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Effect of Processing Parameters on Wear Properties of Hybrid AA1050/Al_2O_3/TiO_2 Composites

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Abstract: In this study, hybrid AA1050/Al_2O_3/TiO_2 composites have been produced via combined liquid casting and powder metallurgy techniques. Degassing was utilized to improve the wettability of molten aluminum alloys, and then successful bonding was generated between aluminum matrix and reinforcement particles during the powder metallurgy technique. As the base matrix and reinforcements, AA1050 alloy, Al_2O_3 and TiO_2 particles were taken, respectively. Then, content values of 5Wt.% of Al_2O_3 in the mesh size of 20 µm and 2.5 and 5 wt. % of TiO_2 particles with mesh size of 5µm were added to the AA1050 matrix. For each composite sample, ceramic particles were warmed to 600°C in order to improve wettability and distribution. An identical scattering of subdivisions was observed through aluminum (as matrix) in the microstructural study. To measure the wear resistance, the mechanism of rotary wear test was used. The achieved results illustrated that the fabrication of hybrid composites is an ideal approach to improve the wear resistance of Al-based composites. By increasing of TiO_2 Wt.% up to 5% for all composite samples, the wear rate improved to less than half of the monolithic Al alloy value for each composite sample.

Keywords: AA1050; campsites; TiO_2; wear properties; aluminum

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1. Introduction

Materials are continuously developed with time due to the requirements of human civilization and therefore the advancement of each material in its highest class is the best research necessity [1–3]. The search for new and modern materials is always a significant topic for contemporary technological requirements, as well as making a product at the optimum cost, which is a basic consumer demand. New materials are continually developed and materials' properties are improved in line with existing technological developments in order to meet safety and operational standards [4,5]. Composites are groups of materials consisting of a combination of two or more parts (phases) that remain separate from each other on a macroscopic scale without any chemical reaction. Composite materials consist of two main parts, including the base or matrix and reinforcements. Usually, the base matrix and reinforcements have very different properties. For example, when malleable metals are combined with brittle and strong ceramics, a material is produced that shows improved properties that are completely different from metals and ceramics [6]. Aluminum is one of the most popular and common metals. Aluminum is simple to handle, lightweight and has good corrosion resistance and high strength. Additionally, the economical manufacturing of aluminum is possible through many methods of production. Successful processing and commercial applications of aluminum were started in the 19th century [7,8]. Additionally, Al is a relatively cheap material. Moreover, Al-based composites as a needing group of metal matrix composites (MMCs) have desirable properties such as being light-weight, having high thermal and electrical conductivity and high coefficient of thermal expansion [9–11]. Therefore, having all of these properties together makes them desirable for application in numerous industries such as agriculture, food, medicine, aerospace, automobile, vessels, chemical and electrical [12–14]. During the recent two decades, one of the frequent and major barriers against the progress of aluminum-based composites (ALMCs) in industries was the necessity for a material containing high necessary tribological application and wear resistance [15–17]. Therefore, an increasing demand is felt for the use of these composites due to having continuous progress in science. Nowadays, ALMCs have found usages in many fields of daily life [18]. These kinds of composites could be used in automotive and aviation industries [19,20]. However, their application in certain fields has been weakened due to having unsatisfactory fracture toughness and fatigue properties [21–23]. Usually, the high hardness value of reinforcement particles makes them a desirable additive for utilization as an emergent reinforcement in aluminum-based composites [24]. As mentioned before, the increasing demand for advanced materials in aerospace and automotive industries, and also many other areas, have led to a rapid development of these materials [25,26]. Aluminum, magnesium, titanium and copper have been widely used as metal matrixes in the group of metal matrix composites [27]. However, the production cost of these products is high; this problem can be solved by using cheaper reinforcements and using simpler manufacturing methods. Therefore, finding cheaper and simpler construction methods has always been the favorite issue of many researchers. In comparison with metal alloys, AMCs are not solid solutions and one of the most important requirements in their production is to improve the wettability between Al and its reinforcements [28–30]. Due to low wettability between Al and a ceramic reinforcement and because wettability is an intrinsic property of materials, it is not an overstatement that the history of AMC progress has been developed by various attempts to overcome the poor wetting. Therefore, efforts have been made to achieve this via various strategies including infiltrating molten Al into the ceramic preform under high pressure (infiltration method), high-energy stir casting (i.e., casting), the formation of reinforcements in the Al matrix via in situ reaction and consolidating a mixture of Al and reinforcement powders (powder metallurgy) [31]. All of these techniques account for almost 92% of the production processes adopted in the global market for producing aluminum matrix composites (AMCs) [32–34]. Based on this, the manufacturing methods can be divided into two main groups: solid state and liquid phase methods. Among the solid state