

Wear Behavior of Aluminum LM4 Reinforced with WC and Ta/NbC Hybrid Nano-Composites Fabricated Through Powder Metallurgy Technique

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The present investigation aims to understand the wear behavior of aluminum LM4 matrix reinforced with the different weight percentage of tungsten carbide and tantalum-niobium carbide (0.5, 1.0, 1.5 and 2wt %) hard particles. Composite Specimens prepared by powder metallurgy route followed by cold pressing and hot sintering process. The wear tests were carried out using pin-on-disc machine under varying load (10, 20, 30 and 40N) speed (1, 1.5, 2 and 2.5m/s) and sliding distance (400, 600, 800 and 1000m) at room temperature. Characterization was done on nanopowders, developed composites and worn out surfaces of tested samples using Transmission Electron Microscopy (TEM), X-ray diffractometer(XRD), Scanning electron microscopy (SEM), Energy Dispersion Spectroscopy (EDS). The obtained results indicate that higher content of hybrid nanoparticles up to 2wt% in the composite reduces the wear rate up to 43.75% as compared with pure aluminum LM4 alloy.

Keywords: Aluminum, Tungsten carbide, tantalum niobium carbide Powder metallurgy, Scanning Electron Microscope, mechanically mixed layer.

1. INTRODUCTION

The Recent trend in metal matrix hybrid nano-composites (MMHNCs) are getting more and more attractive in the field of automotive and aerospace industries [1-3]. Major automotive and aerospace parts made up of nanocomposite material exhibits superior properties. Metal Matrix Nanocomposites (MMNCs) are the combination of certain metals (Al, Mg, and Cu) as a matrix material and ceramic particles (Carbides, nitrides, and oxides) as a reinforcing material [4-6]. The physical and mechanical properties of the nanocomposite vary from those of the alloys. When compared with the MMCs, the MMNCs have an enhanced property even at a low volume fraction.

Hybrid reinforcement plays a vital role in enhancing the properties of MMNCs. The combination of primary and secondary reinforcements provides improved characteristics which cannot be established by single material [7]. Hard nanoparticles play a significant role in enhancing the overall properties of the composites. Aluminum metal matrix nanocomposites (AMMNCs) have proved superior in the field of automotive due to their high thermal shock resistance and high wear resistance [8].

A lot of research work has been carried out to understand the development of composites using different techniques that were reported in the literature [9]. Conventional stir casting method is one of the cost-effective

processes to develop aluminum matrix composites. But main drawbacks in the stir casting process is clustering of ceramic particles and poor wettability of ceramic particles [10]. To overcome these drawbacks, many researchers illustrated the incorporation of solid-state fabrication techniques such as powder metallurgy method [11]. The literature on the process of fabrication of hybrid nanocomposites was less reported. Effect of hybrid nano reinforcement particles in the enhancement of tribological properties of the composites using different fabrication techniques published in some literature.

Saeid Pournaderi, Farshad Akhlaghi [12] studied the influence of particle size and percentage volume fraction on the wear rate of Al6061-Al₂O₃ composites produced by in-situ powder metallurgy technique. Wear behavior of the developed composites examined under pin-on-disc tester. The test results showed that the best wear resistance was found in composite containing 20 vol% of Al₂O₃ with a mean particle size of 150µm. Further, they concluded that a decrease in particle size and volume content could increase the wear resistance of the developed composites.

Yahya Hisman Celik [13] investigated on hardness and wear behavior of Aluminum reinforced with B₄C particles using a powder metallurgy method. Varying particle size and percentage of reinforcement influence the hardness and wear behavior of the composites. Result reveals that increased B₄C particle percentage increases the hardness of the developed composites, increased hardness can reduce the wear rate. Ravikumar [14] investigated on characterization and mechanical properties of aluminum/tungsten carbide composites. Results revealed that the density and hardness of the composites were increased by increasing reinforcement percentage and there is a strong interface bonding

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between aluminum and tungsten carbide. B.N. Yadav et al. [15] synthesize aluminum-SiC-MWCNTs nanohybrid composites. According to their investigation, the wear properties of composites increase by incorporating the secondary reinforcement. Compared with the other peer findings the composite with single reinforcement the value of microhardness and wear rate are found to be significantly less. But by the incorporation of secondary reinforcement in the composite that enhances the interfacial interaction between matrix and primary reinforcement. Arivukkarasan, V. Dhaalakshmi, B. Stalin et al. [16] reported that aluminum LM4 reinforced with WC by varying the percentage of reinforcement of 5, 10 and 15 weight% using stir casting process. They analyzed the mechanical and tribological behavior of the composite. Finally, an SEM analysis reveals the uniform distribution of WC particles in LM4 alloy. Also, they reported that a decrease in the mass loss for the composite contains 15 wt% of WC during wear test. Lekatou et al. [17] studied the microstructural behavior of aluminum reinforced with WC and TiC nanoparticles. They developed in-situ and ex-situ based composites and observed the wear mechanism based on SEM analysis of the wear surface.

