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Production and characterization of AA2014-B_4C surface-modificated composite via the squeeze casting technique

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ABSTRACT: Metal matrix composite (MMCs) materials provide superiority to monolithic materials in various mechanical properties such as tensile, yield, abrasion resistance, impact resistance by adding reinforcements such as B_4C, SiC, Al_2O_3. While liquid metal processes offer an important advantage, such as low-cost production in high volumes, the heterogeneous clustering of reinforcements in the matrix and the formation of porosity in the area between the reinforcement and matrix pose a problem for composite production. The squeeze casting method stands out in composite production due to its low cost, suitability for mass production, allowing high reinforcement ratio, and ease of homogeneous distribution of reinforcements. In this study, a composite layer reinforced with B_4C was produced with a thickness of 1 and 2 mm on a substrate of aluminum 2014 wrought alloy using the squeeze casting method. The mechanical properties of the composite materials produced were characterized via tensile, wear, impact, and hardness tests, and were examined with the help of Scanning Electron Microscopy (SEM). It has been observed that the composite region contains 50 vol.% of B_4C reinforcement and the particles of reinforcement were homogeneously distributed into the matrix. All results of the tests mentioned above are better than those obtained in the monolithic 2014 aluminum alloy.

KEYWORDS: Aluminum matrix composites; B_4C; Metal matrix composites (MMCs); Squeeze casting; Surface modification

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RESUMEN: *Producción y caracterización de una capa superficial de material compuesto AA2014-B_4C mediante la técnica de fundición por compresión*. Los materiales compuestos de matriz metálica son superiores respecto a los materiales monolíticos en varias propiedades mecánicas, como el límite elástico, tensión máxima, resistencia a la abrasión o la resistencia al impacto al agregar refuerzos como B_4C, SiC, Al_2O_3. Si bien los procesos de metal líquido ofrecen una ventaja importante, como la producción de bajo coste en grandes volúmenes, el aglomeramiento heterogéneo del refuerzo en la matriz y la formación de porosidad en la zona entre el refuerzo y la matriz plantean un problema para la producción de materiales compuestos. El método de fundición por compresión se destaca en la producción de materiales compuestos debido a su bajo coste e idoneidad para la producción en masa, lo que permite utilizar una alta cantidad de refuerzo y conseguir una distribución homogénea del mismo fácilmente. En este estudio se elaboró una capa de material compuesto reforzado con B_4C de 1 y 2 mm de espesor sobre un sustrato de aleación de aluminio 2014 forjado mediante el método de fundición por compresión. Las propiedades mecánicas de los materiales compuestos producidos se caracterizaron mediante pruebas de tracción, desgaste, impacto y dureza, y se examinaron con la ayuda de microscopía electrónica de barrido (MEB). Se ha observado que la región compuesta contiene un 50% en volumen de refuerzo B_4C y las partículas de refuerzo se distribuyeron homogéneamente en la matriz. Todos los resultados de las pruebas mencionadas anteriormente son mejores que los obtenidos en la aleación de aluminio monolítico 2014.

PALABRAS CLAVE: B_4C; Fundición por presión; Materiales compuestos de matriz de aluminio; Materiales compuestos de matriz metálica (MMC); Modificación de superficial

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1. INTRODUCTION

Metal matrix composites (MMC's) are materials formed by combining two materials different from each other in terms of their physical and chemical properties - the soft metal matrix and the reinforcement phase, which is harder than the matrix (Chawla N. and Chawla K.K., 2006a). MMC's offer features such as high specific strength, wear resistance or/and damping capacity. In addition to its superior properties, the possibility of being designed according to the purpose, through the control of parameters such as reinforcement and matrix material selection, reinforcement ratio, reinforcement distribution, constitutes the most important elements that distinguish MMC materials from monolithic materials (Surappa, 2003; Miracle, 2005). The functional properties of MMCs allow the use of these materials in a wide variety of applications. Land transportation, aerospace, electronic/thermal applications, and various industrial applications constitute the main application areas of MMC's (Chawla N. and Chawla K.K., 2006b).

Boron carbide reinforced aluminum matrix composites have attracted the attention of researchers due to their properties such as low density, high wear resistance, and high strength. However, difficulties are encountered due to their high production cost, the segregation of reinforcements, and the low wettability of the reinforcement by the aluminum matrix (Kerti and Toptan, 2008; Shorowordi *et al.,* 2003).

In the design and manufacture of MMCs, wetting is a problem that must be overcome due to the high surface tension of liquid metals. The presence of a good interface bond is critical in terms of efficient load transfer from the matrix to the reinforcement and thus the performance of the composite material (Chawla N. and Chawla K.K., 2006a; Ibrahim *et al.,* 1991).

