Experimental investigation of hardness and effect of wear on sintered composites containing AA6061 matrix and TiB$_2$/Al$_2$O$_3$ reinforcements

N D Raja and D S Prakash*

Department of Mechanical Engineering, Vel Tech Rangarajan Dr. Sagunthala R&D Institute of Science and Technology, 400 Feet Outer Ring Road, Avadi, Chennai – 600062, Tamil Nadu, India

* Corresponding author: dsurvyaprapakash@gmail.com

Abstract. Materials prepared using powder metallurgy route exhibit dimensional stability which is required to manufacture critical parts such as brake pads, inlet valves and nozzles. In this paper, composite materials consisting of 5 and 10 wt. % of Titanium-diboride reinforcement in AA6061 aluminium alloy was produced by the sintering process via green compaction. 2 wt. % of alumina was added as the secondary reinforcement to produce the hybrid composites. The produced composites and its hybrid were compared with the base material AA6061 to determine the changes in its density, wear and hardness. The green density and sintered density of the composites and its hybrid were reduced by as much as 8.47 % compared to the respective theoretical density. Microscopic examinations showed that the addition of alumina with the Titanium-diboride reinforcement increased the wear resistance of the hybrid composites by 1.22 % compared to the base material. The hardness of the sintered composite increased with the addition of the two reinforcement materials. The hardness for the hybrid composite increased by 74.94 % compared to the base material. After wear test, the hardness of the base material reduced by 6.83 % but the hardness of hybrid composite having the highest composition of both reinforcement particles increased by 6.36 %. The study revealed that the sintering process and subsequent hardening of the composites and its hybrid enhance its hardness property compared to the base material. Interestingly, the wear which leads to the changes in its tribological properties increased the hardness property of the composite and its hybrid.

Keywords: AA6061, TiB$_2$, Al$_2$O$_3$, Wear, Hardness.

1. Introduction

Aluminium Alloy AA6061 are used as principle alloy for applications that extend from covers and panels for building structures, automobile body, railway wagons and shells for air crafts [1]–[3]. Metal Matrix Composites (MMC) comprising of ceramic particles like Silicon Carbide (SiC), Titanium Carbide (TiC), Titanium diboride (TiB$_2$), Alumina (Al$_2$O$_3$) and Tungsten Carbide (WC) as the reinforcement materials are replacing traditional alloys because of its beneficial properties like low density and corrosion resistance [4]–[12]. Selection of reinforcement particles is an important factor since it can alter the properties like tensile strength, hardness, impact strength, flexural strength, corrosion resistance, thermal properties and tribological behavior of the produced MMC [11], [13]. Compared to MMCs, the Hybrid Metal Matrix Composites (HMMC) shows enhanced properties, achieved by infusing the distinctive properties of two or more reinforcements into the matrix element [14].
Addition of TiB$_2$ and Al$_2$O$_3$ as the reinforcement particles either as standalone reinforcement or as hybrid materials are found to enhance mechanical properties of the produced composites. TiB$_2$ in AA6061 is noted to reduce the density and wear of the produced composite, at the same time increase the tensile strength, hardness and corrosion properties of the MMC and HMMC [15–18]. Similarly, Al$_2$O$_3$ enhances tensile strength, microhardness and wear resistance of AA6061 [19], [20]. Several research articles suggest that increasing the hardness of the material can enhance the tribological properties of the material [21]–[24]. Though there are numerous choices of reinforcement materials, TiB$_2$ and Al$_2$O$_3$ are the ideal choices to increase the wear resistance property. Wear is an undesirable property of a material that mainly occur because of the frictional resistance at the surface which moves over another material [8], [25], [26]. Majority of the research articles have shown improvement in the resistance to wear after addition of TiB$_2$ and Al$_2$O$_3$ as an individual component or as hybrid reinforcements [27]–[29]. The composites were produced using methods like stir casting, compo casting, in-situ process and friction stir processing [28], [30], [39], [31]–[38]. Materials manufactured using liquid metallurgy route like casting exhibit defects like stress-induced cracks, air voids, and porosity. The in-situ process may overcome these defects but are not effective for widely varying compositions. They rely greatly on the skill of the operator producing the composite. Friction Stir Processing (FSP) technique is useful to improve the mechanical properties of MMCs [40], [41]. But it is not a suitable option for composites produced using powder metallurgy route. There are very few articles that have been projected in the area of wear, especially on composite materials containing AA6061 matrix and TiB$_2$ and Al$_2$O$_3$ as the reinforcement materials manufactured using Powder Metallurgy (PM) route. However, these articles do not discuss the effect of sintering on the wear-resistant property of the composite material [42]–[45]. The presence of porosity in the green compacts can interfere with the bonding. Such a phenomenon can greatly affect the hardness and wear of the composite materials. However, the sintering process reduces the porosity of the produced composite which benefits by improving the hardness and wear resistance of the material. The subsequent changes in wear behaviour of AA6061 composite produced by PM and its wear resistance behaviour after sintering is poorly studied and demands research. Hence, there is a need to analyze the mechanism concerning the wear resistance property of sintered AA6061 composites. Further, the wear resistance of MMC and HMMC is compared against the base alloy AA6061 subjected to sintering. Two composites are produced using Titanium Boride (TiB$_2$) as the reinforcement particle in two different weight composition (5 and 10 wt. %). The composites are manufactured using powder metallurgy technique. Additionally, two more hybrid composites are produced using the same technique by adding 2 wt. % of alumina (Al$_2$O$_3$) as the reinforcement particles. Densities of the produced materials are also compared to check the influence of the reinforcement particles over the base alloy. Moreover, Scanning Electron Microscope (SEM) analysis is used to find the wear on the surface of the materials. The test results revealed that compared with theoretical density, the sintered density of the composites and its hybrid reduced due to the compaction and subsequently hot press. Addition of the two reinforcement particles reduced the wear of the hybrid composites compared to the base material (AA6061) and its counterparts. The novelty of the paper is the correlation of the hardness of the base material, TiB$_2$ reinforced composites and its hybrids before and after wear test. The hardness of the hybrid composite having 10 wt. % of TiB$_2$ and 2 wt. % of Al$_2$O$_3$ increased after sintering. The hardness increased further after wear test on the same composite.

