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Tribological and Aging Behavior of Hybrid Al 7075 Composite Reinforced with B_4C, SiC, and TiB_2

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Abstract:

In this study, B_4C+SiC/B_4C +TiB_2/SiC+TiB_2 hybrid reinforcements were added to Al 7075 matrix via the powder metallurgy method. The powders were subjected to mechanical grinding for 240 min, as shown in the graphical abstract. The hybrid composite powder structure produced by the mechanical grinding was then placed in a vertical casting chamber and molten Al 7075 (main matrix) was added to obtain the samples. Samples were dissolved for 1 h at 480 ºC, and then aged at 120 ºC in 4-h increments (32 h in total). After the aging process, the wear behavior of the samples was investigated. In the study, FESEM images were examined for microstructural analysis, and hardness plots of the aged samples were created depending on the time after the solutioning treatment. The friction coefficient, volume loss, and worn surface images were investigated to determine the wear behavior of the hybrid structures. Results showed that the increased reinforcement rate and the reinforcement size and type directly affected the hardness and wear behavior. In the experiments, the highest hardness and wear resistance behavior were obtained in the hybrid sample of 3% B_4C + 3% SiC after 12 h of aging.

***Keywords**: Al 7075 hybrid composite; PM + casting technology; Aging; FESEM; Wear behavior.*

1. Introduction

Aluminum (Al) and its alloys are desireable materials due to their properties of resistance against corrosive environments, their long-term ability to maintain mechanical properties, and their suitable workability. They are used extensively in the automotive, aviation, chemistry, food processing, and marine sectors [1]. In the form of composite material, Al finds a more important application in the fields of technology where a high strength/weight ratio is desired [2]. For manufacturers, increasing the mechanical properties of the material and providing the advantage of lightness are especially sought after. Although Al and its alloys are quite diverse, the Al 7075 alloy is among the most preferred in the sector in terms of usage areas and densities. The Al 7075 alloy contains 5.6 % Zn, 2.5 % Mg, and 1.6 % Cu and is superior to other Al alloys in terms of hardness and strength values. It has a high solid solubility because of the Zn and Mg in its chemical composition. Thus, the mechanical strength properties of the material can be increased by applying precipitation hardening (T6) [3].

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Various production methods and processes are used with Al and its alloys. In the production of composites with a reinforced Al matrix, two different approaches can be used: the molten liquid method and the solid state method [4]. In the molten liquid method, the solid ceramic particles added to the molten Al metal are not homogeneously distributed, resulting in irregular distribution within the matrix structure. In the solid state production method, in composites produced by powder metallurgy (P/M), the matrix and reinforcing element powders are mixed in solid form, and composite powder metal is produced by pressing and sintering after achieving homogeneous distribution. In this process, the mechanical grinding of the powders contributes to a more balanced powder distribution and more particles are homogeneously distributed. High-strength products are produced with the optimum adjustment of the powders used in the composite production. This production is then achieved according to the selected parameters, such as mixing ratio, distribution shape, pressing pressure, and sintering stage (temperature, duration, etc.) [4, 5]. In the composite production process, the P/M method allows for easier and more controlled production of difficult-to-produce alloys compared to other manufacturing methods (casting, machining, chipless, etc.). Parts that are more complex and in mixed shapes can be produced more easily and cheaply using P/M, and these products are quite acceptably close to the desired dimensions [6]. With the P/M method, it is possible to form metal powders into the desired shapes and dimensions economically within a short time, which facilitates mass production [7]. The microstructure and mechanical properties of metal materials are improved by heat treatment. In particular, both hardness and strength values of Al and its alloys can be boosted by aging [8]. In the 7xxx series of Al alloys, after the solution process, quenching and aging can increase strength [9, 10]. The hardness and strength expected from the material provide the advantage of prolonging its service life. A machine is exposed to wear and friction due to the contact of its parts with one another during operation. In particular, specified heat treatments (T6, etc.) are applied to increase the mechanical properties and wear resistance of these parts. On machines, this wear can be defined as "wear and tear or decrease of the material from the part surfaces when the two surfaces are in contact with each other". There are a variety of wear mechanisms, including abrasive, adhesive, corrosive, erosive, and surface fatigue wear. The most common wear is abrasive wear [11, 12].