The wettability between the liquid metal and the solid ceramic particle can be improved by reducing the contact angle. Various methods have been developed in order to reduce the contact angle. These;

- As a result of coating the ceramic reinforcement surface with copper or nickel, the interface is converted from the ceramic-metal system to the metal-metal system,
- Reducing the surface tension by adding reactive elements such as Mg, Li, Ti, Zr, Ca to the matrix alloy,
- Preheating the ceramic reinforcements and forming different compounds (oxide etc.) on their surfaces,
- With a mechanical effect applied externally to the reinforcement and matrix (stirring, ultrasonic waves, applying pressure, etc.), it is possible to wet the reinforcement by the metal by exceeding the surface tension of the metal (Girot *et al.*, 1987; Oh *et al.*, 1989; Manning Jr. and Gurganus, 1969; Rohatgi, 1991; Occhionero *et al.*, 1999; Shorowordi *et al.*, 2003; Vijayaram *et al.*, 2006; Sseyed Reihani, 2006).

Squeeze casting is a casting method that is a combination of high-pressure casting, gravity casting, and forging. In the squeeze casting method, the application of pressure in the process from the mold filling to the completion of solidification prevents the formation of air gaps at the mold/metal interface and the casting structure becomes finer as a result of better heat transfer. The applied pressure allows the wetting angle between the reinforcement/liquid metal to increase, thus creating favorable conditions for the formation of interfacial reactions. In addition, the fact that the reinforcement and the matrix are not in contact at high temperatures for a long time prevents the occurrence of unwanted interface reactions. Besides being a simple and low-cost method suitable for mass production, the high mechanical properties it provides make the process among the most ideal methods for metal matrix composite production (Girot *et al.*, 1987; Vijayaram *et al.*, 2006, Sseyed Reihani, 2006; Sukumaran *et al.*, 2008; Suresh, 2013; Jayalakshmi and Gupta, 2015).

In this study, boron carbide reinforced aluminum matrix composite was produced using the squeeze casting method to obtain metal matrix composite having a homogenous surface. The novelty of this research is to procure a tough surface as well as a ductile matrix to overcome fracture due to instantaneous violent dynamic loads during service of the composites. The microstructural properties of materials containing composite layers of 1- and 2-mm thicknesses were revealed by SEM examinations. With the help of hardness, tensile, notch impact, and pin-on-disc wear tests, the mechanical properties of the material were characterized and compared with the monolithic aluminum sample produced by the same method.

2. MATERIALS AND METHODS

Aluminum wrought alloy, AA2014, the chemical composition of which is given in Table 1, was used as a matrix alloy.

Table 1. Chemical composition of the 2014 alloy

Cu	Si	Mn	Mg	Al
4.06	0.74	0.51	0.26	Bal.

Boron carbide used as reinforcement was supplied from ESK Ceramics GmbH and has 22-60 µm grain size and mixed in the shape, some round, and a fair amount of angular. Commercial purity aluminum powder with a grain size of less than 40 µm was used to create space between reinforcement particles.

In the grinder, boron carbide and aluminum powder are mixed mechanically in equal proportions by volume, then laid on the bottom surface of the steel mold (60x18x18 mm^3) which was preheated to 450 °C. The aluminum alloy melted in the electric furnace was poured into the mold at 750 °C and a pressure of 100 MPa was applied through a hydraulic press, enabling the molten metal to fill the spaces between the reinforcement particles. The pressure was applied until the molten metal completely solidified, after cooling the samples having 60 mm of length and 18x18 mm cross-section was removed from the mold. The process steps are shown schematically in Fig. 1.

The sections of the samples produced were metallographically prepared with the grid sand paper from 220 to 1200 mesh and were examined by SEM. Also, the reinforcement ratio of the composites were measured with the image analysis software, ImageJ. For the tensile and notch impact tests, the samples were brought to the appropriate geometry by machining, after that tensile and impact tests were carried out according to ASTM E8/E8M-21 (2021) and ASTM E23-18 ((2018), respectively. The wear test was carried out on a pin-on-disc wear test machine following the standart of ASTM G99-17 (2017). The wear test was carried out continuously for 1000 m at a speed of 0.1 $m{\cdot}s^{-1}$ under a 15 N constant load. A 10 mm diameter 100Cr6 steel ball with 847 HV hardness is used as the opposing surface. The hardness