B.V. Manoj Kumar et al. [18] investigated the effect of secondary carbides on the erosive behavior of TiCN-Ni cemented carbides. TiCN-20wt% Ni cements eroded by SiC particles with a mass flow rate of (2.33g/s) at different angles of impingements (300,600,900) on an erosion wear test machine. The results revealed that the secondary carbides exhibit similar behavior like ceramics. R. Van der Merwe et al. [19] worked on the effect of TaC and TiC on the wear behavior of WC-6wt% Co cemented carbides. The result indicates that the addition of less than one weight% TaC was found to improve the wear resistance. They found that the addition of TiC did not provide any improvement in the wear resistance properties. Velickovic et al. [37] reported the overview of tribological properties of nanocomposites with aluminum matrix through equipment used for testing, amount size, and type of reinforcement, process, and test conditions.

Keeping above aspects in view, it is clear that there is a lot of scope for the study on hybrid metal matrix nanocomposites. Also, it is imperative to understand the wear behavior of the developed composites. However, very few studies have been carried out to understand the effect of hybrid nano carbide particles reinforced aluminum metal matrix composites on microhardness and wear measurements. In the present research work, an effort has been made to investigate the wear behavior of aluminum LM4 reinforced with hard nano tungsten carbide (WC) and tantalum-niobium carbide (Ta/NbC) particles.

2. EXPERIMENTAL DETAILS

2.1 Materials and Method

The chemical composition of the Al LM4 powder is 0.3 Cu, 0.5 Si, 0.2 Mg, 0.5 Mn (wt %) and aluminum balance purchased from Chemsearch India with an average particle size of 10 μ m. The WC (average size: ~200 nm) and Ta/NbC (Average size: ~150nm) used as reinforcing particles purchased from Kennametal India.

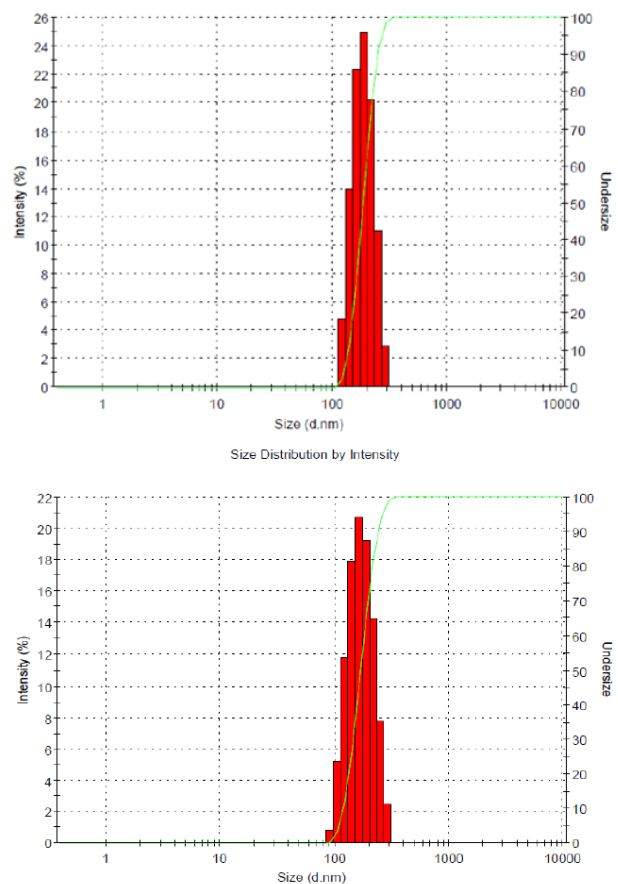


Figure 1. (a&b) Particle size analysis of as received WC and Ta/NbC nano particles.

Figure 1 (a & b) depict the average particle size of the WC and Ta/NbC nanoparticles which are measured using particle size analyzer. From the particle size analysis it is clear that the selected reinforcing particles are well within nano size range. The hybrid nanocomposites developed by varying the percentage of reinforcements from 0.5 to 2wt % in step of 0.5wt% (0.25wt%WC+0.25wt%Ta/NbC) using a powder metallurgy method. A weighed amount of aluminum LM4 powder first milled to reduce the size from micro to a nano using planetary ball mill with a 25:1 ball to nanopowder weight ratio. The time and speed of milling were set to 30 min and 300 RPM respectively. A weighed quantity of preheated Tungsten carbide (WC) and Tantalum Niobium Carbide (Ta/NbC) nanopowders were added to the mixing jar along with aluminum powder to achieve homogenization and proper mixing of matrix and reinforcement particles. A 12 mm dia and 18 mm height pellets developed by using the pellet-pressing machine. The compacted samples then sintered at a sintering temperature of 560 $^{\circ}$ C for 60 min in a vacuum tube furnace under argon gas atmosphere. The final sintered samples were allowed to cool at room temperature for the duration of 3 hours in the furnace itself. The developed specimens were shown in figure 2.