methods, mechanical alloying and mechanical grinding can be mentioned. Liquid state methods include vortex casting, pressure casting and injection casting [35]. Meanwhile, the casting method is one of the most cost effective and economical methods of producing metal matrix composites. As one of the casting procedures to fabricate MMCs, the liquid casting process is applicable and popular. This process is especially applicable to produce atomized metal powders with a small amount of environmental contaminations during the fabrication process. Atomization involves the formation of a powder from a stream of molten metal that breaks into tiny droplets [36,37]. Elemental and pre-alloyed powders can be formed, including all metallic powders. An important aspect of this technique is the rapid solidification of metallic particles from the melt as the main technique for producing metal powders [38]. The superheated melt is prepared in an induction furnace and poured into one or more nozzles. The rapid expansion of the gas stream can crush the melt [39,40]. First of all, by the formation of this plate, strings, ovals and spheres are formed. Powder is collected under pressure and a cycle allows the gas to be removed and recycled and the output are very fine particles [41]. The effective parameters in this process are factors such as: alloy composition, metal feeding speed, melting temperature, melting viscosity, pressure, gas temperature, gas type, nozzle geometry, homogeneity of the product and lack of contamination due to conditions [42]. The reason that aluminum composites, as a kind of modern material with desirable characteristics such as wear resistance and high strength to weight ratio, are suggested above is the protection of the contact surface of the composite by a variety of ceramic hard reinforcements. Different kinds of ceramic particles with desirable properties such as high hardness, wear and compressive strength are suggested to be added as reinforcements, such as Al_2O_3 and TiO_2 [43]. Additionally, being reinforced with hard particles dispersed through a relatively ductile matrix, aluminum-based matrix composites usually have an ideal structure for wear resistant materials, which led to a generation of new tailorable engineering materials with improved properties [44,45]. There are several methods to fabricate structural composites such as squeeze casting, powder processing or another process similar to one of these [46]. To produce aluminum-based composites (AMMCs), powder metallurgy is famous as a low-cost process for having the capability for high volume production and its simplicity to generate MMCs with a uniform scattering of reinforcements trough the metallic matrix [47]. Powder metallurgy is one of the methods that has been highly regarded in the manufacturing of aluminum composites. Among the advantages of this method, it can be mentioned that the use of lower temperatures in the composite manufacturing during this process reduces the possibility of reactions between the background (Al matrix) and other phases (usually reinforcing particles). Additionally, the uniform and homogeneous distribution of reinforcing particles is one of the special advantages of this method. In addition, this process creates a low cavity (porosity) structure with an unvarying scattering of the additive particles through the metallic matrix [48]. The wear resistance of aluminum-based composites has been investigated by many researchers, but the comparison between their results is not possible because the resistance to friction and wear are not the only parts of the material's inherent properties and depend on conditions such as applied load, ambient temperature, sliding speed, type and volume percentage of reinforcement particles. In strengthening aluminum alloys with ceramic particles, it has been observed that the wear behavior of the material is usually improved. If the reinforcement is well bonded to the Al matrix, the wear rate is controlled by the wear rate of the reinforcement and in most cases, increasing the volume fraction of reinforcement particles reduces the wear rate of the composite. In this study, and as its novelty for the first time, hybrid AA1050 composites containing Al_2O_3 and TiO_2 particles have been produced using combined liquid casting and powder metallurgy techniques. Then, the effect of variable values of TiO_2 as finer particles (containing constant 5 Wt.% of Al_2O_3 for all composite samples) have been shown on the wear rate vs. different wear velocities and loads. Additionally, using SEM microscopy, the worn surfaces of composites have been studied.

2. Experimental Procedure

The material used in this study was Al alloy 1050. The chemical composition of this alloy is presented in Table 1. Figure 1 illustrates the SEM morphology of particles used as reinforcements. Combined liquid casting and powder metallurgy processes were used together to fabricate hybrid AA1050/Al_2O_3/TiO_2 composites. To fabricate aluminum matrix composites (ALMCs) in this study, Al_2O_3 particles with mesh sizes of 20 μm and TiO_2 with average grain (particle) sizes of 5μm with 0, 2.5 and 5 Wt.% were milled with atomized aluminum alloy 1050 with average size of 40 μm in nitrogen gas as transporter. Alpha alumina, which was originally produced for abrasive products, has been and still is widely used for the fabrication of particle reinforced MMC (PRMMC). This kind of powder is produced by comminution, which results in very sharp particles. A portion of these particles even contain cracks. When introduced into a metal matrix, the result is a brittle composite. The milling process was performed via a ball mill stainless St vial with 10 mm diameter for a full 24 h. During this process, the rotational speed and powder to ball weight ratio were 400 rpm and 16:1, respectively. Additionally, as the process control agent (PCA), 2 Vol.% of Stearic acid was used. Then, at a constant pressure of 870 MPa in a steel die with dimensions of 15 and 25 mm in height and diameter, the resultant powder mixtures were cold pressed, respectively.