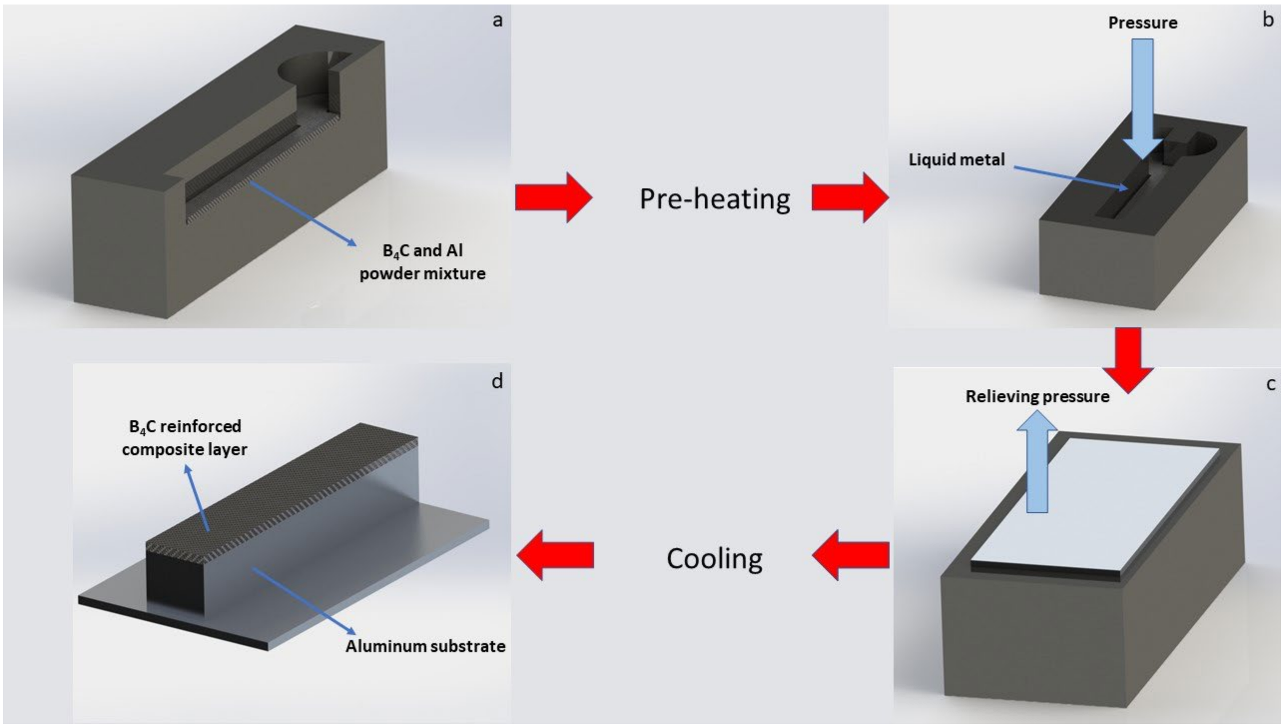

Figure 1. MMC production steps with squeeze casting.

of the composite and aluminum substrates were measured and compared using the Vickers method with a load of 1000 g and a dwell time of 10 seconds according to ASTM E92-17 (2017).

3. RESULTS AND DISCUSSION

3.1. Microstructural investigations and hardness measurement

In the first examination of the sample sections (Fig. 2), it was seen that the composite layer had a homogeneous thickness in the sample where the composite layer thickness was 1 mm, and some heterogeneous regions were detected in the 2 mm thick sample. Figure 3 shows the SEM view of the cross-section of the 1-mm thick composite material. It can be seen that the black-colored boron carbide particles are distributed homogeneously. The reinforcement ratio measurement made with the image analysis program of ImageJ on this image showed that the composite region contains 50 vol.% volume of B_4C.

As a result of the detailed examination of the boron carbide particles and the aluminum interface, no porosities that could negatively affect the mechanical properties of the material were found. In other studies, (Abou El-Khair, 2005; Shalaby *et al.*, 2016), it has been proven that the high pressure applied during solidification prevents the formation of porosity and provides a good bonding of the matrix and the reinforcement. The squeeze casting method improves the wettability between the reinforcement and the matrix (Shalaby *et al.*, 2016). As it can be