2.2 Dry sliding wear measurements

To understand the tribological properties of the developed composites dry sliding wear test was carried out according to ASTM standard G99-05 using a pin-

on-disc tester. The machine (TR-20LE-PHM400 Ducom instruments) consists of a stationary pin holder arm kept in contact with the counterface of a disc made up of a hardened steel disc of hardness HRC 65. The pin holder arm is connected using a pulley through which the load applied to the pin. The motor controls the speed of the counterface disc.

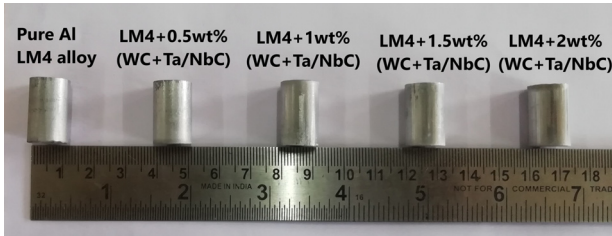


Figure 2. Wear test Specimens

Wear rate of the developed samples determined by taking the initial and final weight. High precision weighing machine with an accuracy of 0.0001g used to measure the difference in weight of the specimens before and after each test. The disc rubbed with emery sheet for better contact between the specimen and disc interface. Then cleaned with acetone to remove the test contaminants. The objective of the present research is to determine the wear rate of the hybrid nanocomposite and comparing the wear result with pure LM4 alloy. The wear rate was calculated by using following equation (1).

$$W = \frac{m}{\rho \times D} \quad (1)$$

where m is the mass loss (Initial weight of the specimen before wear - Final weight of specimen after wear in gram), ρ is the density in gram per mm^3 , D is the sliding distance in meter. W is volumetric wear rate in mm^3/m . The experimental conditions and test parameters to conduct wear test listed in table 1.

Table 1. Experimental condition and parameters selected for wear test.

| Parameters* | levels |
|-----------------------|-----------------------|
| Specimen dimension | 12mm dia, 18mm length |
| % reinforcement | 0.5, 1, 1.5 and 2wt% |
| Applied Load(N) | 10, 20, 30, 40 N |
| Sliding velocity(m/s) | 1, 1.5, 2, 2.5 |
| Sliding distance(m) | 400,600,800 and 1000 |

*In the present work only some combination of test parameters were used to conduct the wear tests. Wear test with all test parameters will be performed in some further work.

3. RESULTS AND DISCUSSION

3.1 Microstructural Examination and Composite Characterization

The morphology of the selected nanopowders and milled Aluminum LM4 powder was examined using HRTEM, SEM analyzer. Figure (3a & b) shows the FESEM images of the WC and Ta/NbC particles. The random morphology of the powder particles witnessed. Figure 3c depicts the FESEM image of the milled Aluminum LM4 particles. Noticed from the figure 3c

that the reduced particle size of the aluminum LM4 after milling.

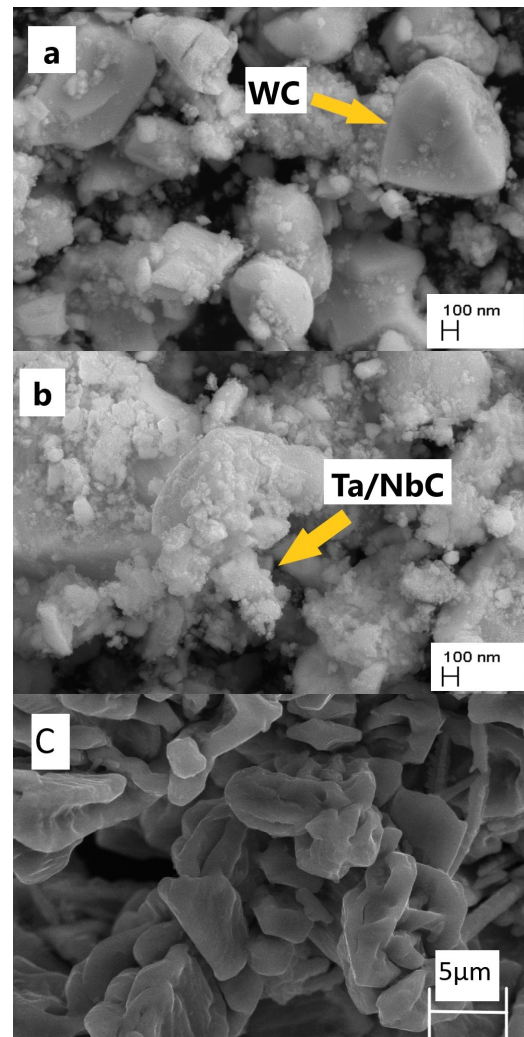


Figure 3. (a&b) FESEM images of as received WC, Ta/NbC nano particles (c) mechanically mixed LM4+ (WC+Ta/NbC) nano particles.

