

Wear Characterization of Aluminum Matrix Hybrid Composites Reinforced with Nanoparticles of Al_2O_3 and TiO_2

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Abstract: In the present work, the dry sliding wear behavior of Al-12wt%Si matrix composites reinforced with hybrid addition of 2, 4 and 6wt.% ($\text{Al}_2\text{O}_3 + \text{TiO}_2$) nano particles were investigated. All nanocomposites samples were fabricated by powder metallurgy and mechanical milling of micro powders of the base alloy (Al-12wt.%Si) and nanopowders of Al_2O_3 and TiO_2 , followed by cold pressing at 100 bar and sintering at 520 °C for 90 min. Archimedes technique was used to measure the density of sintered samples and porosity calculated as physical tests of sintered samples. Also AFM (Atomic force microscope), SEM (Scanning electron microscope) were used to investigate the morphology of mixed powders and nanocomposites samples. Pin – on disk wear tests were carried out at room temperature under dry sliding conditions with using different normal loads and sliding times. Worn surface micrographs were investigated based on the optical and scanning electron microscopy observations of wear tracks and wear debris morphology. It has been found that the hybrid nanocomposite with 6wt.% ($\text{Al}_2\text{O}_3 + \text{TiO}_2$) nanoparticles shows the highest hardness than other nanocomposites. Nanocomposites show lower wear rate (better wear resistance) than the base alloy within the same conditions.

Key words: Nano composites, powder metallurgy, Al-alloy, wear resistance.

1. Introduction

Recent investigations have revealed that further improvements in the wear resistance of AMCs (Aluminum matrix composites) can be achieved by synthesizing nanocomposites where hard nanoparticles are embedded in aluminum matrix [1]. In recent studies the hybrid composite refers to double or triple of oxides or carbides addition to get new properties.

Alumina (Al_2O_3) is the most useful oxide ceramics, therefore it has been used in many fields of engineering such as coatings, heat-resistant materials, abrasive grains, cutting materials and advanced ceramics [2]. Solid-state by PM (Powder metallurgy), mechanical milling is especially used to produce nanocomposites with the reinforcement of

nanoparticles. In mechanical alloying by ball mill the particles are expected to fracture again into submicron matrix particles with fine dispersion of the reinforcement phase. Those stages could occur repeatedly if the process continues and this was proven by scanning electron microscope which could easily track the particle morphology at different milling times [3, 4].

Fathabadi [5] investigated nanocomposite coatings fabricated by thermal sprayed $\text{Al}_2\text{O}_3\text{-xTiO}_2$ ($x = 0, 3, 13$ and 20wt.%). The addition was to the matrix of Al_2O_3 in a form of nano-particles. The most important conclusions are enhanced hardness for nanostructured coatings associated with high residual compressive stresses. Jung Ho Ahn et al. [6] prepared the Al/AlN particulate composite using powder metallurgy. The bi-modal particulate composite of Al/AlN, consists of nano-sized AlN dispersoids (10 ~ 50 nm) particles embedded in the fine grained Al matrix (grain size 0.3

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$\sim 1 \mu\text{m}$).

Hao Yu [7] used alloy A356 as matrix and reinforced by silicon carbide nano-particles and Tantalum (Ta) nano sized powder, the result 22% hardness improvement was achieved.

Alizadeh et al. [8] investigated the wear properties of a nanostructured matrix of Al prepared via mechanical milling and hot extrusion before and after incorporation of B_4C nanoparticles. Mechanical milling was used to prepare the nanocomposite samples by addition of 2 and 4 wt.% of B_4C nanoparticles into the Al matrix. A pin on-disc setup was used to evaluate the wear properties of the hot extruded samples under dry condition. They revealed a lower friction coefficient and a lower wear rate for the structured matrix of Al in contrast to a commercial coarse grained Al matrix. Mazahery et al. [9] used zircon particles reinforced aluminum matrix composite which was fabricated by a powder metallurgy process. They used different volume percentages of zircon (2.5, 5, 7.5, 10, 12.5 and 15) with average particle size of $20 \mu\text{m}$ were added to aluminum powders ($33 \mu\text{m}$) by means of a planetary ball mill without protective atmosphere and with 1% zinc stearate applied as lubricant. The samples were compacted at CIP (Cold isostatic press) of 440 MPa and sintered at 600°C under argon atmosphere for 65 min. Sliding wear tests were conducted in pin-on-disc wear testing apparatus under varying applied loads. It was noted that the weight loss of the composites was less than that of unreinforced alloy, increases with increase in sliding distance, and has a declining trend with increasing the particles volume fraction.