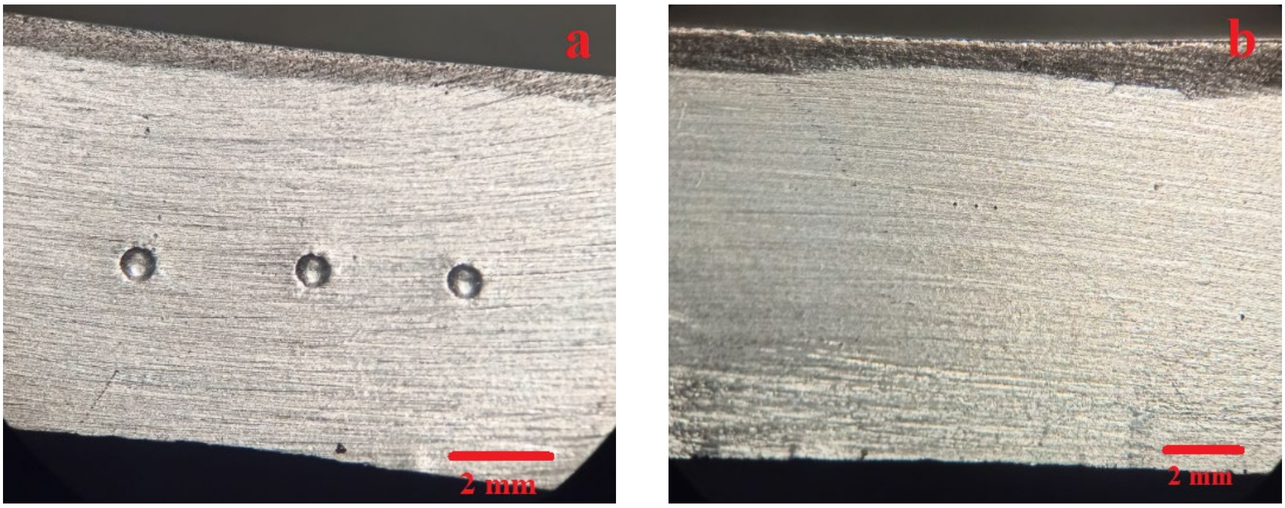

Figure 2. Stereomicroscope images of the cross-sections of the composite materials a) 1 mm, b) 2 mm.

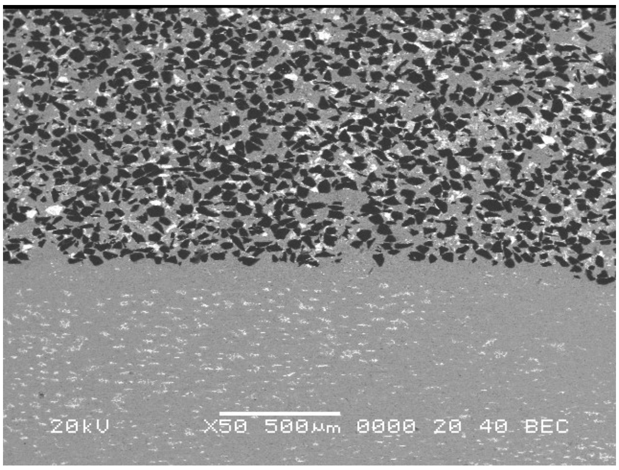

Figure 3. SEM image of the composite cross-section of 1-mm thick.

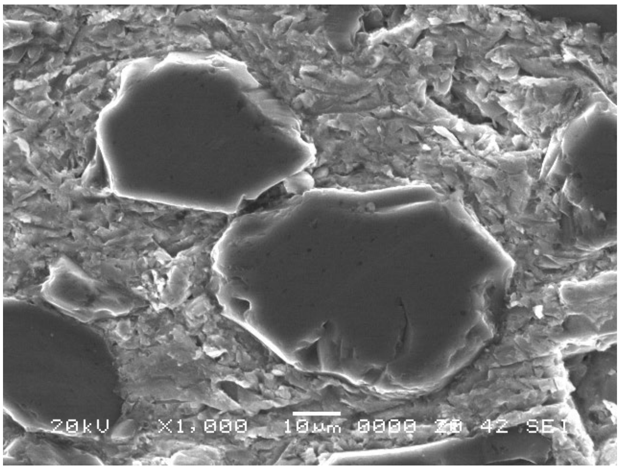

Figure 4. SEM image showing the boron carbide particle and the surrounding aluminum matrix of 1-mm thick specimen.

seen from Fig. 4 of 1-mm thick specimen, as a result of the increase in wetting, the B_4C particle is completely surrounded by the aluminum matrix, also the same image profile was obtained from the 2-mm thick specimen.

The measurement results of Vickers test are summarized in Table 2. While values of the matrix hardness of the reference sample and the sample with 1 mm thickness were close, it was observed that the matrix hardness of the 2 mm thick sample was higher. Furthermore, monolithic 1 mm specimen has a value of 90.97 HV_1, whereas composite of 1-mm has 275.56 HV_1, there is notably an improvement about three times. In other respects, the same scenario is carried out between a 2-mm specimen and its composite having nearly four times increase. In the study conducted by Sukumaran *et al.* (2008), it was observed that the amount of porosity decreased with the increase of compression pressure, and the hardness increased with the decrease of porosity. Based on this, it is thought that the increase in the hardness of the matrix is caused by the increase in compression pressure. Hard particles in the structure resist plastic deformation and increase the hardness of the structure and the material (Kalaiselvan *et al.*, 2011).

TABLE 2. Hardness values of the matrix and the composite layers

	Hardness (HV1)	
Sample	**Matrix (Al)**	**Composite (B_4C)**
Reference	88.42±0.50	-
1 mm	90.97±6.00	275.56±10.00
2 mm	97.80±8.00	324.75±4.00

3.2. Tensile test

The test results performed using a universal tensile testing machine are summarized in Fig. 5. According to the results, although there is no significant change in tensile strength, there is an increasing trend in the yield strength with 120 MPa of 1-mm and 140 MPa of 2-mm specimens. On the other hand, as expected, there is a gradual decrease in the elongation value from 16% to 13% and 7%, respectively. However, compared to similar studies in the literature (Sukumaran *et al.*, 2008), the decrease in elongation at fracture value has been observed to be more limited. As a result of the test, composite materials have less elongation compared to monolithic AA2014 aluminum alloy and the elongation values decrease as the thickness of the composite zone increases, proving that the composite region has less elongation value than the aluminum monolithic matrix.