Figure 4a shows the HRTEM dark field images of Al LM4+2wt % (WC+Ta/NbC) nanocomposite powder. Images depict that there is no inter-metallic layer formation at the interface of aluminum LM4 matrix and reinforcement. Figure 4(b) shows HRTEM images of nanocomposite powder showing lattice fringes after ball milling. Inset was the selected area diffraction pattern along with continuous rings, which shows a nanocrystalline nature of the composite powder [20]. The brighter areas in the micrograph represent the nano-sized reinforcement crystal [21].

Microstructure examination carried out on the surface of the developed specimen. Initially, surface preparation made using emery sheets. The sample then polished by using the double disc polisher. For better understanding of surface morphology of the WC and Ta/NbC particles in the LM4 alloy good surface finish was maintained. Keller's reagent was used to etch all the samples before the specimens were tested under Radical RXM-7T advanced metallurgical microscope at different magnifications. Figure 5 shows the microstructure of the fabricated specimens. Figure 5 depicts that the WC and Ta/NbC particles are well distributed through out the alumi-

num matrix material and that is the attribute for the presence of WC and Ta/NbC particles in the developed specimens. The microstructure analysis shows that no agglomeration was observed in the samples. The microscopic examination ensures that the grains are closely packed and there is a strong interface between Aluminum matrix and WC+Ta/NbC reinforcing particles. As it was observed from the micrographs that there was a considerable amount of grain refinement in the specimens.

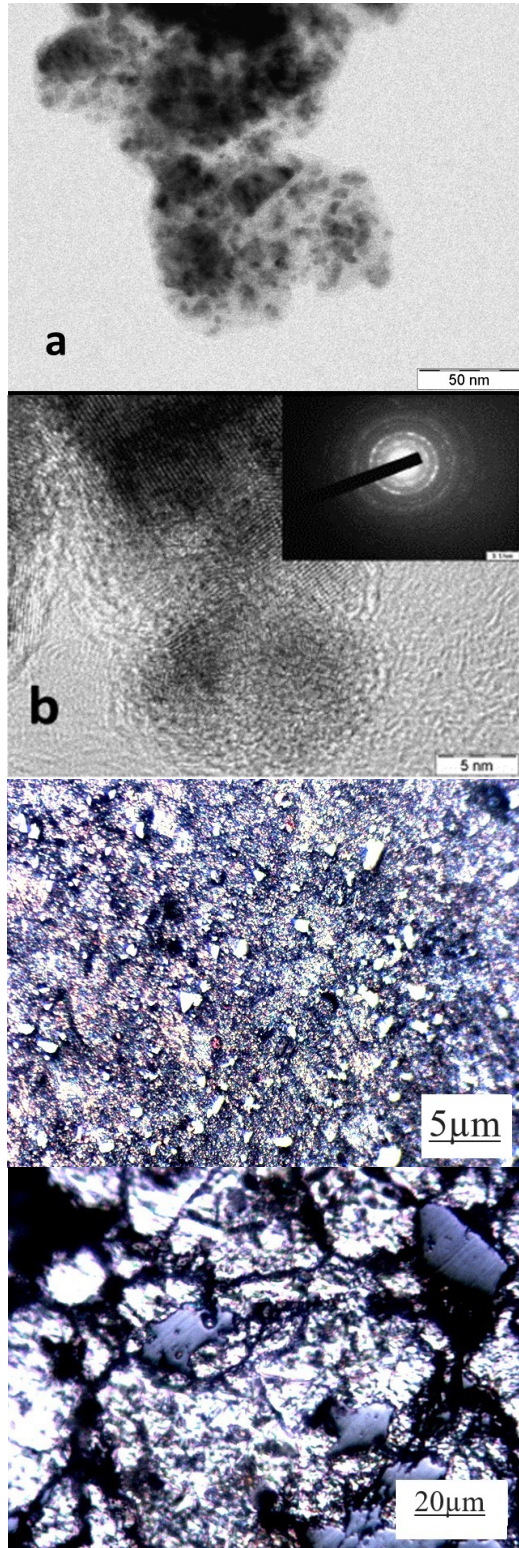


Figure 5. Microstructure of fabricated specimen LM4+1.5wt% (WC+Ta/NbC) and at LM4+2wt% (WC+Ta/NbC) different magnifications.