Many literature studies have been carried out with the aim of increasing wear resistance. In one of these studies, Arık produced a silicon mat-reinforced composite material with an Al matrix by applying the P/M production method. In his study, he showed that the homogeneous distribution of SiC in the matrix structure was acomplished by mechanical mixing and thus, its mechanical properties were improved [13]. In addition, Şimşek et al. investigated microstructural changes and their effect on the wear performance of Al 7075 alloy aged over five different periods and the effect of aging time on wear behavior were revealed. Within the scope of the study, the lowest weight loss was obtained in samples aged for 25 h, while the highest weight loss was in samples aged for 15 h. The surface wear investigations determined that abrasive and adhesive wear mechanisms had occured [14]. In their study using Al 7075 alloy produced via P/M, Yıldırım et al. showed that the microstructure and grain size of the $MgZn_2$ precipitates varied depending on the aging temperature and time following the T6 heat treatment. They observed that the precipitate size increased in direct proportion to the increase in aging temperature and time. The largest precipitate was measured at 130 ^oC and the highest hardness values after 24 h at 120 ^oC [2]. Furthermore, Panigrahi and Jayaganthan investigated the mechanical and microstructural changes that occurred in the Al 7075 alloy structure by rolling it at liquid nitrogen temperature and at room temperature. After subjecting the alloy to different rolling conditions and durations, they found that aging at 100 ^oC for 45 h yielded optimum microstructure and strength values [15].

In this study, unlike other studies [2, 13-15], mechanically ground powders were placed in a stainless steel casting chamber and samples were formed by pouring in the molten

Al 7075 alloy. Unlike the work done by Aksöz and Bostan, rather than by directly applying the casting process, this study utilized the mechanical properties provided by the grinding to create better hardness and homogeneous distribution properties in the parts produced via P/M [16]. As a result of the secondary processes applied after hot pressing, Taşkesen et al. also improved the mechanical properties of Al 7075 alloy reinforced with B_4C using the P/M method [17]. An examination of academic studies reveals that they generally try to increase the mechanical and wear properties of the material using standard P/M production techniques (mechanical alloying-pressing-sintering processes) [18]. However, in this study, the authors produced the parts via mechanical alloying and applied the casting in one system, resulting in good hybrid reinforcement distribution, good wet ability, and good wear properties. In the present study, after the grinding was applied, a homogeneous composite structure was achieved and the desired wet ability properties were created among the hybrid powders via free casting. The aim was to improve the metallurgical and wear properties by means of the hybrid composite structures created using the mechanical grinding and casting methods together.

2. Materials and Experimental Procedures

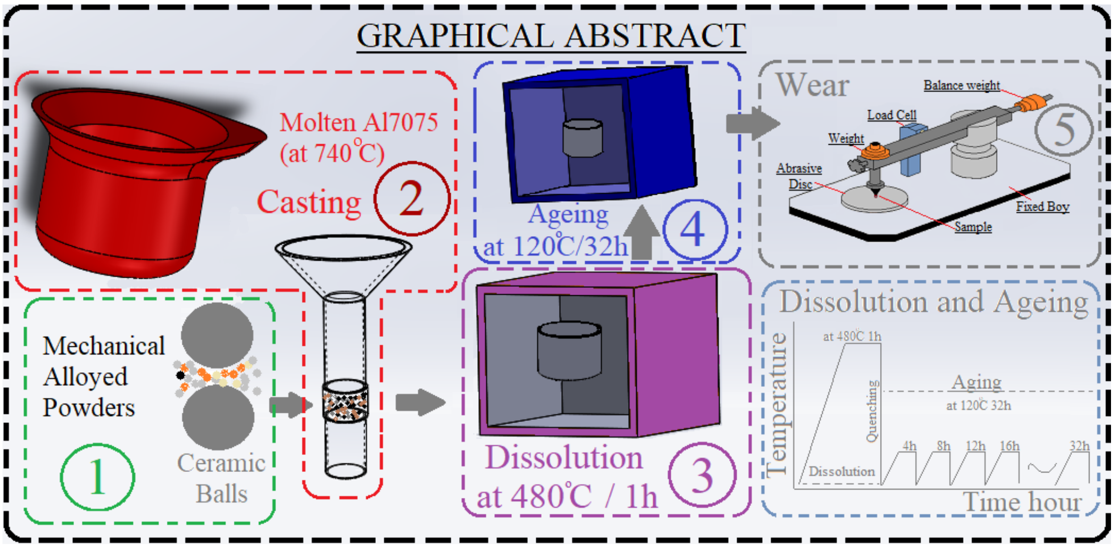

Fig. 1. Graphical abstract of experimental study.