The aim of the present research is to study the effect of hybrid addition of Al_2O_3 and TiO_2 nanoparticles on wear characteristics of Al-12wt.%Si matrix composites and microstructural characteristics of worn surfaces by using optical and SEM were also investigated.

2. Experimental Work

The materials used for preparation the MMNCs

(Metal matrix nanocomposites) are $25 \mu\text{m}$ of Al powder, $25 \mu\text{m}$ of Si powder have 99.98% and 98.5% of purity respectively, and two types of nano particles of 50 nm of gamma- Al_2O_3 and 30nm of rutile- TiO_2 have 99.98% and 99.8% of purity respectively. Dry mixing method was used to mix the powder of alloy (Al-12wt.%Si) with nano particles of (Al_2O_3 and TiO_2). Rotating balls mill with 20mm diameter of alumina balls was used for mixing these powders together in order to obtain a good dispersion. The powders mixing was performed by putting the 10gm powder of alloy (Al-12%Si) in a balls mill in 20 : 1 together with different weight percent of the hybrid nano particles ($\text{Al}_2\text{O}_3 + \text{TiO}_2$) with 2, 4 and 6wt.% will add. The mixing time was for 4 hours with using speed of 650 rpm in dry jar to get good particles distribution. Cold compaction was carried out at 10 Mpa pressure followed by sintering process at 520°C for 90 min by using electric tube furnace with argon flow rate 2 L/min.

XRD (X-ray diffraction) was used to characterizing the purity of aluminum powder also used after mixing with silicon and sintering, as shown in Figs. 1 and 2. Many tests and inspections were performed including: density and porosity measurements; hardness test and wear test under dry sliding condition. Microstructure tests by optical microscope and SEM (Scanning electron microscope), and AFM (Atomic force microscope) of selected samples are also investigated. Micro hardness Vickers tester was used to measure hardness of all samples. The applied load was 1.96 N for loading time of 15 sec. Three -five indentations were made on each specimen surface and the average reading was taken to find the Vickers hardness. The final densities and porosities (after sintering) of samples were measured according to ASTM D 792 standard which is based on Archimedes principle. The specific gravity of material is given by Eq. (1) and the porosity is given by Eq. (2), respectively.

Sp.Gr for sample =

$$[W_1 / (W_1 - W_2)] \times \text{Sp.Gr. of liquid} \quad (1)$$

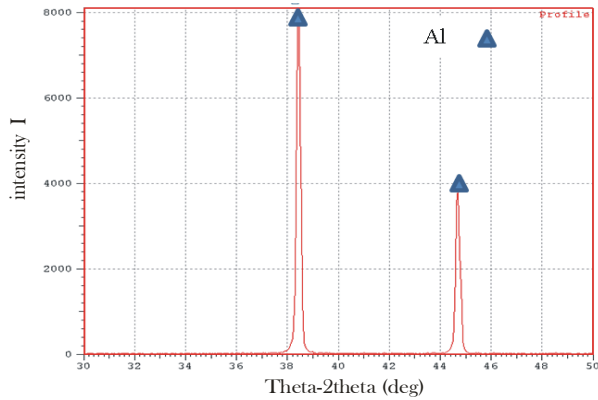


Fig. 1 XRD analysis results of pure Al.

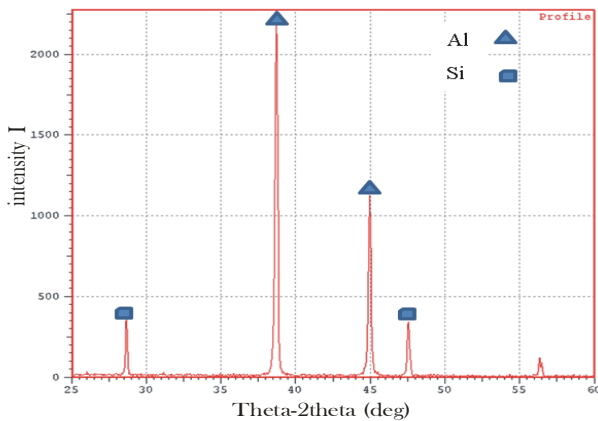


Fig. 2 XRD analysis results of sintered (Al-12%Si).

where, Sp.Gr for sample is the specific gravity of material (g/cm^3), W_1 is the weight of material in air (g) and W_2 is the weight of material suspended in liquid (g).

$$P = (W_3 - W_1) / \{(W_3 - W_2) \times \text{Sp.Gr. of liq.}\} \quad (2)$$

where, P is the porosity of material, W_1 is the weight of material in air, W_2 is the weight of material suspended in liquid, W_3 is the weight of wet material i.e. weight of soaked material in air.