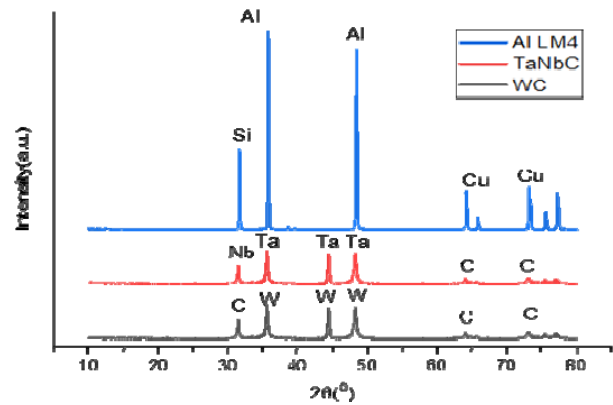


Figure 6. XRD patterns of Al LM4, Ta/NbC and WC nano particles

Figure 6 represents the XRD patterns of the pure WC, Ta/NbC, and Al LM4 matrix nano powder particles. The XRD pattern of the nano crystalline powder exhibits different peaks corresponding to the face centered cubic lattice of Al. The absence of other peaks of matrix elements can be attributed to the limitations of the filtered X-ray to detect phases with amount less than 2% volume fraction [20].

In the present research work only one experiments per each test was conducted and there was no repetition of the tests.

3.2 Effect of Percnetage reinforcement on wear rate of Hybrid nano composites

The effect of reinforcement percentage of wear rate of the developed hybrid nano composites are evaluated. Figure 7 shows the variation of wear rate of hybrid nano composites are reported. The experiment conducted at 20N load and 1.5m/s sliding speed over a 600m sliding distance. It is clear from the graph that wear rate decreases with an increase in the percentage of WC+Ta/NbC particles. The improvement in the wear properties may due to the hardness provided by the WC particles [40]. Further improvement in the wear resistance properties observed due to the addition of secondary reinforcement Ta/NbC particles.

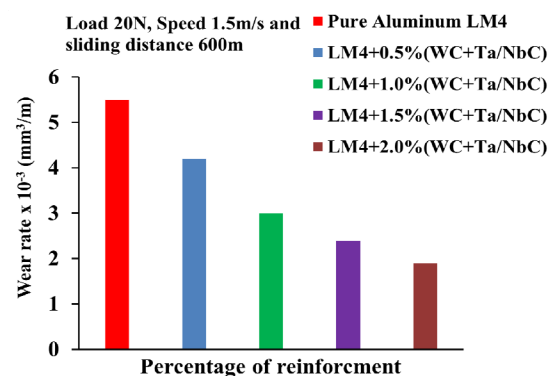


Figure 7. Effect of reinforcement percentage on wear rate of LM4+(WC+Ta/NbC) hybrid nano composites.

Similar observations were made by Venkataraman B and Sundararajan G [23]. The pure LM4 alloy exhibits an average wear rate of around $5.5 \times 10^{-3} \text{ mm}^3/\text{m}$. LM4+2wt % (WC+Ta/NbC) shows less wear rate of about $2 \times 10^{-3} \text{ mm}^3/\text{m}$. Improved wear behavior of the

hybrid nanocomposites due to restriction in plastic deformation during sliding wear by the high hardness provided by WC and Ta/NbC hard particles. SEM images of Worn out surfaces are the evidence for the restricted grooves and improved surface properties. Also, strong interface bonding between matrix and reinforcement particles is one of the reasons for improved wear resistance on the developed composites.

3.3 Effect of sliding velocity

The effect of sliding velocity on the wear rate of the fabricated hybrid nanocomposites and matrix material explained and presented in figure 6. The experiment conducted at 20N load and 600 m sliding distance over 1, 1.5, 2, 2.5 m/s speed. The wear rate was strongly related to the sliding speed reported in some literature [24-26]. The wear rate of LM4+2wt % (WC+Ta/NbC) exhibits improved wear resistance as compared with the other percentage of reinforcements. The wear rate of LM4+2wt % (WC+Ta/NbC) varies from $1.6 \times 10^{-3} \text{ mm}^3/\text{m}$ to $2.6 \times 10^{-3} \text{ mm}^3/\text{m}$ with an increase in the sliding velocity from 1 m/s to 2.5m/s respectively. The composite LM4+2wt % (WC+Ta/NbC) exhibits improved wear resistance of 33.67% when compared with remaining weight percentages. It can also highlight that at low velocity the formation of oxide layer was stable and that makes the less wear rate during initial conditions of around $1.6 \times 10^{-3} \text{ mm}^3/\text{m}$. However, as the sliding speed increases the wear rate increases rapidly of around $4.5 \times 10^{-3} \text{ mm}^3/\text{m}$. When rubbing surface temperature increases, the bonding between matrix and reinforcement decreases, which enhances the wear loss. Also, due to the micromachining effect of hard nano-reinforced particles removes the oxide layer that leads to the direct metal-to-metal contact makes rapid metal removal rate [27].