The chemical composition of Al 7075 (as taken from the supplier) is shown in Tab. I. The grain sizes of the Al 7075, B_4C, SiC, and TiB_2 powders used in the study were approximately 40 µm, 10 µm, 150 µm, and 150 µm, respectively (Fig. 2). A planetary ball mill was selected for milling the powders, with a 1:3 ball-to-powder ratio at 600 rev/min.

After milling, the hybrid composite powder structure was transferred to a stainless steel chamber with an internal diameter of 10 mm, creating a powder layer on the lower filter. The molten Al7075 was deposited on this layer, with the specific gravity of the molten metal compelling it to flow downward. The stainless steel chamber was heated to 150 °C in order to achieve the desired properties of fluidity and viscosity. Upon examination of the final products from the chamber, it was observed that the samples had been obtained under the desired conditions.

In the microstructure analysis of the produced samples, field emission scanning electron microscopy (FESEM) and energy dispersive spectroscopy (EDS) analyses were performed using a FESEM ZEISS SUPRA 40 VP device. The microstructural analyses were carried out in the laboratories of the Technology Faculty Metallurgy and Materials Engineering Department and the Advanced Technology Application and Research Center of Pamukkale University.

Tab I. Chemical composition of Al 7075.

Fe	Cu	Si	Mg	Cr	Mn	Ti	Zn	Other	Al
0.5	1.4	0.4	2.5	0.22	0.25	0.2	5.5	0.15	Balance

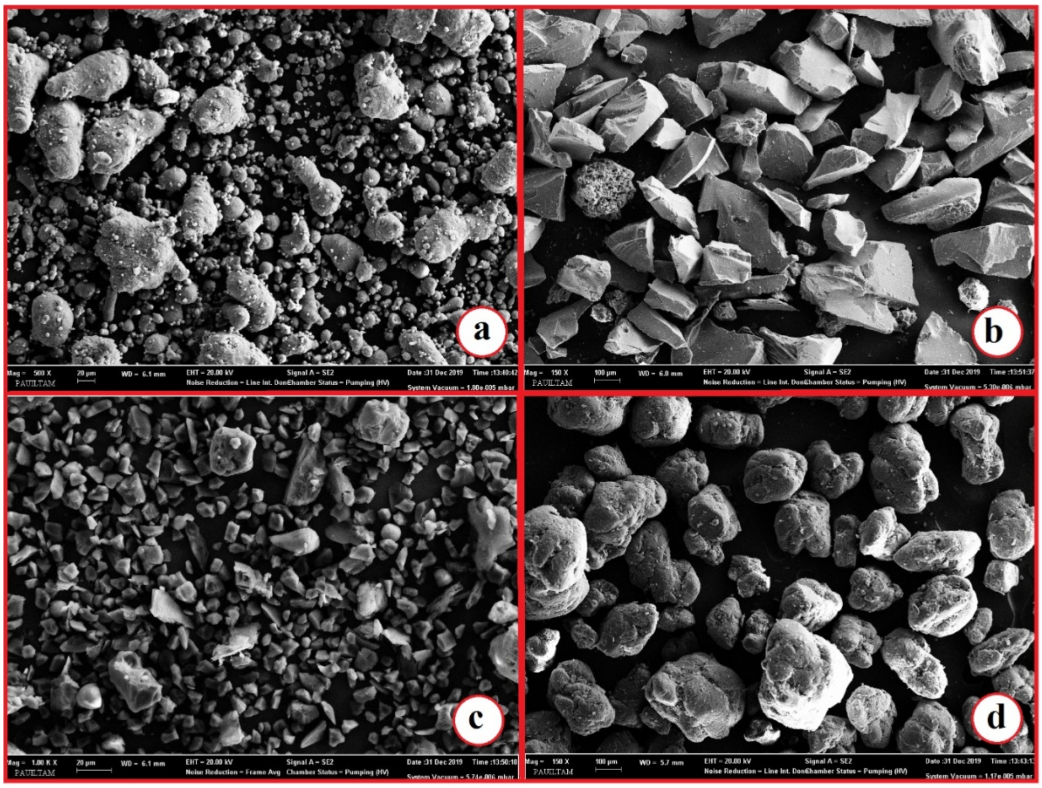

Fig. 2. Field emission SEM images of powders used in the study: (a) Al 7075, (b) SiC, (c) B_4C, (d) TiB_2.