Weight method was used to determine the wear rate of specimens. The specimens were weighted before and after the wear test by sensitive balance type DENVER instrument (Max-210gm) with an accuracy of 0.0001 gm. The weight loss (ΔW) was divided by the sliding distance and the wear rate was obtained by using the Eq. (3) [10].

$$\text{Wear rate (W.R)} = \Delta W / \pi D.N.t \text{ (gm/cm)} \quad (3)$$

where, D is sliding circle diameter (cm), t is sliding time (min), N is steel disc speed (rpm).

Wear surfaces topography and microstructure were investigated by using optical and SEM microscope type VEGA II TESCAN. AFM again used for measuring the surface roughness of polished surfaces for three samples of Al-12wt.%Si matrix hybrid nanocomposites reinforced with 2, 4 and 6wt.% (Al₂O₃ + TiO₂). Dry sliding wear tests for all samples were examined using a pin-on-disk setup according to the ASTM G99-05. The samples were used as pins of 10mm diameter. (63HRC) was used as the counter face in the pin-on-disk. The wear tests were conducted at room temperature with a two plans; first was under the applied load, respectively, of 5, 7.5, 10 and 12.5 N and fixed period of time; second plan was fixed one load and different periods of time (5, 10, 15, 20 and 30 min). Wear characterization of MMNCs was studied as a function of applied load and periods of times. SEM type used to examine the resulted worn surfaces. Polarized optical observations were also used for imaging to explain the worn surfaces.

3. Results and Discussion

The results were shown that the effect of milling process on the refinement of Al and Si grains is more significant at the presence of TiO₂ nanoparticles. These results agree with researchers Hanmin Bian et al [10]. Different factors such as uniform distribution, fine grain size of both matrix and reinforcement and strong interfacial bonding can greatly improve the hardness of the mechanically milled composites. Hardness in Table 1 confirmation to this, one can mention to the results obtained by Durai et al [11] and Srinivasa Rao et al [12].

50 nm of nano powder Al₂O₃ is used as shown in Fig. 3, which indicates the topography, distribution of particles and average particles size is 78.46 nm.

30 nm TiO₂ is used as shown in Fig. 4 while the AFM images for topography AFM images for mixed powders of base alloy (Al-12wt.%Si) and 4wt.% (Al₂O₃ + TiO₂) are shown in Fig. 5, the average particle size for mixed powders is (100.69 nm).

Table 1 Hardness, density and porosity% results for nanocomposites samples.

Sample	HV (kg/mm ²)	Density g/cm ³	Porosity %
Base alloy (Al-12 wt.%Si)	87	2.6079	0.12
Base alloy + 2 wt.%(Al_2O_3 + TiO_2)	114	2.6096	0.0978
Base alloy + 4 wt.%(Al_2O_3 + TiO_2)	123	2.6523	0.0930
Base alloy + 6 wt.%(Al_2O_3 + TiO_2)	135	2.8325	0.0833

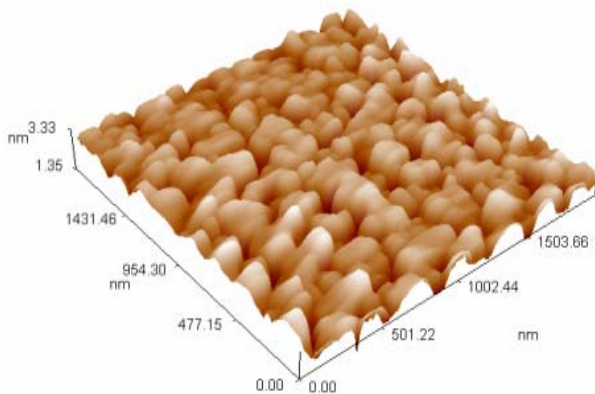
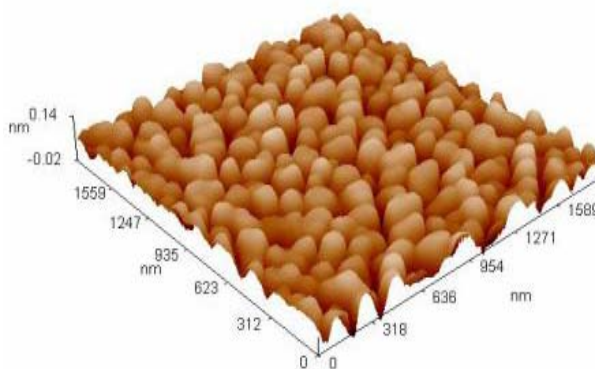
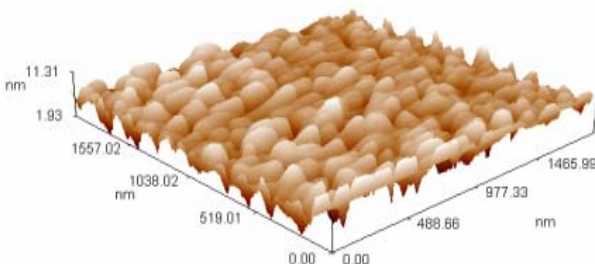
**Fig. 3** AFM results for nanopowder of Al_2O_3 .**Fig. 4** AFM results for nanopowder TiO_2 .**Fig. 5** AFM results for mixed powders of hybrid nanocomposite of base alloy (Al-12%Si) reinforced with 4 wt.%(Al_2O_3 + TiO_2).