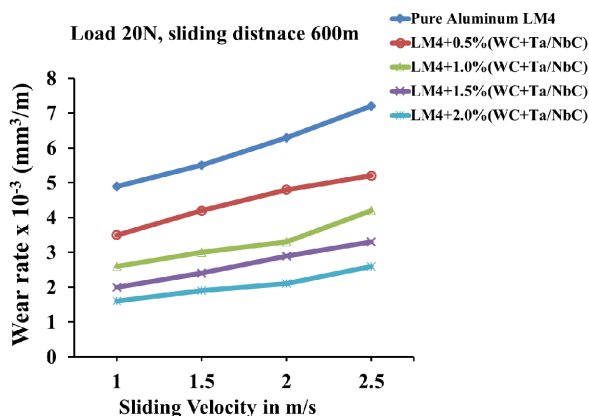


Figure 8. Effect of sliding velocity on wear rate of LM4 + (WC+Ta/NbC) hybrid nano composites.

3.4 Effect of sliding distance on wear loss

Figure 9 shows the effect of sliding distance on the wear rate of the Aluminum LM4 alloy along with synthesized hybrid composite specimens. The experiments conducted at 20N load and 2.5 m/s sliding speed over sliding distance of 400m, 600m, 800m and 1000m on each sample. The result obtained as a function of sliding distance over a specific load and speed in the form of wear rate.

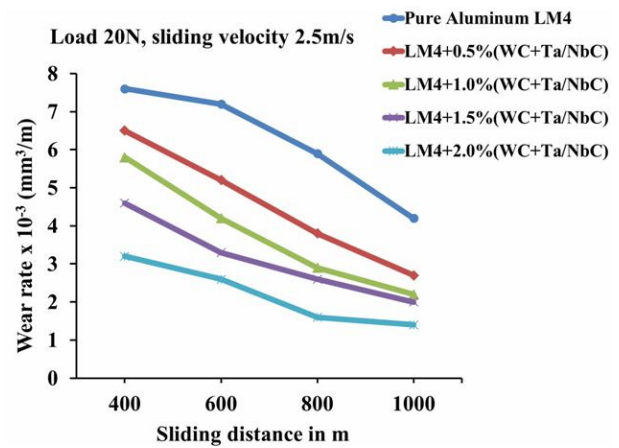


Figure 9. Effect of sliding distance on wear loss of LM4+(WC+Ta/NbC) hybrid nano composites

Obtained wear rates are in correlation with the result obtained by [39]. From the figure 9 as sliding distance increase wear rate decreases gradually in all the developed specimens. It can be seen that pure Al LM4 sample exhibits more wear rate as compared with hybrid specimens. The reason for the decrease in wear rate of hybrid samples due to the hard reinforcement particles. A stable mechanically mixed layer (MML) formed from the fragmented WC and Ta/NbC nanoparticles, iron oxide from the counter body and aluminum oxide from the pin. The MML acts as a protecting layer between pin and counter surface. During increase in the sliding distance the wear debris generated from the pin and entrapped between the pin and disc that prevents the further wear. Also due to constant applied load and speed, abrasiveness on the surface of the pin decreases and become smooth surface and that reduces the further wear.

3.5 Effect of applied load

Figure 10 shows the variation of the wear rate of Aluminum LM4 along with hybrid nanocomposites against an applied load. The experiments conducted at a constant sliding distance of 600m and 2.5m/s sliding velocity, over applied load of 10N, 20N, 30N and 40N. The graph shows at constant sliding speed, wear rate increases with increase in applied load. The composite LM4+2wt % (WC+Ta/NbC) shows higher wear resistance over the remaining percentages. WC and Ta/NbC particles act as load bearing elements due to high density and hardness. During increased loading condition a large amount of plastic deformation and delamination occurs on the rubbing surface leads to distortion of the oxide layer, spalling and surface fracture takes place. As the applied load exceeds, high rate of metal removal and grass damage occurs on the rubbing surfaces [29-31]. From results, the wear rate of LM4+2wt % (WC+Ta/NbC) hybrid nanocomposites varies from $1.8 \times 10^{-3} \text{ mm}^3/\text{m}$ to $4.2 \times 10^{-3} \text{ mm}^3/\text{m}$, whereas wear rate of LM4+1.5wt % (WC+Ta/NbC) ranges from $2.2 \times 10^{-3} \text{ mm}^3/\text{m}$ to $5.8 \times 10^{-3} \text{ mm}^3/\text{m}$ under applied load from 10N to 40N respectively. Result reveals that as the applied load increases the wear rate of the composite increases. Further, noted that pure aluminum alloy exhibits increased wear rate as compared to the hybrid composites.