Table 1. Chemical composition of aluminum 1050.

Element	Al	Ni	Cu	Ti	Zn	Mn	Fe	Si	Cr
Wt. %	balance	0.0016	0.125	0.004	0.013	0.035	0.225	0.11	0.0015

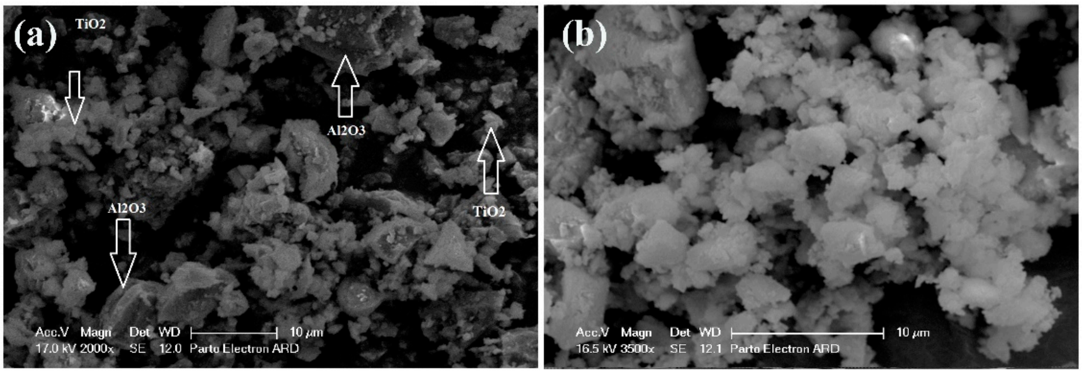

Figure 1. SEM image of mixed 5 Wt.% of Al_2O_3 and (**a**) 2.5 Wt.% and (**b**) 5 Wt.% of TiO_2 particles.

The degreasing of Al particles obtained by liquid casting and ceramic particles in acetone bath for twenty minutes prior to the powder metallurgy is necessary to obtain a satisfactory bonding between Al matrix and particles. Usually, surfaces are full of contaminations such as absorbed ions, grease and dust particles, which are barriers against the formation of a successful bonding. Additionally, this is valid for particles' surfaces where, by decreasing the contaminations, the diffusion between Al and ceramic particles (Al_2O_3 and TiO_2) increases based on the Fick's first law according to the diffusion bonding theory. The diffusion bonding theory is one of the major mechanisms of the powder metallurgy process because of the sintering temperature. It has been found that bonding can be obtained when the diffusion of metallic or nonmetallic surfaces to be contacted together reaches a value sufficiently large to establish contact bonding between phases. Based on Fick's first law and during the sintering process, the diffusion mechanism is greatly improved by increasing the sintering time and temperature, because diffusion is a thermally activated process [49]. Diffusion bonding is ideal for joining sheets and small components with simple interface geometry since it produces joints with no abrupt microstructural discontinuity [50].

To sinter the composite samples, a 70,000 kg hydraulic press at a linear speed of 60 mm/min was utilized. Composite bar samples in 8 mm diameter were extruded with an extrusion ratio of 11:1. The sintering was conducted at 570 °C for two hours. After the sintering process, samples were ready for the wear test. To perform the wear test, a rotary wear test was performed with specimens of 20 mm height and 8 mm diameter achieved from the composite manufactured with constant 5 Wt.% of aluminum dioxide and variable 2.5 and 5 Wt.% of TiO_2. After metallographically polishing the end surfaces, wear studies were conducted under different sliding velocities and loads. During the wear test, by measuring the mass loss of pin as volumetric loss and while the disc speed varied from 150 to 450 rpm in air at room temperature, the applied load varied between 2 and 4 kg. Therefore, the wear volumetric loss was as:

$$VL = \frac{\text{Primary weight} - \text{final weight}}{Density\ of\ the\ composite}$$

3. Result and Discussions

3.1. Microstructure Investigation

The SEM cross section of samples fabricated via 5 Wt.% of Al_2O_3 and TiO_2 particles is shown in Figure 2. The value of Al_2O_3 is 5 Wt.% for all hybrid composite samples. According to Figure 2, the microstructure shows a uniform dispersion of Al_2O_3 and TiO_2 particles through aluminum pattern (matrix) with a desirable adhesion between them. Therefore, it can be concluded from Figure 2 that the fabrication of ALMCs by increasing the powder metallurgy process can improve the adhesion (boding) between the reinforcement particles and Al matrix.