2.1 Hardness Measurement and Wear Experiments of Al 7075 Composites

The hardness measurements were determined by using microhardness devices. The Vickers testing method was carried out using a 136^o pyramidal diamond indenter. The applied microhardness load was 50 g with 10 s dwell time using the Hardway DV-1AT-4.3 hardness tester. For each hardness value, seven measurements were taken and averaged.

All the wear tests were carried out in accordance with ASTM standard G99 for using a pin-on-disk tribometer under dry test conditions. The Al 7075 composite samples were Ø10 × 25 mm in diameter. The wear tests were applied with an AISI 52100 steel disk (60 HRC) used as counterface. In the experiments, the wear test was carried out at two different distances (500 and 1000 m) under 15 N loads at a speed of 2 m/s. The pin-on-disk test apparatus is shown in the graphical abstract in Fig. 1.

The applied load on the specimen was recorded during the wear test (for the calculation of the friction coefficient parameters) and the coefficient of friction was calculated [19] using Equation (1).

The coefficient of friction is $\mu = \frac{F}{P}$ (1)

where, *F* is the frictional force and *P* is the normal load on the specimen. In Equation (2), the weight loss was used to calculate the volume loss:

$$Volume\ Loss\ (mm^3) = \frac{Weight\ Loss\ (g)}{Density\ (g/mm^3)} \quad (2)$$

the specific wear rate is defined in Equation (3):

$$Specific\ Wear\ rate\ (mm^3/Nm) = \frac{Volume\ \ Loss\ (mm^3)}{Sliding\ \ Distance\ \ (m)\ x\ Load\ \ (N)} \quad (3)$$

3. Results and Discussion

3.1 Microhardness and tribological tests

The hardness profiles of the solutionized and aged Al 7075 composites are given in Fig. 3. In Fig. 3, 0 h represents only sintering and solutionizing and shows that the hardness values of the solutionized samples were close to each other. However, the aging treatment up to 12 h caused the hardness values to increase significantly. The highest hardness measurements were obtained as 158 HV and 160 HV for 2 % SiC + 2 % B_4C and 3 % SiC + 3% B_4C, respectively. The microhardness values showed a falling tendency after 16 h of aging, while changes in hardness were not observed until 32 h. This situation described the effects of the mechanical alloying process, with the Al matrix and hybrid composite particles homogeneously distributed [20]. The hard, fine hybrid composite particles were located near the grain boundaries and blocked grain growth during the aging process. Therefore, hardness was increased with the aging at 12 h, and did not decrease much after 16 h [21].

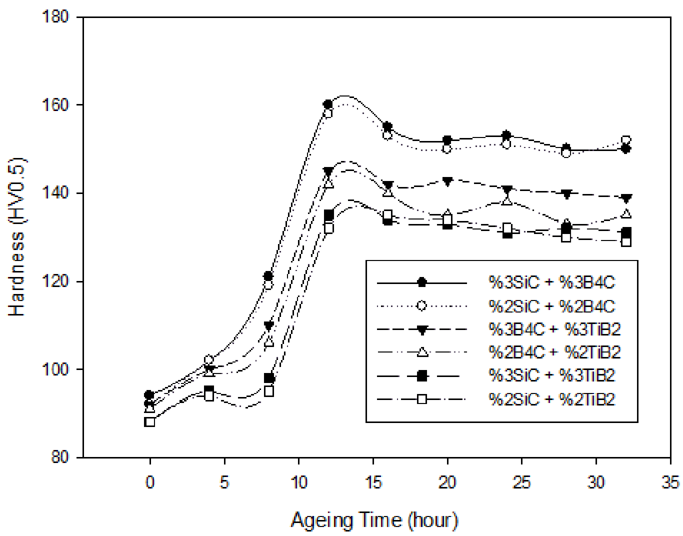

Fig. 3. Hardness values of Al 7075 + B_4C/SiC/TiB_2 hybrid composites after aging.