2. Experimental work

2.1. Production of composite through powder metallurgy route

The MMCs and HMMC in this study were prepared by including two different reinforcement materials in the matrix of AA6061. Table 1 and 2, shows the chemical composition and physical properties of the matrix element. Compared to the manufacturing techniques like casting, chemical vapour deposition and in-situ processes available to produce MMCs, powder metallurgy are most suited to make small parts with simple geometry requiring accuracy in its dimensions [10]. Materials produced using PM method can exhibit an absence of undesirability like brittle reaction and segregation effects [46].
Table 1. Chemical composition of AA6061.

| Material | Cr  | Cu   | Mg  | Fe  | Si   | Mn  | Al  |
|----------|-----|------|-----|-----|------|-----|-----|
| Weight (%) | 0.35 | 0.15~0.4 | 0.8~1.2 | 0.7 | 0.4~0.8 | 0.15 | Bal. |

Table 2. Physical properties of AA6061

| Description | Yield Strength (MPa) | Tensile Strength (MPa) | Vicker’s Hardness (GPa) | Elongation (%) | Density (g/cc) | Impact strength (J) |
|-------------|----------------------|------------------------|-------------------------|----------------|----------------|---------------------|
| AA6061      | 240                  | 290                    | 39.5                    | 22             | 2.7            | 3.5                 |

For the experiment, powders of aluminium, copper, manganese, iron and the other constituents shown in Table 1, each of size 67µm were procured along with 45 ~ 37µm size of Al₂O₃ and 200 nm size of TiB₂. The reinforcements TiB₂ (5 wt. % and 10 wt. %) and Al₂O₃ (2 wt. %) are added to the matrix element. Apart from this, two more composite materials comprising of TiB₂ only (5 wt. % and 10 wt. %) as the reinforcement material is produced. Table 3 shows the individual compositions of the matrix element and the reinforcement materials in each of the composite materials as well as the base alloy.

Table 3. Composition of composite materials.

| Description  | Quantity (%) by wt. |
|--------------|---------------------|
| Base Material | AA60601 TiB₂ Al₂O₃ |
| Composite A  | 95 5 0              |
| Composite B  | 90 10 0             |
| Composite C  | 93 5 2              |
| Composite D  | 88 10 2             |

The fabrication procedure of the base material, MMC and HMMC having two different percentage composition of TiB₂ and Al₂O₃ began by mixing the required quantity of powders. The elemental powders were taken within the proportion mentioned for matrix alloy in a spherical bowl of 200g capacity. The powders are subjected to intense mixing using a stirrer revolving at 50revs/min for 6h. The mixed powders were uniaxially compacted at 380MPa in a 40Ton Universal Testing Machine (UTM) with dies as shown in figure 1.