Fig. 6 shows the SEM micrographs for mixed powders of (Al-12%Si) base alloy reinforced with 4wt.%(Al_2O_3 + TiO_2) hybrid nanoparticles at various

magnifications.

The density increases with an increase in the nanopowder addition and the created porosities will result in reduction of density. The highest density value belongs to nanocomposite sample containing 6wt.%(Al_2O_3 + TiO_2) hybrid nanoparticles. The amount of porosity level in all samples remained below 1% indicating the near net shape forming capability of the processing methodology in the compaction and sintering, this result is confirmed by researchers Francis et al [13]. Fig. 7 shows the SEM micrographs of nanocomposite sample of (Al-12wt.%Si) reinforced with 4 wt.%(Al_2O_3 + TiO_2) hybrid nanoparticles after sintering.

As expected, the wear rate of the coarse grained Al-12wt.%Si sample is higher than that of the nanostructured sample, which is due to the enhanced hardness of the latter. As shown in Fig. 8, the wear rate of the (Al_2O_3 + TiO_2) reinforced samples are lower comparing to that of the base sample. It also decreases with the increase in the (Al_2O_3 + TiO_2) content. This can be attributed to the high hardness and also the strong interfacial bond between the (Al_2O_3 + TiO_2) and Al-matrix, which in turn improves the load transfer from the matrix to the hard particles.

For all of the samples, the wear rate increases with an increase in the applied load, this is more significant in the coarse grained (Al-Si) sample in comparison to the other samples.

The wear rate decreases with increasing the hardness that increases with hybrid nano particles additions to nanocomposites. The hardness increases with 64% of hybrid MMNC with 6wt.%(Al_2O_3 + TiO_2) hybrid of nanoparticles as compared with base