In Fig. 4, volume loss and specific wear rates are given for each of the six alloys depending on the two different sliding distances. Fig. 4a clearly shows that the reinforcement material in the microstructure directly affected the volume loss. The volume loss increased with the increasing of the sliding distance [2]. Moreover, the lowest volume loss was obtained in the 3 % B_4C and 3 % TiB_2 combination. The 2 % B_4C and 2% TiB_2 combination also exhibited good tribological performance. When B_4C and / or SiC were used in the Al 7075 hybrid composites, it was observed that volume loss was significantly reduced compared to TiB_2 used under the same conditions.

The specific wear rates are given in Fig. 4(b). The wear rate shows the volume loss per unit distance, which is independent of the applied load, while the specific wear rates depend on mass, density, load, and sliding distance. The specific wear rate is an accurate description of the wear characteristics of any material, particularly for metals, alloys, and composites [22]. It was used as a precise indication of the wear properties of the sliding bodies under different loads, speeds, and sliding distances or time periods. The specific wear rate results were also similar to the volume loss results. The lowest specific wear rate was obtained in the 2 % SiC+ 2 % B_4C combination.

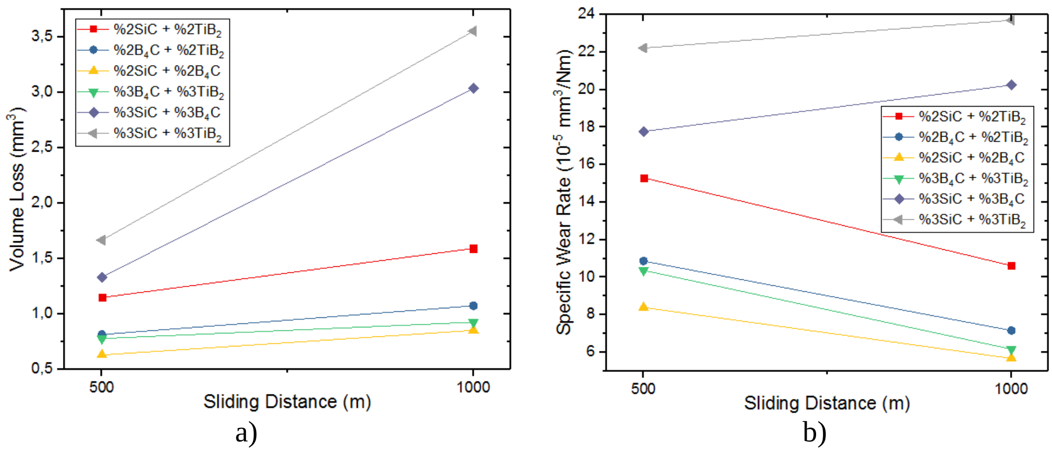

Fig. 4. (a) Volume loss and (b) specific wear rate of Al 7075 hybrid composites.

Another important parameter used in the characterization and investigation of wear behavior is the friction coefficient (CoF), which gives important information about tribological behavior during wear experiments [23]. The CoFs of the Al 7075 composites depending on sliding distance are given in Fig. 5 and the average CoFs are given in Fig. 6. In the wear experiments, the lowest CoF was determined as 0.12 for the 2 % SiC + 2 % B_4C reinforcement and the highest CoF as 0.2 for the 3 % SiC + 3 % TiB_2 reinforcement. These findings were similar to the volume loss and specific wear rate results. The large size of the powder grain (150 μm) of the SiC and TiB_2 led to increased and decreased CoF effects, respectively, because of the the thermomechanical process in the material and the oxide film layer on the material surface, i.e., the effects of the press force and the friction-induced heat [24]. In addition, this situation can be seen in the wear surface of FESEM images (Fig. 7). There is partial oxidation on the surfaces of the abraded samples. These oxides were formed on the sample surface as a result of the increase in temperature during sliding [25] and provided a solid lubricant effect. This situation was also reported in a study by Yıldırım et al. [26]. The EDS analyses were performed on the worn surface of the composite materials and were effective in confirming the homogeneous distribution of the reinforcement additives in the Al7075 matrix. According to the EDS data, the worn surface of the matrix displayed a composition of Al 7075 with a fine distribution of the other reinforcement elements.

