Hardness and Wear properties of Al-AlN Composite Produced using Powder Metallurgy Route

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Abstract:

Aluminium nitride (AlN) is known for its enviable qualities which include; good wear and corrosion resistance, low coefficient of thermal action, low resistivity, wide band gab similar to that of silicon and other properties. For these reasons, the use of AlN to form composite material with aluminium matrix has been employed using powder metallurgy route. Non-wetting of AlN reinforcement by aluminium matrix during sintering, however, has continued to be a subject of concerned, hence necessitating the use of magnesium as a wetting agent. In this paper, the effect magnesium addition on hardness and wear resistance properties of Al-AlN composite has been investigated and presented.

Keywords: Al-AlN Composite, Wear, hardness, wetting agent, Powder metallurgy, SEM

I. Introduction

The enviable properties of aluminium nitride are responsible for its choice for applications in areas such as; automobiles, aerospace, electronic industry, refractories, optical and optoelectronics devices and several other areas [1-4]. Despite all its properties and wide areas of applications, AlN is hardly ever used alone for any applications, but is rather used in conjunction with other matrices as reinforcement to produce composite materials. It has been used severally as reinforcement in aluminium matrix to form particle reinforced aluminium matrix composite [5-7]. As reinforcement, it induces strength, chemical and thermal stability, hardness and wear resistance.

Several production processes have been employed to produce composite using AIN as reinforcement: Stir casting [5, 8, 9], infiltration method [10, 11], in-situ method [7, 12, 13] and powder metallurgy route [6, 14, 15]. Powder metallurgy route has continued to dominate the production of Al-AIN composite due to its ability to enhance uniform distribution of reinforcement in the matrix and due to the relative simplicity of the process.

The major challenge usually encountered when employing this method is the wettability of AlN by aluminium matrix [16]. The main reason for this difficulty in wetting is the unavoidable presence of thin aluminium oxide on the surface of aluminium powder due to its high affinity for oxygen [17]. The use of magnesium as a wetting agent has been employed as a solution to wetting problems [18]; it facilitates bonding between the matrix and reinforcement which also influences other properties of this composite positively or otherwise.

In this paper, however, the effects of weight fractions of AlN reinforcement and magnesium additions on wear and hardness properties of Al-AlN composite are the main focus.

II. Materials and Method:

The raw materials used for the production of Al-AlN composite include: aluminium, aluminium nitride, and magnesium powders all sourced from Bumi Padu Solution Sdn. Bhd. Kuala Lumpur, Malaysia. Other materials used were Oxygen free nitrogen (OFN) and hydrogen gas. The equipment used in this study would be mentioned as the work is being presented.

A. Composite production

In order to produce this composite of different compositions, aluminium and aluminium nitride powders were weighed in different proportions and properly mixed together in a mortar for about 15 minutes. The different compositions of AlN reinforcement in the composite were thus; 0, 6.5, 9.75, 13.0 and 16.25%. The amount of magnesium added into the composite ranges from 0.5% to 2.0%.

The mixed powders were poured into a tool steel mould of diameter 10.2mm and pressed under 4.5 tons load using Carver Compaction Machine, Model 3851-0. The green compacts produced where charged into stainless steel tube furnace and sintered (two step sintering) at 620 $^{\circ}$ C for 30 minutes in nitrogen atmosphere. The details of how this composite was produced has already been reported in one of our journal paper on liquid exudation during sintering which is still under review. The sintered compacts were then tested for density using AccuPyc II 1340 Gas Pycnometer to certify that the compacts meet the acceptable range of sinteribilities.

B. Vickers Hardness Test

The sintered samples produced were tested for hardness using Vickers Hardness Tester; model FV-700e. A load of 98N was applied on the well prepared surfaces of the compacts for a dwell time of 10 seconds. The diagonals of the diamond indenter were measured in each case and the hardness numbers displayed on the screen were recorded.

C. Wear Test

In order to investigate the wear property of the compacts, a pin-to-disc tribometer was employed using METKON wear testing machine, Model GP04 (Fig. 1). The counter body made of stainless steel rotating disc was polished and fastened to the base of the machine. The surfaces of the compacts to be used for the wear test were abraded with 800 emery paper and their initial weights recorded. The first specimen (pin) was fastened to the upper portion of the machine. The pressure or load of 5 Newton was released to pin the test specimens to the disc at a radial distance of 70mm from the centre of the rotating disc spinning with the speed of 200rpm. The tests were run for sliding time of 20 minutes. The weight losses were recorded.



Figure 1. METKON wear testing machine, Model GP04

The wear volumes, V were determined using the relationship [19];

$$V = \frac{W}{\rho} - \cdots$$
 (1)

Where; ρ is density of materials (g/cm³) and W is weight loss in grams.

The wear rate r, at the other hand, was determined using the formula [20]:

$$r = \frac{W}{t} - \frac{W}{t}$$
 (2)

Where, t is the wear time in minutes.

D. SEM Analysis

SEM machine was used to study the wear surfaces and to determine the mechanism of wear that has taken place on the surfaces of the compacts.