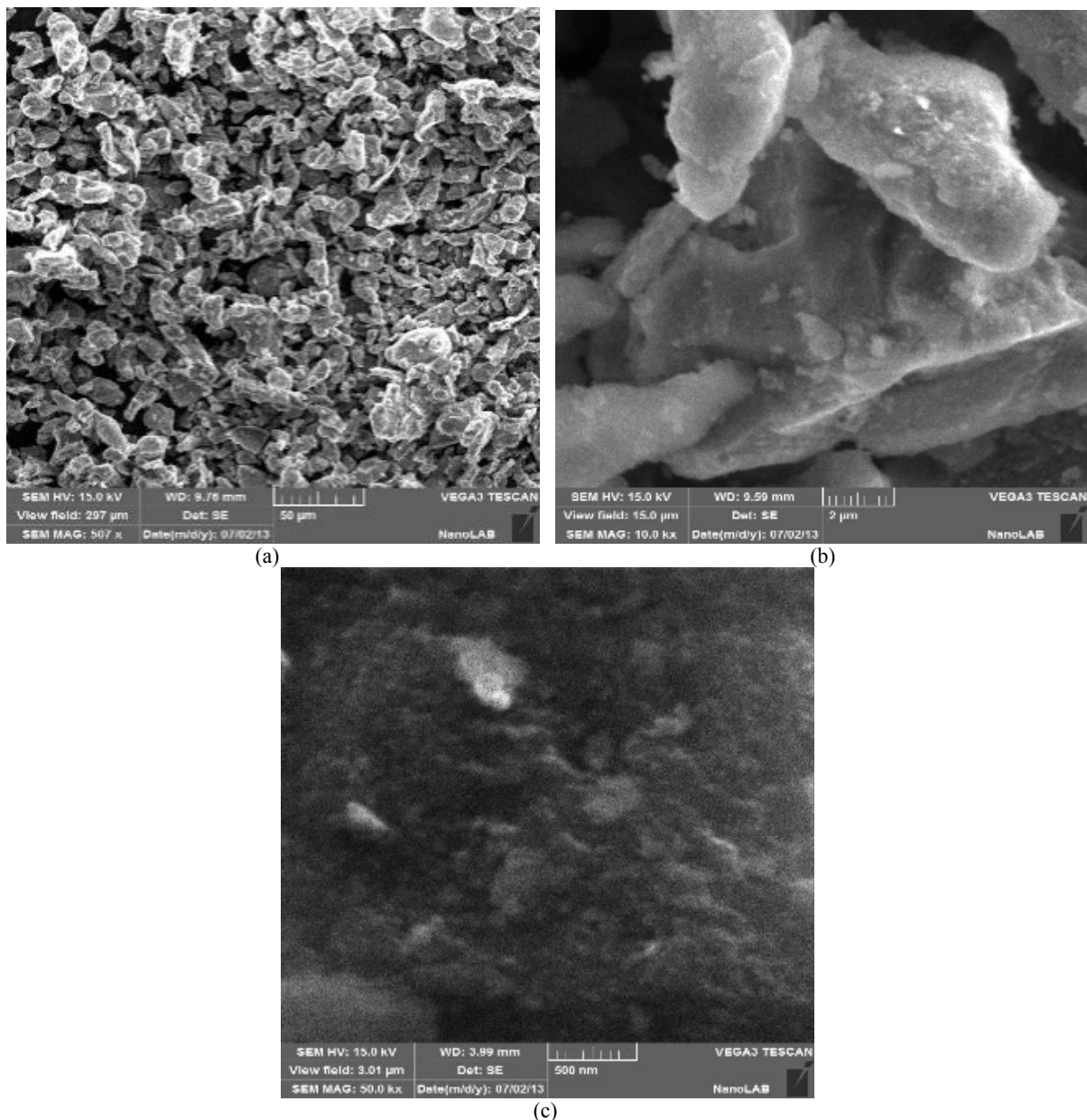


Fig. 6 SEM micrographs for mixed powders of (Al-12%Si) base alloy reinforced with 4 wt.% ($\text{Al}_2\text{O}_3 + \text{TiO}_2$) hybrid nanoparticles: (a) at 500x, (b) at 10,000x and (c) 50,000x.

alloy. This advanced strengthening system reduces the wear rate. Based on the SEM observations, as shown in Fig. 9, plastic deformation of the Al-Si matrix and progressive transfer of the material to the counterface during the sliding process and the wear mechanism is adhesion. The cracks along wear tracks of the base sample tested at applied load of 12.5 N.

The worn surface and wear debris of the nanocomposite samples are shown in Fig. 10. As

seen, the grooves of different sizes and shallow craters are evident in the worn surface. In addition, examination of the wear debris for this sample revealed a plate or sheet-like also as described by Alpas and Embury [14].

Fig. 11 shows the SEM micrographs of worn surface for hybrid nanocomposite reinforced with 4 wt% ($\text{Al}_2\text{O}_3 + \text{TiO}_2$) nanoparticles under a normal load (12.5 N) at various magnifications, it can be seen