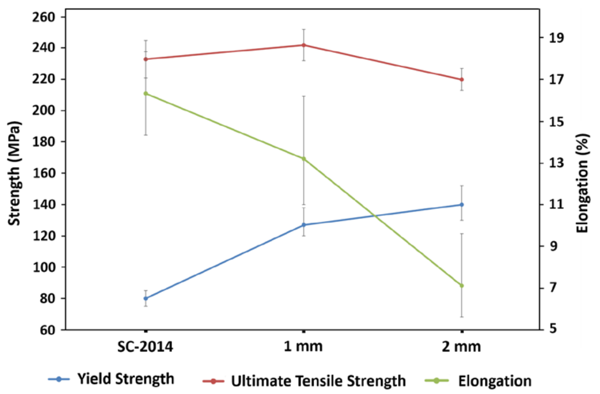

FIGURE 5. Tensile test properties of the composites compared to the AA2014 aluminum wrought alloy.

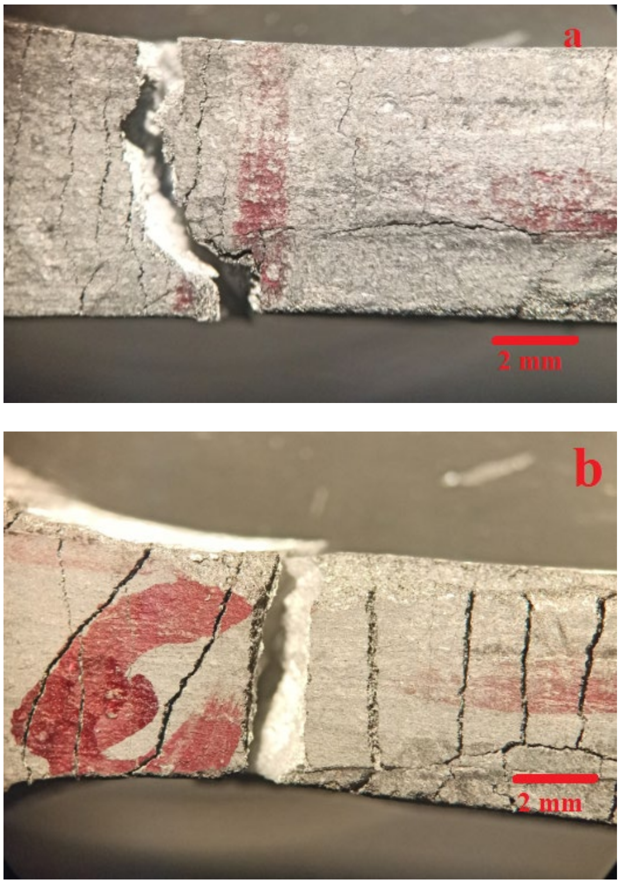

FIGURE 6. Cracks formed perpendicular to the tensile direction in the composite layer a) 1 mm, b) 2 mm.

In the visual inspection performed after the test, it was observed that these cracks propagation, seen in Fig. 6, are much severe in the 2-mm thick layer compared to the sample containing a 1-mm thick composite layer. It is thought that the reason for this situation is the lower elongation value of the com-

