasive paper, which caused a slight reduction in the friction coefficient. Therefore, the increase in the ZrO₂ reinforcement ratio in the composite structure led to gradual decrease in the wear losses. Similar results are reported in the studies in the literature (Ramachandra et al., 2015; Karthikeyan and Jinu, 2015a; Karthikeyan and Jinu, 2016; Madhusudhan et al., 2016; Pandiyarajan et al., 2017; Veeresh Kumar et al., 2019). In another study in the literature, it was stated that the oxide formed at the matrix-reinforcing interface plays an important role in reducing both the friction coefficient and the amount of wear (Karthikeyan and Jinu, 2015b). It is possible to relate the wear behavior of composite materials with the hardness of the composite structure. According to the Archard equation, the abrasion resistance of the composites is directly related to their hardness (Eq. 3).

$$Q = KWL / H \tag{3}$$

where Q is the total volume of wear, K is a dimensionless constant, W is the total normal load, L is the sliding distance, and H is the hardness of the softest contacting surfaces. In addition, the slippery ZrO_2 reinforcing particles helped the composite test samples slide more easily on the abrasive paper, which caused a slight reduction in the friction coefficient. Therefore, the increase in the ZrO_2 reinforcement ratio in the composite structure led to gradual decrease in the wear losses.



FIGURE 8. Wear loss values of ZrO2-reinforced Al 2024 composites.

Similar results are mentioned in the studies in the literature (Ramachandra et al., 2015; Madhusudhan et al., 2016; Karthikeyan and Jinu, 2016; Pandiyarajan et al., 2017; Veeresh Kumar et al., 2019). In another study in the literature, it was stated that the oxide formed at the matrix-reinforcing interface plays an important role in reducing both the friction coefficient and the amount of wear (Karthikeyan and Jinu, 2015b). As a result of good wetting between matrix and reinforcing element ZrO₂, the strength of the composite structure, as well as the wear resistance, increased. In a study in the literature, it was stated that the wettability and interfacial strength, microhardness value and friction coefficient of the reinforcement in the matrix were related to the wear property of the metal matrix composite (Veeresh Kumar et al., 2011).

There was an increase in the wear loss with the increase of the applied load, which is an expected result. However, with the applied load increasing from 10 N to 20 N, the wear losses also increased on average by 2 times, while the load increased from 20 N to 40 N, the wear losses increased by 2.5 times on average. Although the load applied was doubled each time, the wear losses more than doubled. With the increase of the load to 40 N, the temperature of the composite sample surface in contact with the abrasive paper increases more. The increasing temperature decreases the hardness of the composite structure, making it ductile and less resistant to wear. The increase in temperature on the wear surface might have helped to break the ZrO₂ particles from the structure by weakening the binding in the matrix-reinforcing interface. In this case, the ZrO₂ particles removed from the composite structure might have shown an abrasive effect like abrasive grains and increased the wear losses. Therefore, wear losses under 40 N load were more higher.

In a study in the literature, it is stated that the wear mechanism changes to the load and the loss of material at low loads occurs by the rolling of the abrasive, and at high loads it is caused by the shearing effect. Again, the same authors determined that the increase in the applied load decreased the surface hardening (Singh et al., 2006). In another study, it was found that wear with increasing load decreased proportionally. It has been stated that wear at low loads occurs mainly through the nucleation and increase of microcracks. In the case of high load, it is stated that the abrasion depth is higher and the metallic matrix is plastically deformed (Sawla and Das, 2004). One of the important parameters in wear tests is abrasive paper dimensions. Fig. 9 shows the graphs created by abrasive size and ZrO₂ reinforcement rate.

Given the particle size of the abrasive papers, the highest wear losses occurred in tests with 280 mesh abrasives. When the abrasive grain sizes are converted to microns, 1200 mesh corresponds to 12 μ m, 600 mesh corresponds to 19 μ m and 280 mesh corresponds to 52 μ m. Therefore, the amount of the material removed by 280 mesh abrasive paper was directly proportional to the grain size. Figure 9 clearly shows this increase in the amount of wear loss. Therefore, the fact that the abrasive particle size has a significant effect on the amount of wear loss is clear. In a study in the literature, it was reported that the composite material had more resistance to



FIGURE 9. Wear loss values of (a) 0%, (b) 5%, (c) 10%, (c) 15%, (e) 20% ZrO,-reinforced Al2024 composites by abrasive paper size

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Figure 10. Worn surface images of (a) 0%, (b) 5%, (c) 10%, (d) 15%, (e) 20% ZrO,-reinforced Al 2024 composites.