III. Results and Discussions

The sintered densities of the samples used were within the acceptable range though the density results are not presented in this work. The results for hardness and wear tests conducted on the compacts are hereby presented.

A. Hardness Results

The main interest in this test is to determine the influence of weight fraction of reinforcement added and that of magnesium addition on hardness of the sintered compacts. Figure 2 below gives at a glance the relationship between weight fractions of AlN reinforcement added into the composite and the hardness values of the sintered compacts.



Figure 2. Effect of amount of reinforcement on hardness of Al-AlN composite

Figure 2 shows that the unreinforced aluminium recorded the lowest hardness value equivalent to 37.25 Hv, while the composition with 16.25% AlN reinforcement recorded the highest hardness value equivalent to 47.46Hv. The low hardness value of unreinforced aluminium is not unexpected due to its soft and ductile nature. With increase in AlN reinforcements, the aluminium matrix is

strengthened; consequently causing increase in hardness values [8, 21]. With addition of 0.5 and 1.0% magnesium into the composite prior to pressing and sintering, the new hardness values of the compacts were improved as presented in Fig. 3.



Figure 3. Join effects of amounts of reinforcement and magnesium additions on hardness values of Al-AlN composite

The inevitable role of magnesium during processing of aluminium matrix composite has been reported in [22] where it was stated that magnesium is a powerful surfactant capable of reducing the unavoidable aluminium oxide film to free aluminium hence enhancing bonding and densification of the compacts during sintering. From the results in Fig. 3, therefore, it can be observed that magnesium addition brought about slight increase in hardness values of the compacts.

The increase in hardness values were not much probably due to the small amounts of the reinforcements used compared with the large volume of soft aluminium matrix. In order for volume fraction of AlN to bring about a significant increase in hardness, reference [23] has suggested that SiC be added into the aluminium matrix where higher strength is needed.

B. Wear Volume

During this investigation, the test specimens (pins) with diameter 10.2mm were pinned to the stainless steel rotating disc (200rpm) with the force of 5N. With 70mm radial distance from the centre of the rotating disc and wear time of 20 minutes; it means that the total distance travelled by each specimen was about 1.76 kilometres. The test specimens were ran such that they travelled equal distances.



Figure 4. Effect of increase in weight fraction of AlN reinforcement on wear volume of Al-AlN composite

From Figure 4, it can be shown that the unreinforced aluminium recorded the highest value of wear volume equivalent to $4.06 \times 10^{-2} \text{cm}^3$, while the compact containing 16.25% AlN gave the lowest wear volume equivalent to $2.91 \times 10^{-2} \text{cm}^3$. The high wear volume associated with the unreinforced aluminium could be attributed to the lack of hard ceramic particles of AlN in the matrix. However, the results show a decrease in wear volume with the amount of reinforcement added. This is also expected because as more of AlN reinforcement is added, the matrix is strengthened the more; hence increasing the wear resistance of the composite as supported by the findings reported in [24] and [25]. Reference [26], however, noted that the use of graphite as a reinforcement, though it does not necessarily impart strength and hardness, its lubricating property rather than hardness was helpful in reducing the wear rate as its weight fraction increases in the composite.



Figure 5. Effect composition of AlN and Mg addition on wear volume of Al-AlN composite produced

With magnesium additions as in Fig. 5, the reduction in wear volume was more in compositions with 1wt%Mg than in those with 0.5wt%Mg though the difference was very small. The influence of magnesium which enhances densification, reduces pores volume and enhances strength after sintering is responsible for this increase as reported in [27]. The result obtained in this study suggests that where Al-AlN composite is needed for wear application purpose, it is wise to add magnesium during the production process in order to improve the wear resistance of the composite.

C. Wear Rate

The wear rate is the amount of material that is being worn out per unit time. Figure 6 presents the wear rate of Al-AlN compacts produced with and without magnesium additions for the purpose of comparison.



Figure 6. The effects amount of AlN reinforcement and magnesium addition on wear rate of Al-AlN composite

From Figure 6, it can be observed that the variations in wear rate with compositions of AlN and magnesium additions follow similar trend with those of wear volume since both of them depend on the amount of weight loss. Comparing the results in Figure 6 with those of Figure 5 shows that there is a correlation between wear volume and wear rate: both parameters decrease with weight fraction of AlN reinforcement added and with magnesium additions. This is true because both parameters are determined as functions of the weight losses observed during the tests. Now comparing the results in Figures 3, 5 and 6; suggests that the hardness values of the compacts have inverse relationship with those of wear volumes and wear rates. This means that as the amount of reinforcement in the compacts increases, the hardness values increase while the values of wear rate and wear volumes decrease.

D. SEM analysis of wear surfaces

In order to study the nature of worn surfaces and the mechanism of wear that has taken place on the compacts, let us observe the SEM analysis of the surfaces.