Figure 1. Green compaction of the composite.
Lauric acid (fatty acid) was used as the lubricant to minimize the friction during the compaction process. The densification of cylindrical pellets during the sintering process and microstructures were evaluated on cylindrical pellets (20 mm diameter and 18 mm height). Sintering of all samples was performed in a Nitrogen-rich atmosphere, controlled using the hot pressing machine as shown in figure 2 [47]. The sintering process was allowed to take place in a cycle. Powders were heated to 560 °C at a rate of 10 °C/min. The mixture was then heated to sintering temperatures of 610°C and they were held at sintering temperature for 60 min. The density of the green compacts and also the sintered composites was found by measuring the ratio between the mass and volume [48]. Sintered bars of 20 mm diameter with greater than 98.5% of the theoretical density were obtained. The base material was prepared using the procedure mentioned above without any reinforcements to compare the property variation with the composites and its hybrid.

2.2. Testing of the produced composites
The materials were first tested to find the changes in the green density and sintered density. The density of the specimen was measured using Archimedes’ principle. For this, the specimen followed ASTM B962-13 standard [49]. The theoretical density was measured using the rule of mixture. The hardness for each material was determined by calculating the average hardness at various locations along its surface using the Vickers hardness test machine. Each specimen for the hardness test was prepared according to ASTM E384 standards and loaded with 5N force for 10s [49]. The corresponding readings were noted down and its average was used as the hardness value for the corresponding material. This was followed by the determination of the wear exhibited by respective materials.

Specimen for wear test was prepared by machining the sintered bar to the size of 10mm diameter and 30mm thickness. The wear test was carried out using a pin on disc tribometer [50]. The machine consists of a variable drive mechanism electrical motor capable of rotating a steel disc of size 55mm. The specimen was clamped so that the circular section was resting on the steel disc. A hinged bar supporting 30N load on the opposite side was used to balance the test specimen. The steel disc was allowed to rotate at 1800rpm and transverse at 2m/s for a duration of 5mins. The rate of wear was recorded by calculating the fluctuation of load in the pin side using a strain gauge attached to the hinged support as per ASTM G99-05 standard [49]. Scanning Electron Microscope (SEM) images were used to check the extent of wear that took place on the respective specimen. The hardness of all the specimen before and after wear test were compared.

3. Results and Discussion
Figure 3 compares the densities of the materials used in this study. Each material, including the base alloy and the four different composites, are correlated with the respective theoretical values. It is noted that the theoretical densities of the composite increases because of the addition of the density of the reinforcement materials in the composite. The addition of TiB₂ alone as the reinforcement material
increases the density by 5.19 % and 8.52 % respectively for each 5 wt. % addition of the reinforcement material. Further, the addition of 2 wt. % of Al$_2$O$_3$ increased the theoretical density, i.e., 5.93 % and 9.26 % respectively.

Figure 3. Density Comparison of the selected material.

After compressing the base alloy, the green density reduced by 1.11 % compared to its theoretical density. After green compaction, the density of the composite A and B reduce by 1.12 % and 1.87 % respectively compared to the base alloy. This occurs because of the compression of the powders. Addition of 2 wt. % of Al$_2$O$_3$ showed negligible variation in green density in the composite C. However, the green density increased by 3 % in the composite D.

Sintering the green compact resulted in recrystallization of the composition of the materials. Because of the superior thermal conductivity of the matrix element, it underwent an expansion process. The material had permanently set while it cooled down to the surrounding temperature. This unique phenomenon was also observed when the composite D. For other composite materials, the sintered density was observed to reduce in comparison with its respective green density. This event can be justified by the low thermal conductivity of the reinforcement materials, which hindered the transfer of heat energy during the sintering process.

Table 4 shows the hardness of the sintered base metal, MMCs and HMMs produced for this study. It can be noted that the base metal composed of AA6061 developed the lowest hardness value of 39.5GPa. The powder metallurgy route generates the required shape of the material using the principle of mechanical alloying and subsequent heat treatment during the sintering process. This method though suitable for small shapes and simple geometry relies mostly on the extent of cohesion that takes place between the microparticles. The pressing force that is applied to the powders also influences the cohesion of the particles. However, the combined action of the high temperature and the compressive force is sufficient to vaporize and remove the moisture or air bubble that may have trapped in between the particles but is insufficient in enhancing the properties of the produced materials.