20 μ m and 35 μ m abrasive grain size than to 100 μ m (Modi, 2001). In order to study the wear behavior of composites in more detail, the SEM images of the worn surfaces are given collectively in Fig. 10. The images were taken from tests where 40 N load and 280 mesh abrasive papers were used with the highest wear losses.

The microstructure images in Fig. 10 suggest that the abrasive wear mechanism is effective in all reinforcement ratios. The Al₂O₃ abrasives used in the tests could be the major factor in the wear loss. The hard phase ZrO, particles, which were removed from the composite structure, might also have contributed to the wear loss by friction onto the surface. A similar result was reported in the literature (Yilmaz and Buytoz, 2011). The SEM images in Fig. 10 displays the wear cavities formed by the abrasive particles and the ZrO₂ particles remaining on the surface. The SEM images also show the noticeable Al 2024 matrix smearing on the wear surfaces due to the increase in the ZrO, ratio. The ZrO, reinforcement material has a slippery structure as said above. Therefore, the increase in the ZrO₂ ratio caused an increase in the slipperiness on the surface and the Al 2024 matrix material in the soft phase was smeared due to the friction. As explained before, increases in the ZrO₂ reinforcement ratio cause agglomerations. The reinforcing elements in the agglomerated regions might have been removed from the composite structure in larger masses during the abrasion tests and formed large pores on the surface. Most probably, these pores were filled and smeared with the Al 2024 matrix material. Therefore, in the composite samples with higher ZrO₂ ratios, lower Al 2024 losses led to lower wear losses. The SEM images in Fig.

11 show porous regions formed by agglomerated ZrO, particles and removed ZrO, particles.

The surface images also indicate that the cavities are irregular, that is, some are larger or deeper than others. The size of ZrO, particles in the composite structure ranged between 20 µm and 105 µm. The ZrO, particles removed from the composite structure by breaking must have been rather small, as particles with smaller surfaces have lower binding between the matrix-reinforcing interface. Therefore, the fine cavities on the worn surfaces must have been formed by smaller ZrO₂ particles. Different studies in the literature report that abrasive wear behavior is associated with the shape, distribution, and condition of the reinforcing element in the composite material (Berger et al., 1999; Candan et al., 2001). Looking at the graphs in Fig. 8, it is understood that the most abrasion is in pure 100% Al 2024 samples. When the worn surface image of 100% Al 2024 sample in Fig. 10a is examined, a surface with continuous scratches appears. This is a result of the micro-cutting mechanism in abrasive wear. These continuous lines indicate that the greatest wear is in the pure 100% Al 2024 sample. This supports the graphic data in Fig. 8. In a study in the literature, it is stated that, in soft materials, in abrasive wear, besides micro cutting, abrasion occurs due to high deformation, and there are continuous and wide scratches. If a general evaluation is made, it can be thought that with the increase in the reinforcement elements in the hard phase in the composite structure, the wear resistance will increase and the hard materials will wear less (Hasirci and Gül, 2010). However, the wear properties of composites should not only be associated with hardness but should



Figure 11. (a) Reinforcement aggregation and (b) porous area, in Al 2024 composites ZrO,-reinforced

be handled from different directions (Saheb *et al.*, 2001). In addition, the physical conditions of the matrix and reinforcing elements in the composite structure, technical properties, heat treatments and production parameters of the composite have been demonstrated by some studies that affect the wear (Sun *et al.*, 1999; Lasa and Rodriguez-Ibabe *et al.*, 2002; Prasad and Rao, 2016).