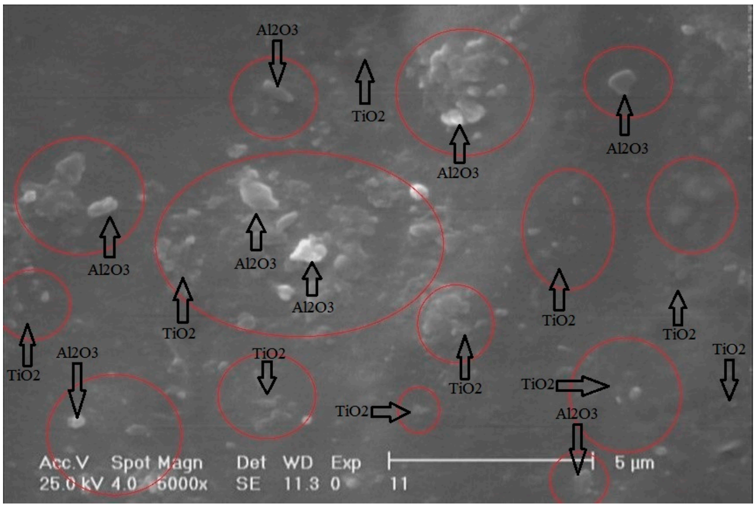

Figure 2. Cross section of hybrid samples reinforced with 5 Wt.% of Al_2O_3 and TiO_2 particles.

3.2. X-ray Diffraction Analysis

Figure 3 shows the X-ray diffraction (XRD) results for the prepared composites. According to Figure 3, the largest and minor peaks indicate the presence of aluminum, Al_2O_3 and TiO_2, respectively. Ceramic peaks can be visibly observed in hybrid composites, including a high intensity of Al_2O_3 peaks and TiO_2 content through the Al matrix. A slow marginal movement of Al peaks to higher angles with an increase in the TiO_2 Wt.% is also obvious. Moreover, there was no oxygen reaction in the samples during the sintering process, as shown in Figure 3.

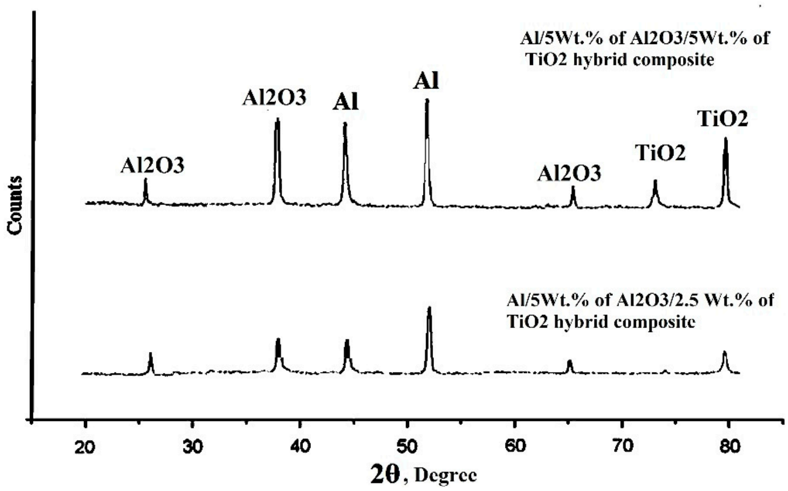

Figure 3. XRD results for the hybrid composite.

3.3. Wear Rate

Wear is the gradual removal of material obtained at contacting surfaces in relative motion. While friction results in important energy losses, wear is associated with increased maintenance costs and costly machine downtime. Wear phenomena are intimately linked to frictional processes. Recall that friction forces are generally the result of two main physical processes, shearing and ploughing. If solid surfaces in relative motion are not separated in some way, wear can be expected. Wear phenomena are heavily influenced by the fact that most engineering surfaces are rough (hence surfaces come in contact at single asperities and the real area of contact is usually much smaller that the nominal contact area). Furthermore, wear behavior is also influenced by the presence of adsorbed species and/or surface layers.

Therefore, it can be obtained from the microstructural results that using both liquid casting and powder metallurgy techniques is a novel approach to fabricate Al-based composites with desirable bonding between particles and the aluminum matrix. Figures 4 and 5 illustrate the variation of wear loss values vs. wear loads. Wear test conditions were 300 rpm of rotary pin at the ambient temperature. These particles were dams against the cutting forming of wear pin, as shown in Figures 4 and 5. Therefore, TiO_2 particles have an enrichment role on the wear properties vs. all wear loads as a lubricant agent in all experimental conditions [48–50]. Additionally, by generating a harder situation for cracks propagation due to decreasing the porosities between Al, Al_2O_3 and TiO_2 particles, which improves the bonding of them, the wear resistance improves. According to the Archard equation and in agreement with a number of reports, the wear resistance of materials is proportional to their hardness. The increased wear resistance of Al-based composites as compared with the base monolithic alloy can be attributed to the presence of the hard Al_2O_3 and TiO_2 phases [12].