Figure 7 (A) represents the SEM image of 100% aluminium compact after wear. The image shows the direction of wear, de-lamination, wear tract and incipient fusion of debris (point k). The severe wear damage observed in the figure could be due to: lack of reinforced particles necessary to increase the wear resistance and due to the action of debris pinned to the disc during the wear which are the reasons for the deep groove or wear tracks on the surface as also observed in [24].



Figure 7: SEM Images showing wear for: (A) 100% Al (B) Al-6.5wt% AlN (C) Al-6.5% AlN-1% Mg, (D) Al-6.5AlN-2Mg

The soft and ductile nature of aluminium, lack of reinforced particles in its matrix and the action debris which contributed in roughening of the surface are responsible for the high wear volume and wear rate of the unreinforced aluminium compact. During the test on this sample (100% Al), the pin which was free to move along the circumference of the rotating disc (up to 180^{-0}) due to the design of the machine, caused turbulence and change in the direction of wear hence the reason for the high value of wear recorded and the rough surface observed in this sample.

Considering the wear surface of Al-6.5% AlN (Figure 7 (B)) and that of Al-6.5% AlN-1% Mg (Figure 7 (c)); the presence of AlN reinforcement helped in increasing the wear volumes/rates of the matrix hence reducing the wear volumes/rates of these compacts; and their wear surfaces were not as badly damage as that of the unreinforced aluminium. The sintered compact containing 6.5% AlN-1% Mg (Figure 7 (C)) was also observed to have better surface after wear than that of Figure 7 (B), possibly due to the presence of 1% Mg in the former.

The turbulence that occurred during wear of the unreinforced compact (Fig. 7(A)) which caused changes in direction of wear and roughness of the surface repeated itself during wear of the sample represented by the image in Figure 8 (b).



Figure 8: SEM Images showing wear for: (a)Al-13wt%AlN-1Mg (b) Al-13%AlN (c) Al-13AlN-2Mg (d) Al-16wt%AlN-1Mg

The wear surface of the compact in Figure 8(b) with composition Al-13% AlN is abnormally rough. Considering the amount of reinforcement in the compact (13% AlN), it would be expected the sample would have smoother surface similar to the ones in Fig.8 (a) or (c). The unusual movement of the pin during the test must have led to an abnormal increase in wear volume and wear rate of this sample. The point denoted 'J' on Figure 8 (b) indicates point where the debris pulled out of the surface is welded back on the matrix due pressure between the pin and the disc coupled with the high temperature generated as a result of friction between the two surfaces. In Figure 8 (C) and (D), the relatively smoother surface observed suggest that the there was steady wear and an indication that the compacts became harder as the weight fraction of reinforcement increases and with magnesium additions. The wear surfaces of compacts shown in Figure 8 (C) containing 16.25% AlN and that of Figure 8 (D) containing 13AlN-2%Mg look similar suggesting that the two surfaces had good resistance to wear. When closely observed, it will be seen that the depth of wear tracks in both surfaces under consideration are equally similar. The presence of 2%Mg in the compact shown in Fig. 8(D) must have balance for the shortfall in amount of reinforcement which is 3.25% less.

From the SEM images presented above, it could be inferred that the mechanism of wear in the compacts

were dominated by abrasion, adhesion and delamination as also observed in [28]. The wear rates observed also suggest that the severity of wear on the compacts were inversely proportional to the amounts of AlN reinforcement present in them (except where unsteady wear occurred due to other factors) as reported in [25].

Reference [21], enumerated the factors affecting wear rate thus: amount of reinforcement added, sliding speed, sliding distance covered and the load applied; and concluded that the sliding distance affect the wear rate the most while the weight fraction of reinforcement affects wear the least. Reference [29] and [30] studied the relationship between sliding speed and coefficient of friction between two moving bodies and how they influence wear rate and both concluded that higher sliding speed reduces the contact relationship between the two bodies in contact hence, reducing the coefficient of friction between the two bodies and consequently, the wear rate. Therefore, with choice of 200rpm which is quite low, the coefficient of friction between the two surfaces during wear will be high. That must have influenced the different wear mechanisms involved and the unusual wear observed in some of the samples due to movement of the pin along the circumference of the rotating disc.

IV. Conclusions

From the hardness values, wear volumes, wear rates and SEM images of the wear surfaces observed in this study, the following conclusions could be drawn:

- a) The hardness of Al-AlN compacts produced increased with amount of reinforcement added. The unreinforced aluminium compact recorded the lowest hardness value. Addition of magnesium into the compacts improved the harness of the compacts.
- b) The wear rates and volumes of the samples decreased with amount of reinforcement added into the compacts. Addition of magnesium into the improved the wear resistance of the compacts.
- c) The results showed an inverse relationship between hardness and wear resistance: the higher the hardness values of the compacts, the lower their corresponding wear volumes and wear rates.
- d) The SEM analysis suggests that the mechanisms of wear on the compacts were dominated by abrasion, adhesion and de-lamination; while the natures of the wear surfaces and wear rates observed suggest that the severity of wear on

the compacts were inversely proportional to the amount of AlN reinforcement in them except where abnormal wear occurred due to other factors.

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