4. CONCLUSIONS

Having evaluated the microstructures and mechanical properties of the vacuum infiltrated Al 2024 composites reinforced with 5%, 10%, 15% and 20% of ZrO_2 , I can summarize the results as follows:

- Al 2024² matrix composites reinforced with 5%, 10%, 15% and 20 of % ZrO₂ have been successfully produced by vacuum infiltration technique with the determined manufacturing parameters
- ZrO₂ reinforcement distribution in the composite structure was partially homogeneous. Strong bond formation occurred as a result of the good wetting between the matrix-reinforcement.
- The ZrO₂ reinforcing elements increased the density, hardness and wear resistance of the composite structure while reducing the breaking strength.
- Breakings generally occurred within the matrix material as a brittle fracture mechanism.
- It was found that the ZrO₂ reinforcement in the composite structure generally increased the mechanical strength values except for the breaking strength.
- It was found that the wear resistance of the composite material could be associated not only with hardness but also the amount and size of the matrix phase and reinforcing elements in the composite structure. The abrasive grain size was also found to be effective on the wear loss.
- The results obtained from this study suggest

that the strong bonding formed by good wetting between the matrix and the reinforcing phases increases the mechanical properties of the composite.

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REFERENCES

- Abdizadeh, H., Baghchesara, M.A. (2013). Investigation Into The Mechanical Properties And Fracture Behavior Of A356 Aluminum Alloy-Based ZrO₂-Particle-Reinforced Metal-Matrix Composites. *Mech. Compos. Mater.* 49 (5), 571-576. https://doi.org/10.1007/s11029-013-9373-z.
- Aruna, K., Diwakar, K., Bhargav Kumar, K. (2018). Development and Characterization of AL6061-ZrO2 Reinforced Metal Matrix Composites. *IJARCSSE 8* (4), 270-275.
- Berger, M., Wiklund, U., Eriksson, M., Engqvist, H., Jacobson, S. (1999). The multilayer effect in abrasion-optimising the combination of hard and tough phases. *Surf. Coat. Technol.* 116-119 (1138–1144). https://doi.org/10.1016/S0257-8972(99)00151-6.
- Candan, E., Ahlatci, H., Çimenoğlu, H. (2001). Abrasive wear behaviour of Al-SiC composites produced by pressure infiltration technique. *Wear* 247 (2), 133-138. https://doi. org/10.1016/S0043-1648(00)00499-3.
- Chong, S.Y., Atkinson, H.V., Jones, H. (1993). Effect of ceramic particle size, melt superheat, impurites and alloy conditions on threshold pressure for infiltration on SiC powder compacts by aluminium-based melts. *Mat. Sci. Eng. A* 173 (1-2), 233-237. https://doi.org/10.1016/0921-5093(93)90221-Y.
- Govindan, K., Raghuvaran, J.G.T., Pandian, V. (2017). Weldability Study of LM25/ZrO₂ Composites by Using Friction Welding. *Revista Matéria* 22 (3), e11855. https://doi. org/10.1590/S1517-707620170003.0189.
- Govindan, K., Gowthami, T.R.J. (2019). Mechanical Properties and Metallurgical Characterization of LM25/ZrO. Composites Fabricated by Stir Casting Method. *Revista Matéria* 24 (3), e12439. https://doi.org/10.1590/S1517-707620190003.0753.
- Hajizamani, M., Baharvandi, H. (2011). Fabrication and Studying the Mechanical Properties of A356 Alloy Reinforced with A1₂O₃-10% Vol. ZrO₂ Nanoparticles through Stir Casting. AMPC 1 (2), 26-30. https://doi.org/10.4236/ ampc.2011.12005.