Moreover, better CoFs were obtained in the 2 % SiC + 2 % B_4C hybrid composite that had small-sized reinforcement elements since the contact ratio of small-sized reinforcement elements on the surface was high (Figs. 5 and 6). The more surface energy the particles have, the larger the driving force, and in general, fine powders possess higher surface energy than large particles [27].

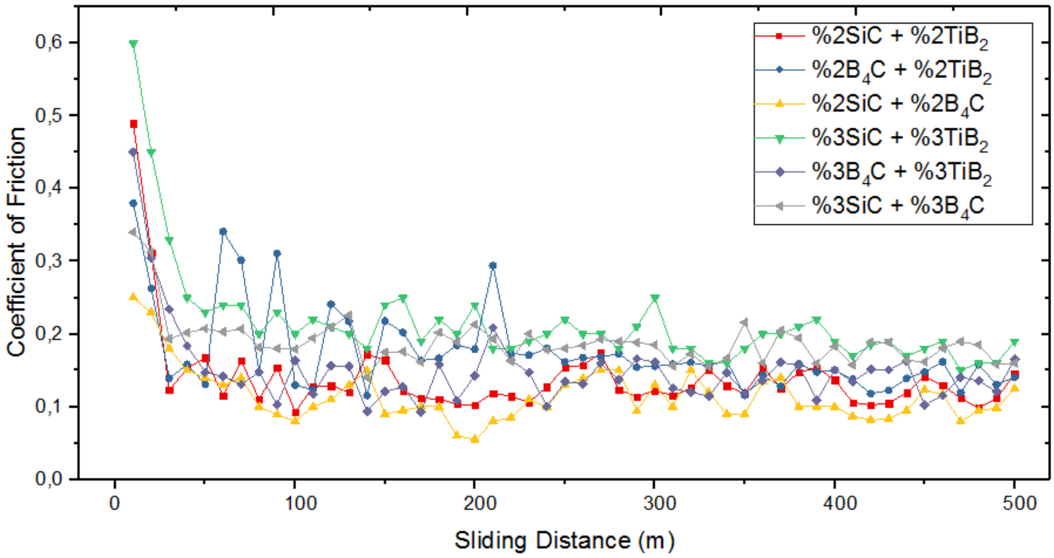

Fig. 5. Coefficent of friction of Al 7075 composites.

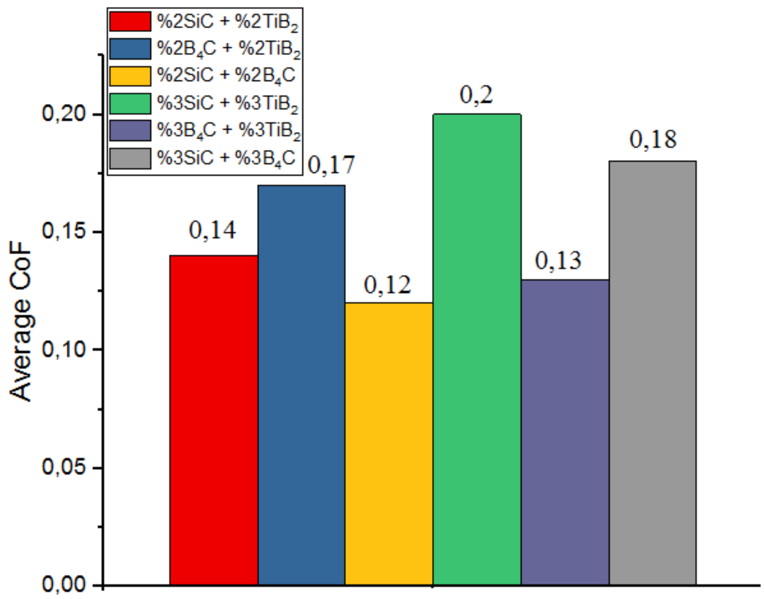

Fig. 6. Average CoF of Al 7075 composites.