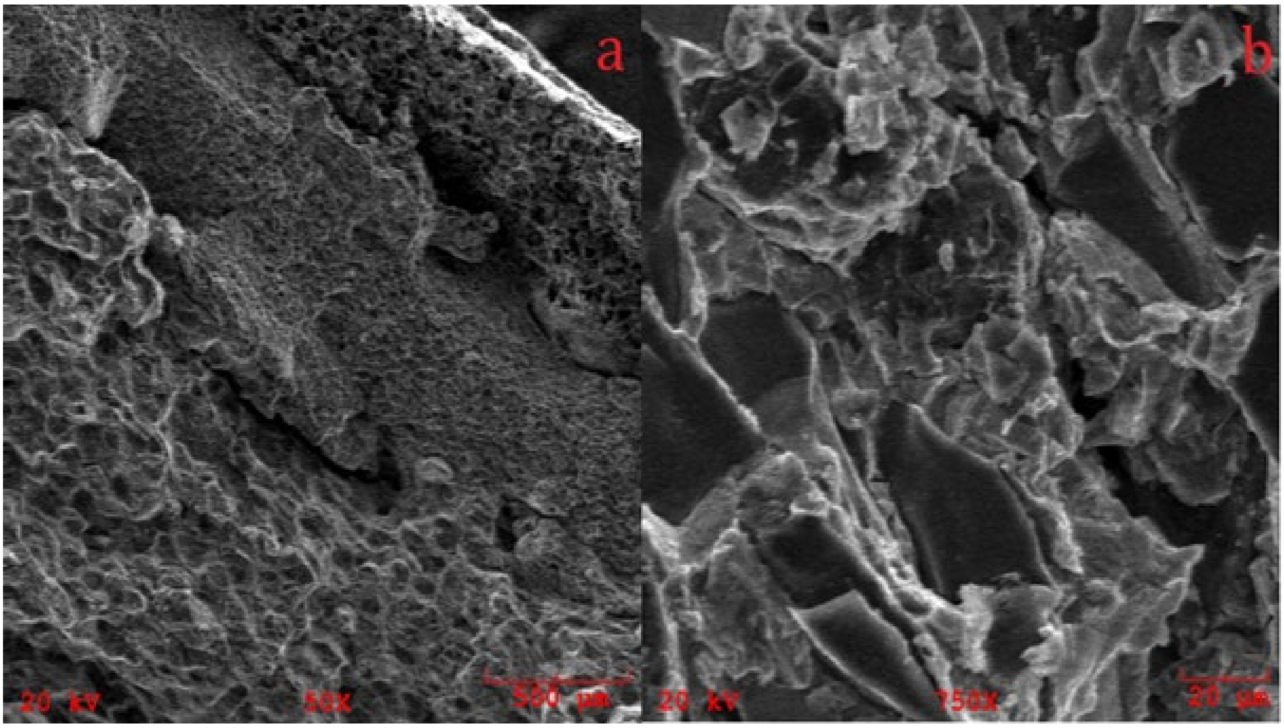

FIGURE 7. SEM images of the fracture characteristics of a) aluminium matrix and b) interface (red arrow) of the tensile specimen of 1-mm thick.

posite layer, and therefore, this layer is separated by cracking while the matrix continues to elongate. The decrease in the elongation ratio as the thickness of the composite layer increases supports this situation.

Figure 7 is the SEM image of the fracture surface of 1-mm thick specimen. The upper right corner of Fig. 7a consists of the composite region and the remaining areas are made of the aluminum matrix. As it can be seen in the image, the aluminum substrate area has been fractured by creating a dimpled structure. Therefore, it can be interpreted that the fracture in the matrix is ductile. This finding is also consistent with the elongation values obtained. In the examination performed at higher magnification in the composite region (Fig. 7b), it is seen that the breakage took place as a result of a different mechanism. It is understood that the fracture occurred as a result of the crack following the reinforcement/matrix interface in this region. It is seen that the reinforcement/matrix interface is separated as shown by the arrow in Fig. 7b. This is due to the lower strength of the reinforcement/matrix interface than the reinforcement, as is the case with most MMCs. The composite region showed less ductile behavior as the reinforcement particles prevented the dislocation movement. Therefore, the reinforced area was fractured by showing brittle fracture. In the studies of Ibrahim *et al.* (2014) and Ibrahim *et al.* (2015), it has been observed that the matrix alloy breaks with dimples similar to the one we see here. Such fractures occur as a result of the growth of micro-voids that begin to occur with the increase of strain in the material, and ultimately the crack follows this path (Zhang *et al.*, 2006).

3.3. Charpy impact test

The amount of energy absorbed by the samples before the fracture is shown in Fig. 8. The impact resistance of composite materials is much higher than that of the monolithic aluminum. In addition, with the increase in the thickness of the composite layer, an increase in the impact resistance has been observed. Ozden *et al.* (2007) have shown that hard ceramic particles added to the aluminum matrix reduce the impact resistance. As the reason for this situation, it has been suggested that as a result of the agglomeration of ceramic particles, it creates a suitable route for crack propagation. In this study, reinforcement particles, which are densely located in the composite area, prevented the dislocation movement and were effective in the absorption of the impact energy by the load transfer mechanism. Also, the formation of a strong interface bond between B_4C and aluminum helped to increase the impact resistance.

Similar to the fracture surface analysis results performed after the tensile test, it was observed in the examination that the fracture occurred following the reinforcement/matrix interface, but fractures also occurred in the reinforcement particles in some areas.

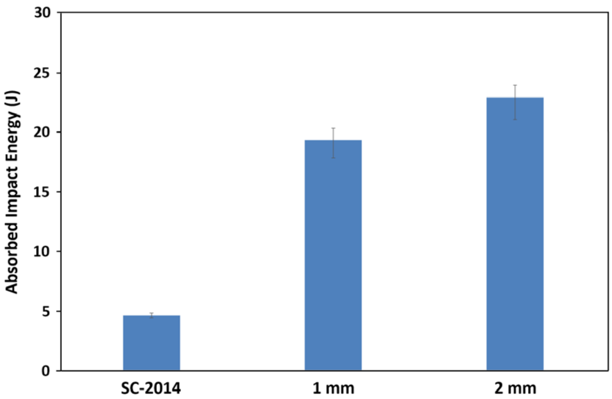

FIGURE 8. Total absorbed impact energies of the specimens.