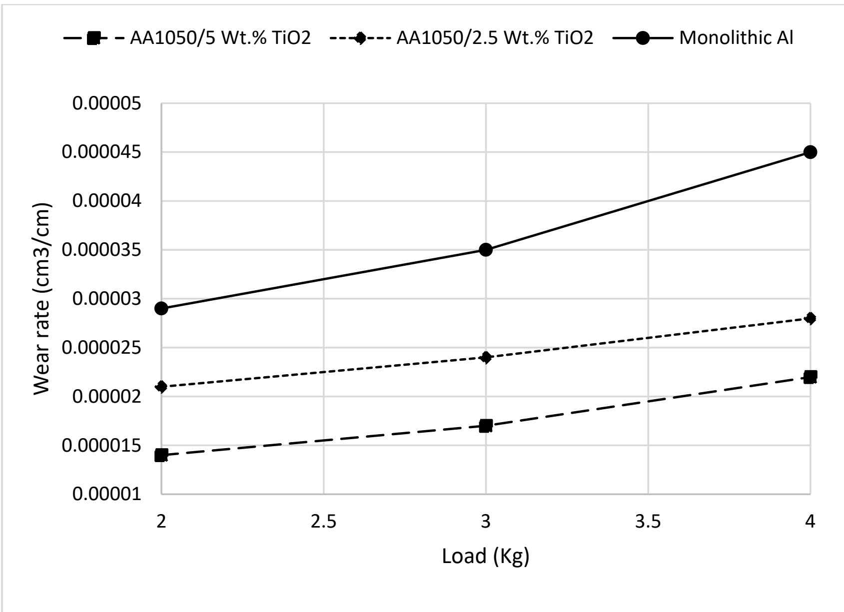

Figure 4. Vol. wear rate vs. applied load (all samples reinforced with 5 Wt.% of Al_2O_3).

Archard's wear model can be broken down into two parts: the single asperity wear model and the contact model. At the single asperity level, the amount of material removed in an asperity interaction is considered proportional to a^3 (a is the asperity contact radius), whereas the sliding distance required to break off the wear particle is proportional to a. This gives the general relationship for the wear rate (worn volume per sliding distance) of a single asperity (Equation (1)):

$$R_1 = \omega A \quad (1)$$

where A is the contact area of the asperity and ω is a generic shape factor, equal to one-third in the case of spherical asperities forming hemi-spherical wear particles. This hypothesis is discussed in and has been verified for isolated debris with molecular dynamics. At the multi asperity level, Archard makes two hypotheses: I. the size and shape of the individual contact areas are given by a contact model considering a rough surface made of spheres with a radius uniformly distributed in depth, with density (number of spheres per unit distance). II. a probability factor K applies on each contact to account for the fact that not all asperity encounters result in a wear particle. Archard assumes K is independent of a.

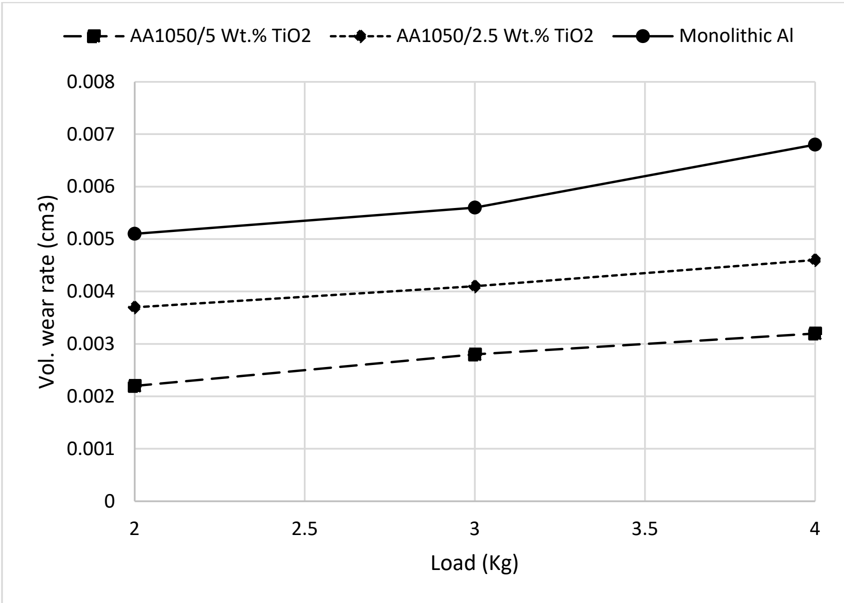

Figure 5. Vol. wear rate vs. applied load at 300 rpm (all samples reinforced with 5 Wt.% of Al_2O_3).