The worn surface FESEM images are presented in Fig. 7 in order to characterize the effect of the hybrid composite structure and aging process on the wear behavior. In addition, Fig. 7 shows different magnifications of the hybrid composite structure reinforced with 2 % SiC+2 % B_4C (a,b), with 2 % B_4C + 2 % TiB_2 (c, d), with 3 % SiC + 3 % B_4C (e, f), and with 3 % SiC + 3 % TiB_2 (g, h). The resistance of the Al 7075 composites to scratches and grooves was improved, as the composite hardness was increased with the reinforcements, which also contributed to the decrease in the friction coefficient and wear rate of the Al 7075. There was a decrease in groove depth with the applied aging and increased hardness. An increasing wear rate led to significantly deeper grooves. Although the highest wear resistance was obtained in the 3 % SiC + 3 % B_4C hybrid composite structure, it was observed that deeper wear cavities had formed in the 3 % SiC + 3 % TiB_2 reinforced structure. The main reason for deep wear cavities was that TiB_2 has low wettability in Al 7075. Ceramics such as TiB_2 need to be treated at temperatures exceeding 1000 ^{o}C for good wetting capability in the Al phase [28]. Moreover, researchers did not achieve good wettability properties using the standard P/M production of Al-based composites with TiB_2 reinforcement [29]. If only the casting method is used in the production of the Al matrix and B_4C type reinforcement composite, secondary sintering could be applied to achieve good distribution and wettability [30]. However, the hot pressing method is an effective method to attain good wettability and distribution of composite particles, which in turn lead to enhanced mechanical [31] and wear [32] properties. Kaner reported that materials having high surface energy should be used for good wettability. The wettability was considered to be insufficient due to the casting temperature of 740 ^{o}C in the experiments [33]. The experiments demonstrated that the optimum level of wettability had been reached because of the high surface energy of the B_4C reinforcement.