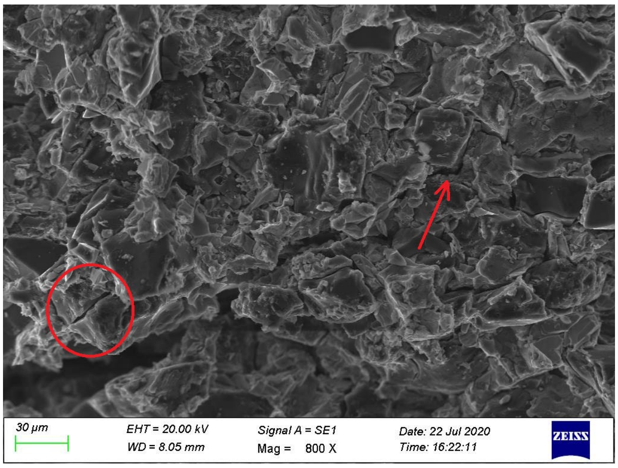

FIGURE 9. The fracture surfaces of the impact test of the 1-mm thick specimen (Cracks have been pinpointed with a red circle and a red arrow).

The fracture types occurred in both 1- and 2-mm thick specimens are nearly same, and are given as an example in Fig. 9. In the area indicated by the circle in the figure, fracture occurred in the reinforcement particle. Such a fracture occurs when the particle/matrix interface is stronger than the ceramic particle (Lu *et al.*, 1998). In the area pinpointed by the arrow, it was observed that the fracture followed the reinforcement/matrix interface. The presence of two different fracture modes indicates that the reinforcement/matrix interface does not possess the same strength throughout the composite. For that matter, some regions of the interface are even stronger than the reinforcement indicated by the fracture of the reinforcement.

3.4. Pin-on disk wear test

The pin-on-disc wear test was carried out following the ASTM G99-17 (2017) standard, and using the equations specified in the standard, volume losses and wear rates in both samples and abrasives were calculated. Since the wear occurring in the monolithic aluminum alloy occurs at a much higher rate compared to the opposite surface, the volume loss taking place here is calculated by Eq. 1. The opposite is the case with composite materials; the wear on the opposite surface is greater than the wear on the sample. For this reason, Eq. 2 and 3 are used to calculate the volume loss in the composites.

$$V = 2\pi R\left[r^2 \sin^{-1}\left(\frac{d}{2r}\right) - \left(\frac{d}{4}\right)(4r^2 - d^2)^{\frac{1}{2}}\right] \quad (1)$$

R: wear track radius; d: wear track width

$$V = \left(\frac{\pi h}{6}\right)\left[\frac{3d^2}{6} + h^2\right] \quad (2)$$

$$h = r - \left(r^2 - \frac{d^2}{4}\right)^{1/2} \quad (3)$$

d: wear scar diameter; r: pin end radius

Different material combinations and ambient conditions affect the wear rate and coefficient of friction, as shown by Andersson and Ylöstalo (1989) and Andersson (1992). Therefore, wear resistance is accepted as a function of the wear system. For this reason, the rate of wear on opposite surfaces is also important in the interpretation of the test.

The very high wear resistance of boron carbide has caused the wear of the composite material to remain at the lowest level and even the wear of the opposing surface. After calculating the abrasion volume losses, the wear rate was calculated in mm^3/Nm and shown comparatively in Fig. 10 and 11 for both pin and disc. Here the pin represents the steel ball, which is the opposing surface, and the disc represents the specimens subjected to the wear test.

Relatively soft aluminum has been easily abraded by the much harder steel surface. While the reference

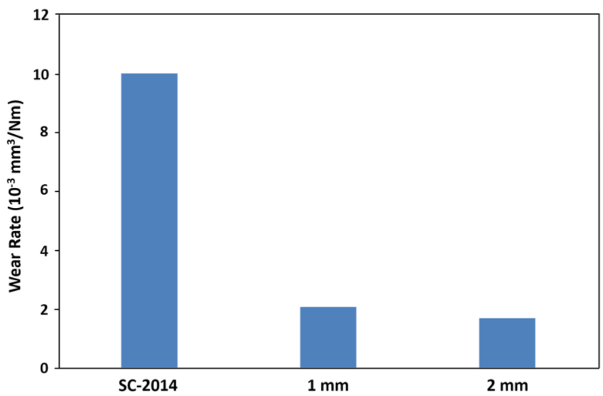

FIGURE 10. Wear rates of the specimens.