Figures 6 and 7 show the effects of disc speed on the wear properties. Test conditions were disc speeds of 150, 300 and 450 rpm at an applied load of 4 kg, respectively. As can be observed in Figure 7, the wear rate of samples increases with increasing the sliding speed. Additionally, the alterations between monolithic and composite samples are more substantial due to the increasing effect of TiO_2 subdivisions on the wear properties. As the load increases, larger metal particles are torn from the rubbing surfaces. The oxidation rate induced by flash temperature is not large enough to fully oxidize the wear particles, and many particles are ejected from the gap and discarded as wear debris. Because the wear rates involved are larger, this is called severe wear. For the highest loads, the resulting high flash temperatures lead to the rapid formation of oxides and a hard tempered layer on the contact surfaces. Fine metal particles are formed, oxidized and removed from the gap between the rubbing surfaces as wear debris. Since the contacting surfaces harden rapidly, the resulting wear rate is relatively small. This is known as the high temperature regime of mild wear.

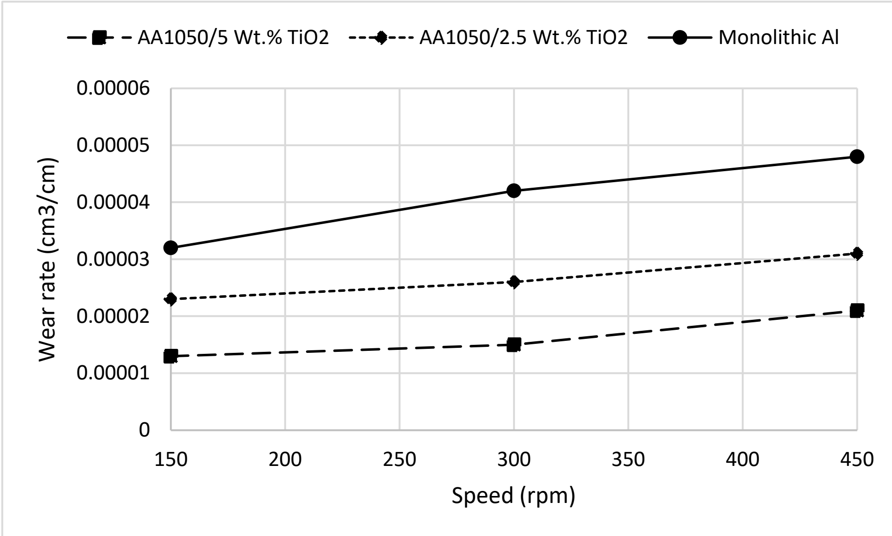

Figure 6. Wear rate vs. the rotating wear pin speed (all samples reinforced with 5 Wt.% of Al_2O_3).

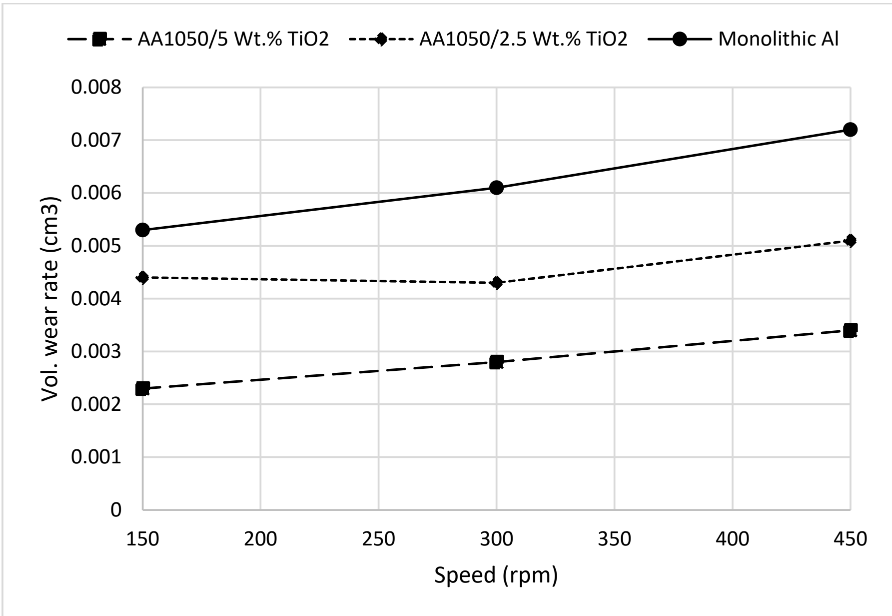

Figure 7. Vol. wear rate vs. the rotating wear pin speed (all samples reinforced with 5 Wt.% of Al_2O_3).