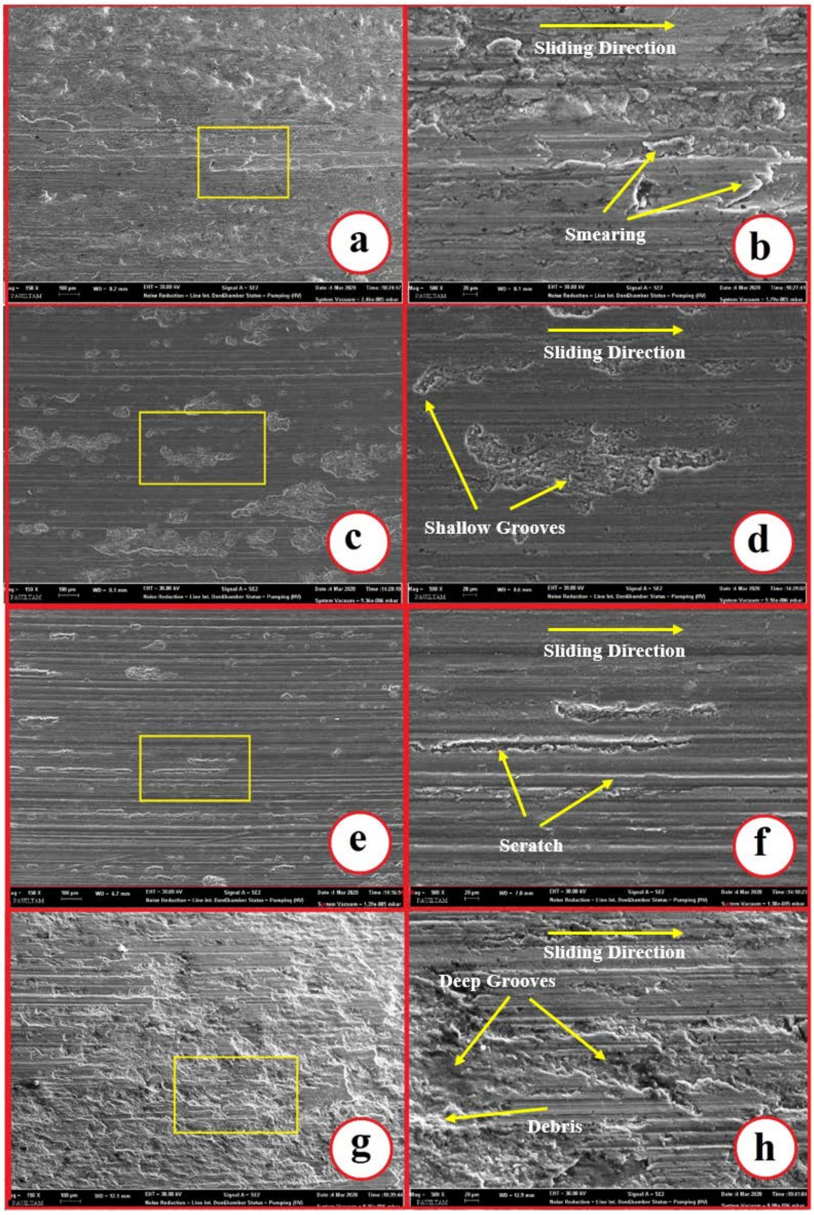

Fig. 7. Field emission SEM images of worn surfaces of Al 7075 reinforced with: (a)-(b) 2 % SiC + 2 % B_4C, (c)-(d) 2 % B_4C + 2 % TiB_2, (e)-(f) 3 % SiC + 3 %B_4C, (g)-(h) 3% SiC + 3 % TiB_2.

4. Conclusion

In this study, the casting and mechanical alloying methods were combined in one P/M system that was used to ensure that the reinforcement hybrid materials were evenly distributed in the Al 7075 matrix. The wear behavior of the fabricated samples was determined as follows.

The wear experiments clearly demonstrated that the filler materials had a significant effect on the Al 7075 material. Moreover, the grain size and amount of the fillers were also important parameters for tribological behavior. The highest hardness measurements obtained were 158 HV and 160 HV in the 2 % SiC + 2 % B_4C and 3 % SiC +3 % B_4C reinforced hybrid composites, respectively. The lowest hardness value was measured as 136 HV in the 2 % SiC + 2 % TiB_2 hybrid composite. The lowest volume loss in the study was found in the 3 % B_4C and 3 % TiB_2 hybrid composite structure. Moreover, the combination of 2 % B_4C and 2 % TiB_2 also exhibited good tribological performance. In addition, when B_4C and/or SiC was selected in place of TiB_2 in the Al 7075 hybrid composite, under the same conditions, the volume loss was significantly reduced. The study demonstrated that grain size was a determining factor in the use of reinforcing elements with similar hardness properties. The reinforcements of SiC and TiB_2 led respectively to increased and decreased CoF effects because of the the thermomechanical process in the material and the oxide film layer on the material surface. This oxide layer formed on the surfaces during sliding provided a solid lubricant effect. According to the EDS worn surface analysis, the matrix displayed Al 7075 composition oxide layers with a fine distribution the other reinforcement elements. The best wettability and associated increase in surface energy were observed in the B_4C reinforcement, while the lowest wettability was seen in the hybrid composite materials with TiB_2 reinforcement. The worn surfaces in the FESEM images revealed that the separation of the reinforcing materials from the base metal created a peeling effect on the surface, causing scratches and cavities.

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Сажетак: *У овом раду, Al 7075 матрици је додато хибридно појачање B_4C+SiC/B_4C+TiB_2/SiC+TiB_2 методом металургије праха. Прахови су механички активирани 240 min, као што је приказано у графичком апстракту. Прах хибридног композита добијен механичком активацијом је стављен у вертикалну комору а истопљени Al 7075 (главна матрица) је додат да би се направио узорак. Узорци су растварани 1 сат на 480 °C, а затим су остављени на по 4 сата на 120 ºC (укупно 32 сата). Након процеса старења, испитивана је отпорност на хабање. У овом раду, FESEM слике су представљале микроструктуру, а криве чврстоће су направљене у зависности од времена третмана. Испитивани су коефицијент фрикције, губитак запремине и слике површине да би се утврдило хабање хибридне структуре. Резултати су показали да повећана стопа побољшања, као и тип и величина, директно утичу на тврдоћу и хабање. У овим експериментима, највиша тврдоћа и отпорност на хабање је примећено код хибридног 3 % B_4C + 3 % SiC након 12 сата старења.*

Кључне речи: *Al 7075 хибридни композит, PM + технологија ливења, старење, FESEM, хабање.*