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- Harish, B.R., Shaik Dawood, A.K., Nagabhushan, A., Pimpale, S., Raja Reddy, C.V. (2016). Comparative Study On Indi-vidual And Combined Effects Of Zirconium Dioxide And Graphite Reinforcements On Mechanical Properties Of Al 6061 Composites. *Int. J. Res. Eng. Technol.* 5 (4), 412-416. https://ijret.org/volumes/2016v05/i16/IJRET20160516090. pdf
- Hasirci, H., Gül, F. (2010). Investigation of abrasive wear beha-viours in B4C /Al composites depending on reinforcement volume fraction. SDU Int. Technol. Sci. 2 (1), 15-21. https:// acikerisim.isparta.edu.tr/xmlui/handle/123456789/333
- Hemanth, J. (2011). Fracture behavior of cryogenically solidified aluminum alloy reinforced with Nano-ZrO, metal matrix composites (CNMMCs). JCEM 2 (8), 110-121. https://academicjournals.org/article/article1379497880_Hemanth.pdf.
- Idusuyi, N., Olayinka, John, I. (2019). Dry sliding wear characteristics of aluminium metal matrix composites: A brief overview. J. Mater. Res. Technol. 8 (3), 3338-3346. https:// doi.org/10.1016/j.jmrt.2019.04.017. Jebaraj, P.M., Chennakesava Reddy, A. (2000). Simulation and
- Microstructural Characterization of Zirconia/AA7020 Alloy Particle-Reinforced Metal Matrix Composites. 2nd National Conference on Materials and Manufacturing Pro*cesses.* Hyderabad, India, pp. 134-140. http://jntuhceh.org/ web/tutorials/faculty/1144_ZrO-7020.pdf. Karthikeyan, G., Jinu, G.R. (2015a). Experimental investigation
- on mechanical and wear Behaviour of Aluminium LM6/ ZrO₂ composites fabricated by stir casting method. *Journal*
- of the Balkan Tribological Association 21 (3), 539-556. Karthikeyan, G., Jinu, G.R. (2015b). Dry Sliding Wear Beha-viour of Stir Cast LM 25/ZrO, Metal Matrix Composites. *Trans. Famena* 39 (4), 89-98. https://hrcak.srce.hr/index.
- php?show=clanak&id_clanak_jezik=223897. Karthikeyan G., Jinu, G.R. (2016). Dry sliding wear behavior optimization of stir cast LM6 /ZrO, composites by response surface methodology analysis. Trans. Can. Soc. Mech. Eng. 40 (3), 351-369. https://doi.org/10.1139/tcsme-2016-0026. Lasa, L., Rodriguez-Ibabe, J.M. (2002). Effect of composition
- and processing route on the wear behaviour of Al-Si alloys. Scripta Mater. 46 (6), 477-481. https://doi.org/10.1016/ S1359-6462(02)00020-9.
- Madhusudhan, M., Vikram, K.V., Mahesha, K., Chandra Babu, C.K. (2016). Evaluation Of Microstructure And Mechanical Properties Of As Cast Aluminium Alloy 7075 and ZRO2 Dispersed Metal Matrix Composites. International Journal of Mechanical and Production Engineering. Special Issue, 93-99
- Madhusudhan, M., Naveen, G.J., Mahesha, K. (2017). Mechanical Characterization of AA7068-ZrO, reinforced Metal Matrix Composites. *Mater. Today Proc.* 4 (2), 3122-3130. https://doi.org/10.1016/j.matpr.2017.02.196.
- Mirjavadi, S.S., Alipour, M., Hamouda, A.M.S., Matin, A., Kord, S., Afshari, B.M., Koppad, P.G. (2017). Effect of multi-pass friction stir processing on the micros-tructure, mechanical and wear properties of AA5083/ ZrO₂ nanocomposites. *J. Alloys Compd.* 726, 1262-1273. https://doi.org/10.1016/j.jallcom.2017.08.084.
- Modi, O.P. (2001). Two-body abrasion of a cast Al–Cu (2014 Al) alloy-Al₂O₃ particle composite: influence of heat treatment and abrasion test parameters. *Wear* 248 (1-2), 100-111. ht-tps://doi.org/10.1016/S0043-1648(00)00534-2.
- Andiyarajan, R., Maran, P., Marimuthu S., Ganesh, K.C. (2017). Mechanical and tribological behavior of the metal matrix composite AA6061/ZrO₂/C. J. Mech. Sci. Technol. 31 (10), 4711-4717. https://doi.org/10.1007/s12206-017-0917-3.
 Parveen, A., Chauhan, N.R., Suhaib, M. (2019). Mechanical and Tribological Bahayiour of ALZPO. Composition: A Review.
- Tribological Behaviour of Al-ZrO, Composites: A Review.