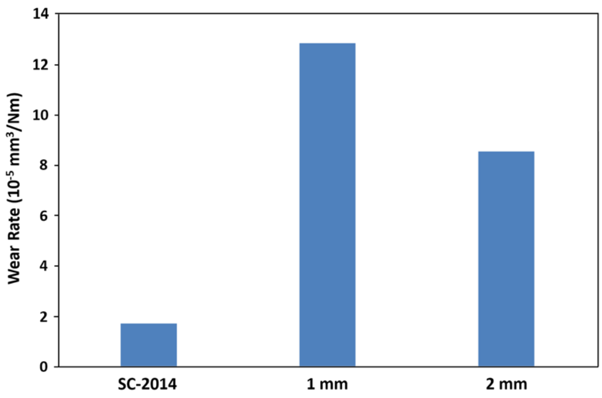

FIGURE 11. Wear rates of the opposing surfaces.

sample wears easily, it is evident from its wear rates that it causes almost no damage to the opposite surface. It is seen that the wear rates of the composite samples are quite low compared to the monolithic aluminum. When the wear rates of the opposing surfaces are examined, it is seen that the composite samples wear the balls at a much higher rate. It is thought that boron carbide particles with high hardness and wear resistance on the surface cause such a result by abrasively wearing the steel. Figure 12 shows the worn surface of 2-mm thick composite sample. Figure 12a shows the aluminum smeared on the surface as a result of severe plastic deformation. In Fig. 12b, the groove of an abrasive particle is seen in the area indicated by the red arrow. This particle is most likely a boron carbide particle that is taken out during the wear process.

In the early stages of the test, boron carbide particles protruding from the surface caused the steel ball to wear and thus increasing the contact surface. With increased surface area more particles interact with the opposing surface, causing a greater removal rate of the material from the ball surface. As the contact area increases, a second mechanism is activated. In this mechanism, the soft matrix undergoes plastic deformation and as a result, the metal-metal two surfaces are worn adhesively. Various studies have been made in the literature that describes the functioning of these mechanisms and their results are discussed (Gale and Tetemeier, 2004; Tang *et al.*, 2008).

4. CONCLUSIONS

- In the measurements made using the image analysis software, the reinforcement ratio was calculated to be 50 vol.%.
- The hardness values of the composites were measured as 275 HV_1 of 1 mm and 325 HV_1 of 2 mm specimens, having increased nearly three and four times, respectively in comparison with the monolithic reference specimen.
- Although the tensile strength did not change significantly, the values of yield strength were increased from 80 MPa to 140 MPa with a 50% increment.
- On the other hand, the rate of elongation decreased with the increase in the volume of the composite area. Also, it was observed that the matrix showed a ductile and the composite area showed a brittle fracture.
- Charpy impact test results showed that the amount of energy absorbed increased from 4 J to 23 J having a six-time improvement.
- In the SEM examination, it was observed that the fracture progressed following the reinforcement/matrix interface as well. However, some broken reinforcement particles were also detected.

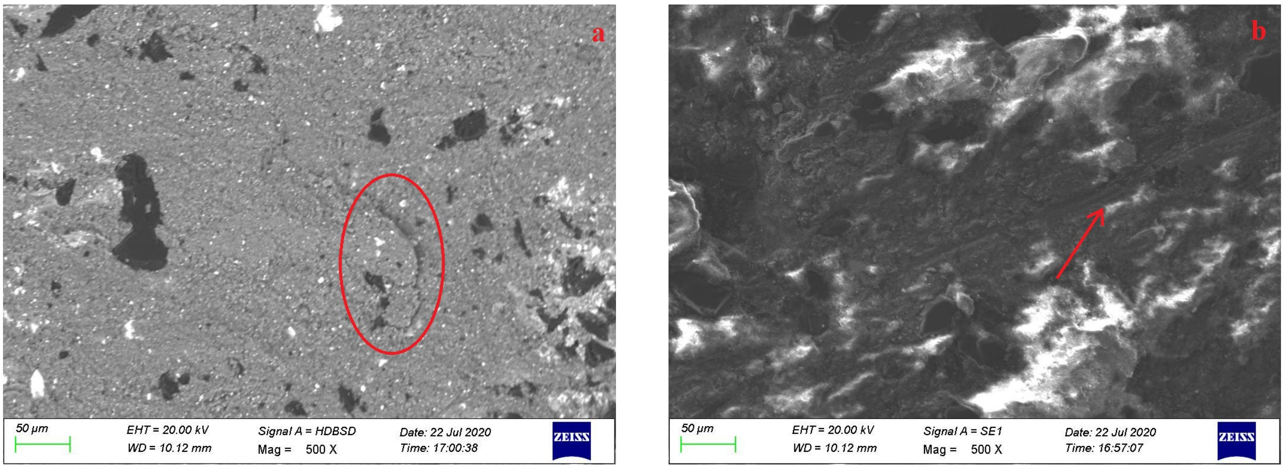

FIGURE 12. SEM image of the worn 2-mm thick composite surface a) plastic deformation of the metal matrix (highlighted by an eclipse), b) groove of an abrasive particle (pinpointed by a red arrow).

- The wear test has indicated that the composite specimens performed much better than the reference material. The rate of wear was calculated by taking into account both the volume loss in the sample and the volume loss on the opposite surface.
- With the SEM examinations of the worn surfaces, it has been concluded that the wear type of composite materials starts as abrasive and turns into adhesive wear.

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