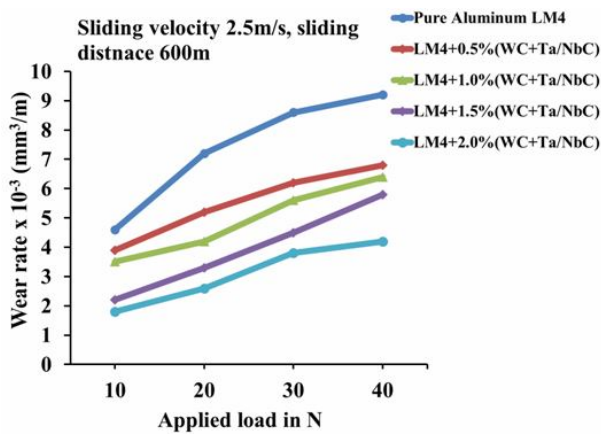


Figure 10. Effect of applied load on wear rate of LM4+(WC+Ta/NbC) hybrid nano composites.

4. SEM ANALYSIS OF WORN SURFACE

The worn surface morphology of all fabricated hybrid composites and Al LM4 samples analyzed using scanning electron microscopy. Incorporation of hybrid nano WC and Ta/NbC reinforcement into Aluminum alloy exhibits some changes in surface morphology as compared with the worn surface of the pure alloy. Restricted grooves and scratches noticed on the worn surface. These are the clear indicators of abrasive action of hard particles.

The higher hardness value imparted by the LM4+2wt% (WC+Ta/NbC) considered one of the reason for the improved wear resistance property. As the sliding continues, the wear debris are detached from the surface with similar chemical composition. The morphology of debris and the corresponding worn surface which has generated during an applied load of 20N for LM4+2wt% (WC+Ta/NbC) shown in figure 13. The larger debris evidence for the severe crack at high speed and load. The average size of the most wear products are around 30µm.

In some cases, severe level of wear occurs due to an increase in load and speed between the contacting surfaces. When Stress between the pin and disc are too large, the contact becomes unstable and larger particles break away from the contact area. These particles larger than 50µm in size and these wear particles mainly originate from the pin material [38].Figure 13 depict such larger debris originated from the specimen and that is the evidence for the severe wear.

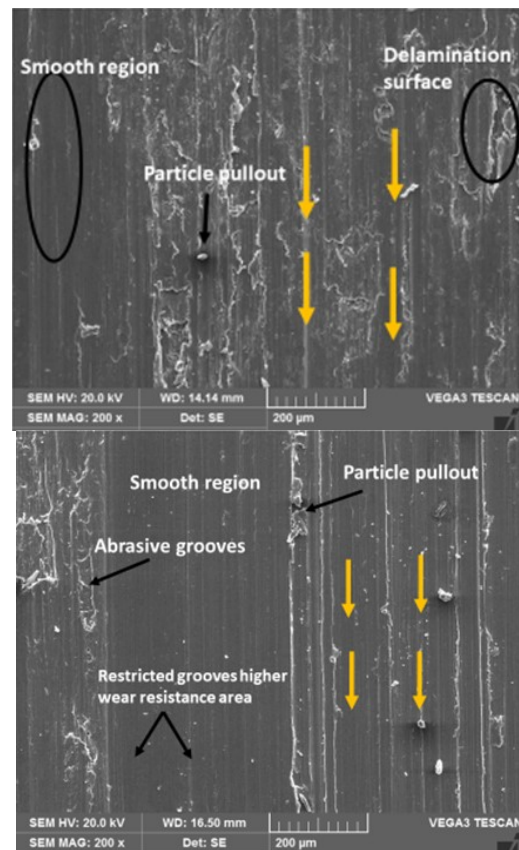
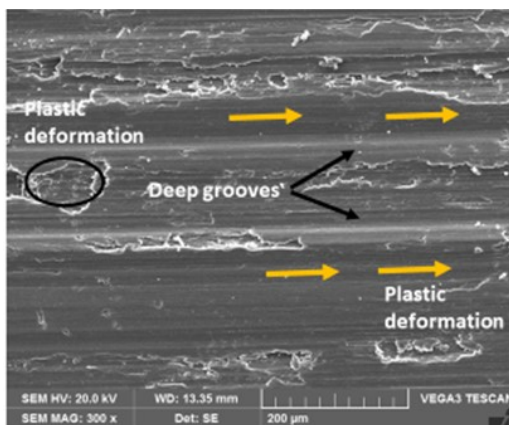


Figure 12. The worn surface morphology of LM4+(WC+Ta/NbC) composites (a) 2.5m/s and 20N load showing deep grooves and scratches. (b) 2 m/s and 600 m sliding distance at 40 N load showing delamination surface and smooth regions and (c) 0.5m/s and 10N load showing abrasive grooves.