In: Advances in Engineering Design. Lecture Notes in Mechanical Engineering. Prasad A., Gupta S., Tyagi R. (Eds). Springer, Šingapore. https://doi.org/10.1007/978-981-13-6469-3_20. Prasad, C., Rao, K.M. (2016). A Study on Effect of Mechani-

- cal Properties Of Al-ZrO, Composite by Liquid Routing. International Journal of Science Engineering and Advance Technology 4 (4), 189-192.
- Pul, M. (2019). Effect of sintering on mechanical property of SiC/B C reinforced aluminum. Mater. Res. Express. 6 (1), 016541. https://doi.org/10.1088/2053-1591/aacee1.
- Radhika, N., Venkata Priyanka, M.L. (2017). Investigation of Adhesive Wear Behaviour Of Zirconia Reinforced Aluminium Metal Matrix Composite. Int. J. Eng. Sci. Technol. 12 (6), 1685-1696.
- Ramachandra, M., Abhishek, A., Siddeshwar, P., Bharathi, V. (2015). Hardness and Wear Resistance of ZrO, Nano Particle Reinforced Al Nanocomposites Produced by Powder Metallurgy. Proc. Mat. Sci. 10, 212-219. https://doi.or-g/10.1016/j.mspro.2015.06.043.
- Rao, P.C.S., Prasad, T., Harish, M. (2017). Evaluation of Me-chanical Properties of Al 7075-ZrO₂ Metal Matrix Composite by using Stir Casting Technique. International Jour-nal of Scientific Research Engineering & Technology 6 (4), 377-381
- Ravi Kumar, K., Pridhar, T., Sree Balaji, V.S. (2018). Mechanical properties and characterization of zirconium oxide (ZrO2) and coconut shell ash(CSA) reinforced aluminium (Al 6082) matrix hybrid composite. J. Alloys Compd. 765, 171-179. https://doi.org/10.1016/j.jallcom.2018.06.177.
- Saheb, N., Laoui, T., Daud, A.R., Harun, M., Radiman, S., Ya-haya, R. (2001). Influence of Ti addition on wear proper-ties of Al–Si eutectic alloys. *Wear* 249 (8), 656–662. https:// doi.org/10.1016/S0043-1648(01)00687-1.
- Sawla, S., Das, S. (2004). Combined effect of reinforcement and heat treatment on the two body abrasive wear of aluminum alloy and aluminum particle composites. Wear 257 (5-6),555-561. https://doi.org/10.1016/j.wear.2004.02.001.
- Singh, M., Mondal, D.P., Das, S. (2006). Abrasive wear response of aluminium alloy-sillimanite particle reinforced compo-site under low stress condition. *Mat. Sci. Eng. A* 419 (1-2), 59-68. https://doi.org/10.1016/j.msea.2005.11.056.
- Sun, Y., Baydoğan, M., Çimenoğlu, H. (1999). The effect of deformation before ageing on the wear resistance of an aluminum alloy. Mater. Lett. 38 (3), 221-226. https://doi. org/10.1016/S0167-577X(98)00162-1.
- Şimşek, İ. (2019). Investigation of Wear Performance of Different Amounts ZrO, Reinforced Al-2Gr Matrix Composite Materials Produced by Mechanical Alloying Method. *El*-Cezerî J. Sci. Eng. 6 (3), 594-605. https://doi.org/10.31202/ ecjse.560741.
- Udayashankar, S., Ramamurthy, V.S. (2018). Development and Characterization of Al6061-Zirconium Dioxide Reinforced Particulate Composites. Int. J. Res. Eng. Technol. 7 (12), 128-132
- Veeresh Kumar, G.B., Rao, C.S.P., Selvaraj, N. (2011). Mecha-nical and Tribological Behavior of Particulate Reinforced
- nical and Tribological Behavior of Particulate Reinforced Aluminum Metal Matrix Composites -a review. JMMCE 10 (1), 59-91. https://doi.org/10.4236/jmmce.2011.101005. Veeresh Kumar, G.B., Pramod, R., Guna Sekhar, Ch., Pradeep Kumar, G., Bhanumurthy, T. (2019). Investigation of phy-sical, mechanical and tribological properties of Al6061– ZrO, nano-composites. *Heliyon* 5 (11), e02858. https://doi. org/10.1016/j.heliyon.2019.e02858. Yilmaz, O., Buytoz, S. (2011). Abrasive wear of Al₂O₃-reinfor-ced aluminium-based MMCs. *Compos. Sci. Technol.* 61 (16), 2381-2392. https://doi.org/10.1016/S0266-3538(01)00131-2.