To clarify the wear phenomenon in metals and composites which are abrasive, there are three mechanisms: adhesion, coexistence and delamination. The study of bond formation between composite layers is essential to explain this phenomenon. Before the bonding and as mentioned before, the surface of materials is composed of dust particles, grease and absorbed ions. Therefore, to obtain a desirable bond formation, degreasing in the acetone bath is necessary. During the powder metallurgy technique, contact of opposite surfaces of layers begins, they reach each other and metallic bonding zones are formed. Therefore, when the crystalline structures of layers are same as together, the powder metallurgy process can generate metallic bonding between them by means of a diffusion mechanism. Increasing the volume of diffusion value during the sintering process creates electron sharing between layers and makes bonding on the atomic scale. Therefore, numerous bonds are formed by noticeably bigger, wide areas of the base alloy or metal. These unbonded (darker) regions, which are determined with red color, look like tree branches where there are suitable zones for crack delamination and propagation due to the presence of these branches during the wear test, as seen in Figure 8.

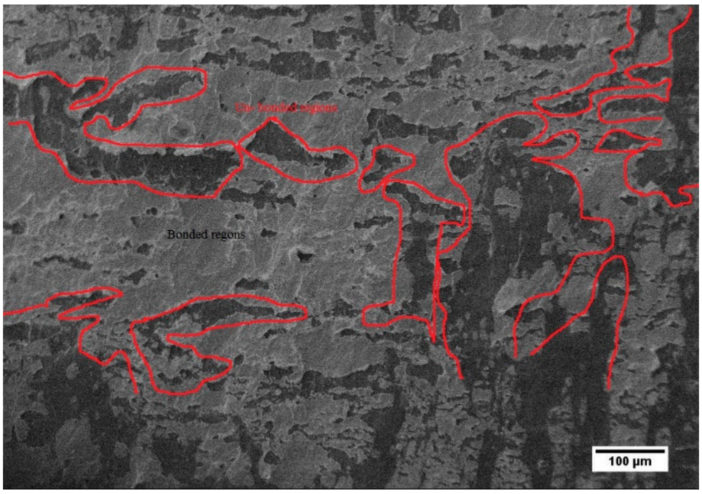

Figure 8. Bonding area between composite layers of Al/5 Wt.% TiO_2 sample.

3.4. Worn Surface

Figure 9 shows the worn SEM surface morphology of annealed Al and Al/5 Wt.% TiO_2 samples at 4 kg and 300 rpm. As noted before, reinforcement particles always decrease the friction between the revolving wear test pin and composite surfaces. In other words, TiO_2 particles have a positive role on wear properties. The layering and wear paths are illustrated in Figure 9 and, due to the improving role of the TiO_2 phase as lubricant, the width of channels decreased. Therefore, in comparison with monolithic samples, hybrid samples have an enhanced wear resistance. After the beginning of wear between worn surfaces and the pin and based on the above-mentioned discussions, the development of the grains happened at the deformed regions due to high temperature, with more growth in the subsurface region than the middle thickness. Therefore, a coarse grain structure was formed under the subsurface, containing a strain incompatibility regarding the fine grain structure and non-equilibrium UFG grains. Additionally, debris particles were formed when the strain incompatibility created a delamination of coarse grains. In other words, these delamination and recrystallization processes happened in repetitive rounds as a result of this kind of wear mechanism and changed the shape of loss of material from flakes to plates. Therefore, the flows of surface material generate abrasive grooves under higher applied loads.