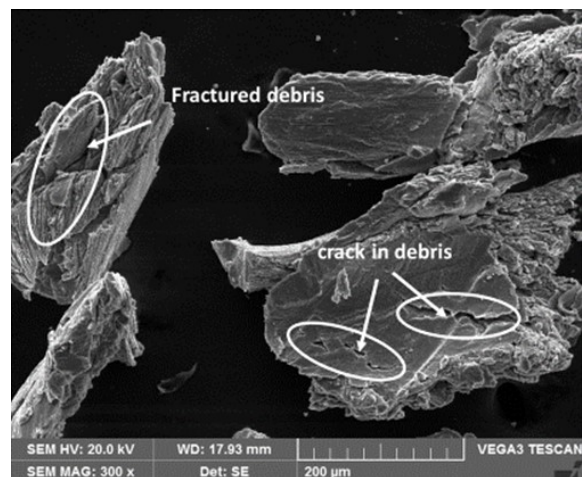


Figure 13. SEM morphology of debris generated at 20N for LM4+2wt% (WC+Ta/NbC) composite.

Figure 14 shows the EDS analysis of the worn out surface of LM4+2wt% (WC+Ta/NbC) composite tested at 20N load. The EDS results shows that the presence of intermetallic components on the surface of the worn out specimen. A Negligible amount of iron element which deployed from the counter surface is evidence for the abrasive wear. Also presence of small amount hard reinforcing particles WC and Ta/NbC are the evidence for the presence of reinforcing particles in the specimen. Further increase in applied load increases the amount of the counter face element peaks in the EDS graph.

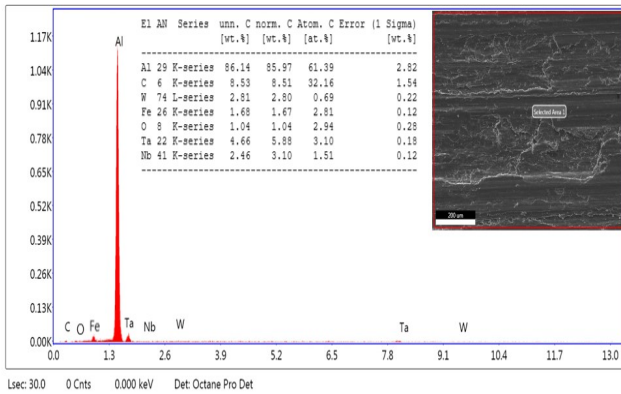


Figure 14. EDS analysis of hybrid nano composite LM4+2wt% (WC+Ta/NbC) at 20N

5. CONCLUSIONS

The present studies on dry sliding wear behavior of Aluminum LM4 alloy reinforced hybrid nanocomposites draws the following influential conclusions.

1. Aluminum-based hybrid nanocomposites reinforced with WC and Ta/NbC successfully developed by powder metallurgy technique.
2. Scanning and transmission electron microscopy studies showed that a uniform dispersion of WC and Ta/NbC particles with good intermetallic bonding with Aluminum LM4 matrix with a minimal amount of porosity.
3. From the dry sliding wear studies, it is evident that the incorporation of nano-sized WC and Ta/NbC particles into the aluminum matrix alloy increases the wear resistance properties up to 45% as compared with the dry sliding behavior of pure aluminum LM4 alloy.
4. Dry sliding wear results revealed that the parameters like varying weight percentage, sliding velocity, and applied load are the direct impact on the wear behavior of the hybrid nanocomposites.
5. A reduction of up to 38% wear rate was observed by incorporation of 2wt% (WC+Ta/NbC) nanoparticles as compared with the remaining percentage of reinforcements.
6. At the same time, the sliding speed and load are directly proportional to the wear rate of the hybrid nanocomposites. As the sliding speed and load increases, the wear rate also increases drastically.
7. Worn out analysis by using SEM revealed that the presence of different wear mechanisms like delamination, scratches, and grooves. EDS result confirm that presence of intermetallic compounds deployed from counter surface.

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**ХАБАЊЕ ЛЕГУРЕ АЛУМИНИЈУМА LM4
ОЈАЧАНЕ СА WС И Та/NbС ХИБРИДНИМ
НАНО КОМПОЗИТИМА ПРОИЗВЕДЕНИМ
ТЕХНИКОМ ПРАШКАСТЕ МЕТАЛУРГИЈЕ**

Сачит Т.С., Н. Мохан

Циљ истраживања је изучавање природе хабања композита са металном матрицом Al LM4 при различитим тежинским процентима тврдих честица волфрамкарбида и танталниобијумкарбида (0,5; 1,0; 1,5 и 2 тежинска %). Узорци композита припрем-

љени су прашкастом металургијом а потом хладним пресовањем и врућим синтеровањем. Испитивање хабања обављено је на пин-он-дису трибометру са различитим оптерећењем (10, 20, 30 и 40N) и клизним растојањем (400, 600, 800 и 1000м) на собној температури. Карактеризација је обављена на нанопрашковима, развијеним композитима и похабаном површинама испитаних узорака помоћу техника TEM, XRD, SEM EDS. Добијени резултати показују да већи садржај хибридних наночестица до 2 тежинска процента у композитима смањује брзину хабања до 43,75% у поређењу са чистом легуром алуминијума LM4.