| Material       | Hardness (GPa) |
|----------------|----------------|
| Base Material  | 39.5           |
| Composite A    | 54.7           |
| Composite B    | 58.4           |
| Composite C    | 59.7           |
| Composite D    | 69.1           |

AA6061 produced using sintering and subsequent extrusion developed Vickers hardness value of 64.53GPa [51]. The base material though having the same composition developed 38.79% lower hardness value because of the insufficient bonding among the particles. Interestingly, the addition of 5
and 10 wt. % of TiB₂ particle reinforcement increased the Vickers hardness value by 38.5% and 47.85% respectively. This justifies that the adhesion between the TiB₂ particles and AA6061 powders was good. The increase in weight composition of TiB₂ particles benefited by contributing its properties and raising the hardness of the MMCs correspondingly with the weight of the added particles. However, the hardness of the sintered MMC was still lower than the sintered and extruded base material as shown above.

Hybridization of the MMC by adding 2 wt. % of Al₂O₃ particle reinforcement resulted in an increase in the Vickers hardness value by 51.14% and 74.94% respectively. The properties of the Al₂O₃ particles were integrated into the HMMCs which increased the hardness values. Comparing with the sintered and extruded base material, the composite D showed an increase in Vickers hardness value by 7.1%. This justifies that the appropriate composition of reinforcement particles can enhance the mechanical properties of the base material.

Figure 4 shows the wear that took place on the base metal AA6061. It is noted that the wear fluctuated with time. During the interval of 130 to 170s, the rate of wear had increased abnormally compared to all other duration. Such conditions can occur when the surface subjected to wear test have alternate regions of the hard and soft surface. This is justified by the fluctuations in the wear observed at 130s, 170s and 225s respectively. The wear is noted to have reduced at these locations, intimating that the surface had high resistance to wear compared to its adjacent surfaces. Unlike cast alloys, the ones prepared using powder metallurgy route exhibit its unique behaviour of uneven hardness along the surface. It is an inference that the porosity in the green compacts created micropores that lead to the uneven wear of the base metal AA6061.

Figure 4. Wear vs time for AA6061.

Figure 5 shows the SEM image of the AA6061 subjected to the wear test. Wear lines occurred as the result of the pin rubbing over the rotating disc. The wire lines are noted to be uneven, prominent lines occurring closer to the region that has micropores. This justifies that the surface is riddled with regions of varying hardness, leading to the fluctuation in the wear.

Figure 6 shows the wear that took place on the specimen coded as composite A. It is noted that the wear increased uniformly with the time during the initial duration of 55s. By this time, the wear elevated to 2000µ/s, thereafter it maintained at the highest wear rate. The TiB₂ particles have different thermal properties than the matrix element and this phenomenon impacted the bonding between the matrix and reinforcement during the sintering process. The SEM image in figure 7 shows the effect of TiB₂ particles (5 wt. %) in the AA6061 matrix. The low concentration of the reinforcement particles created regions that have a high wear rate. Besides this, the poor bonding of the reinforcement particles leads to chipping of the particles, giving rise to microcavity. The presence of cavity gave rise to a trailing surface exhibiting high wear. This phenomenon occurred as the detached particles eroded the surface of AA6061 during the wear test.
Figure 6. Wear vs time for composite A.

Figure 7. SEM after wear test on composite A.

Figure 8. Wear vs time for composite B.

Figure 9. SEM after wear test on composite B.

Figure 10. Wear vs time for composite C.

Figure 11. SEM after wear test on composite C.

Figure 8 shows the wear that took place on the specimen coded as composite B. It is noted that similar to the previous case, the wear increased uniformly with the time at a relatively faster rate than the base alloy. However, in this case, it took 70 s to reach the maximum wear rate of 2000µ/s. The slight increase in time duration is related to the increase in the concentration of the reinforcement particles. The presence of twice the quantity of TiB$_2$ particles resulted in a drastic change in the microstructure of the resulting composite as shown in figure 9. It is observed that 10 wt. % of TiB$_2$ particles affected the recrystallization of AA6061 during the sintering process, leading to an increase in the micropores within the matrix. The variation in thermal conductivity of the two materials made the matrix element to acquire brittle nature. This is evident from the presence of cleavages along the surface of the MMC. The brittle nature of the MMC permitted the chipping of the matrix, leading to an increase in material removal and increased wear.