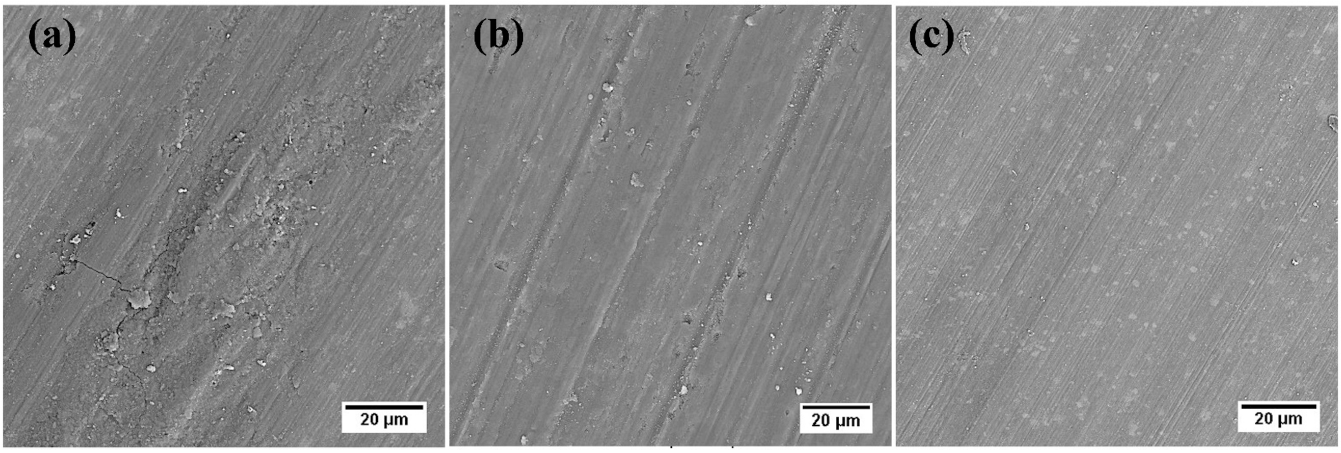

Figure 9. SEM worn surface of (**a**) annealed Al, (**b**) Al/ 2.5 % TiO_2 and (**c**) Al/ 5 % TiO_2 samples (hybrid samples are also reinforced with 5 Wt.% of Al_2O_3).

Therefore, as mentioned before, it is reasonable that the extent of delamination was increased during the wear test. Indeed, during the wear test, the nature of the laminated structure through the composite structure helped more extensive delamination. During the wear test, three regions can be considered for the composite volume that is related to this phenomenon. The second zone describes deformed regions of the base matrix. The elastic–plastic deformation was noted at the interface between regions one and two. Region three is known as the tribolayer and includes formed worn oxide surfaces and counter face material. Additionally, the wear debris made between regions two and three is a zone void and crack formation. Therefore, the conditions of material, environment and sliding wear severely effect the compositional features and extents of these subsurface regions, which are established rapidly [18].

Based on the above-mentioned discussions and after the beginning of wear between the worn surface and the pin, the development of grains happened at deformed regions due to high temperatures, with more growth in the subsurface region than the middle thickness. Therefore, containing a strain incompatibility regarding the fine grain structure and non-equilibrium UFG grains, a coarse grain structure was formed under the subsurface. In other words, debris particles were formed when the strain incompatibility produced a delamination of coarse grains. Additionally, these delamination and recrystallization processes happened in repetitive rounds. The generation of large particle debris in a shape is the result of this kind of wear mechanism. The initiation and nucleation of cracks on the sliding surfaces are the results of high shear stresses. Therefore, this changes the shape of loss of material from flakes to plates. The flows of surface material are toward the sliding direction, which generates abrasive grooves under higher applied loads [15].

4. Conclusions

The investigation of the tribological behavior of hybrid $Al/Al_2O_3/TiO_2$ bulk composites was the main purpose of this study. Wear test and SEM analysis were performed and the following results were attained:

Aluminum and its alloys are one of the most desirable groups of materials, with characteristics that can give most of the current requirements and often exhibit reasonable tribological, mechanical features when used as matrix materials in aluminum matrix composites. Additionally, Al_2O_3 and TiO_2 are the materials generally being used for reinforcements. Moreover, using liquid casting and powder metallurgy techniques together is an applicable approach to fabricate aluminum-based composites.

A uniform scattering of Al_2O_3 and TiO_2 particulates has been achieved inside the aluminum matrix. This means that the milling process and powder metallurgy techniques are good ideas as supplementary processes to obtain desirable particles scattering inside the aluminum matrix.

By increasing TiO_2 Wt.% inside composite samples, the wear properties of composites developed extensively. These particles were dams against the cutting forming of wear pin. Therefore, TiO_2 particles have an enrichment role on the wear properties.

As compared with monolithic AA1050, significant increasing in the wear and volumetric loss was achieved at upper wear speeds and loads by means of enhancing the role of Al_2O_3 and TiO_2 particles in hybrid $AA1050/Al_2O_3/TiO_2$ composites. Additionally, as a result of wear resistance enhancement, the width of channels decreased due to the improving role of the TiO_2 phase as lubricant.

Author Contributions: Y.G., M.H.V. and S.D. conceived and designed the experiments; M.H.V. and S.D. performed the experiments; A.A.A. and O.B. analyzed the data; R.H.C.A. wrote the paper. Each contributor was essential to the production of this work. All authors have read and agreed to the published version of the manuscript.

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