Figure 10 shows the wear that took place on the specimen coded as composite C. The HMMC exhibited a unique wear behavior in which the wear rate was drastically reduced during the interval 65 to 110s. This occurred because the TiB$_2$ and alumina particles acted as the obstacle against the frictional wear as the pin rubbed on the surface of the HMMC. However, the particles could not be held longer because of poor bonding with the matrix element. When the particles got detached, it aided in increasing the wear, leading to a sudden increase in the wear to the maximum of 2000µ/s.
Figure 11 shows the SEM image of composite C after wear test. It revealed the presence of microcavities as the result of particles debonding from the matrix. These particles aided in increasing the wear in the HMMC, observed from the trails of eroded materials along the wear lines. Compared to composite A, composite C has alumina as additional reinforcements. The combined contribution of these two particulates increased the erosion during the wear test after they got de-bonded from the matrix.

**Figure 12.** Wear vs time for composite D.

**Figure 13.** SEM after wear test on composite D.

Figure 12 shows the wear that took place on the specimen coded as composite D. The HMMC exhibited a similar trend in wear that was exhibited by composite C. The duration of which the HMMC revealed a reduction in wear rate is shifted from 75 to 150s. The alumina particulates interacted during the recrystallization and prevented the formation of brittle nature of the matrix element. As a result, the particulates could be retained for a longer duration. Hence the wear rate is low for this HMMC for a prolonged duration. However, the particles could not be held longer because of poor bonding with the matrix element. When the particles got detached, it aided in increasing the wear, leading to a sudden increase in the wear to the maximum of 2000µ/s.

Figure 13 shows the SEM image of composite D after wear test. The image revealed that the particles were clustered because of the changes in thermal conductivity affecting its recrystallization during the sintering process. The increased quantity of reinforcement particles effectively reduced the wear rate for a short duration. However, the wear was much greater than that observed from base alloy produced via PM without adding any reinforcements. This is because the particles that are weakly bonded quickly got detached and aided in the wear of the HMMC. During the wear test, a greater number of particles were de-bonded which resulted in deep wear lines over the surface.

**Figure 14.** Hardness comparison of composites.

Figure 14 shows the comparison of hardness between the base metal, MMCs and HMMCs before and after wear test. It is noted that after the wear test, the hardness of the base material dropped by 6.84%. This is because the base material have characteristics of the ductile material which becomes soft and
elastic under the application of heat. The frictional heat imitated annealing process that induced softness to the base material. Besides, the poor mechanical properties observed after sintering without extrusion increased the phenomenon of metal softening. The tribological characteristics of the post-wear-tested specimen revealed the presence of deep wear lines that justifies the soft nature of the base material.

The influence of heat during the wear test increased the mechanical properties of the composite A and B. As a result, the Vickers hardness value for both the MMCs was noted to increase by 3.66% and 2.22% respectively. The presence of hard and brittle TiB₂ particles contributed to enhancing the mechanical properties of the MMCs while it is subjected to heat treatment through frictional heating. However, the rise in the hardness value is not sufficient to make a considerable challenge to the one observed from past research [51]. It is noted that the frictional heating of composite C and D developed contrasting results. In the case of composite C, the Vickers hardness value dropped by 4.2%. This is because of the phenomenon of particles removal during wear test as the result of erosion and cavitation. Such a phenomenon was not observed in the case of composite D. For this case, the adhesion between the matrix powders and reinforcement particles were stronger. Hence the reinforcement particles were retained even after the wear test. The influence of frictional heating during the wear test increased its hardness by 6.37%.

4. Conclusion

The sintered materials comprising of the composite containing TiB₂ particles and its hybrid containing both TiB₂ particles and Al₂O₃ revealed density that was lower than that of base alloy alone. Wear test on the sintered composites and its hybrid revealed that addition of reinforcement particles increased the wear rate by 1.22% irrespective of the quantity and type of reinforcements. Hybrid composites performed better for a short duration during the wear test but contributed to increasing the wear rates after de-bonding of the reinforcement particles. Increasing the quantity of TiB₂ particles was detrimental to the wear resistance property of MMC. Addition of alumina particles improved the recrystallization of the matrix element, but the presence of TiB₂ particles affected the bonding with the matrix element. Hardness test revealed that the sintering process alone is not sufficient to cause any noticeable change in the mechanical property. However, the influence of friction during the wear test increased the hardness of the composites and its hybrid. It is concluded that the 10 wt. % TiB₂ with 2 wt. % of Al₂O₃ particles as the reinforcements increases the hardness of hybrid composite materials. The hardness further enhances due to the effect of friction developed when it slides over surfaces. Such materials are applied for brake pads, cotter pins, nuts and bushes.

5. References

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