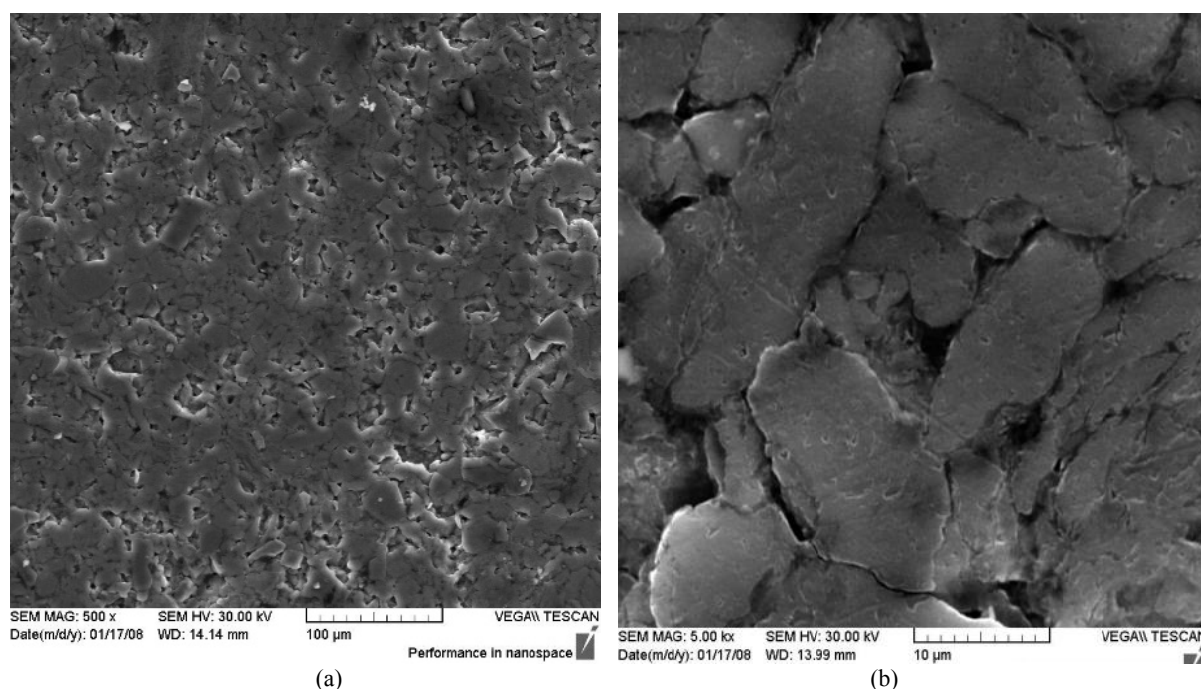


Fig. 7 SEM micrographs for sintered nanocomposite sample of (Al-12 wt.%Si) reinforced with 4 wt.% ($\text{Al}_2\text{O}_3 + \text{TiO}_2$) hybrid nanoparticles: (a) at 500x and (b) at 5,000x.

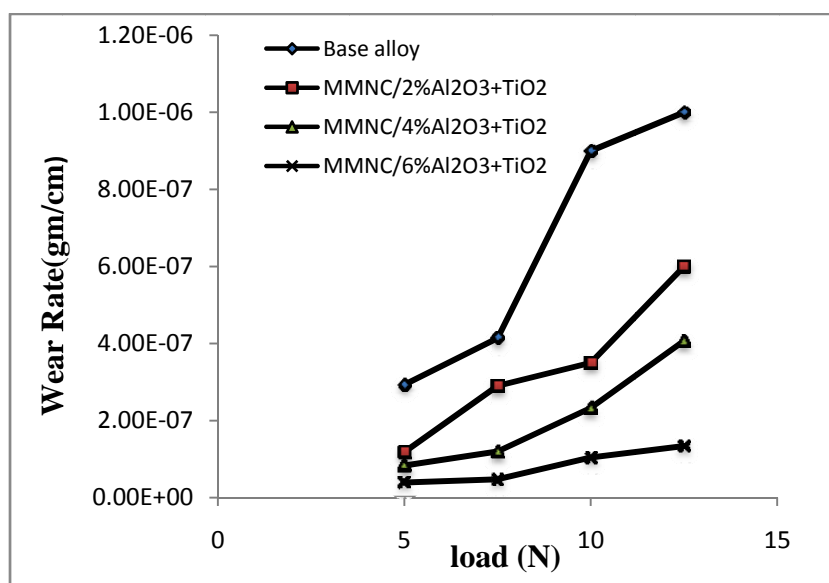


Fig. 8 Effect of applied loads on wear rate for base alloy (Al-12%Si) and hybrid nanocomposite samples at constant sliding time of 15 min.

that there are different sizes of wear debris or particles on the surface and also micro cracks and micro vacancies can be seen clearly from Figs 11a-11d.

On the other hand, these grooves can be considered as evidences of the abrasive mechanism, since hard asperities of the hard counter face or hard nanoparticles of ($\text{Al}_2\text{O}_3 + \text{TiO}_2$) exist between the pin

and disk surfaces. Therefore, it can propose that the dominant wear mechanism for this sample is a combination of delamination and abrasion. The same mechanism has previously been proposed by Ma et al [15]. for Al-SiC composite. Fig. 12 shows the effect of abrasive severe wear under higher normal load (12.5 N) and it was noticed the cracks and the particles

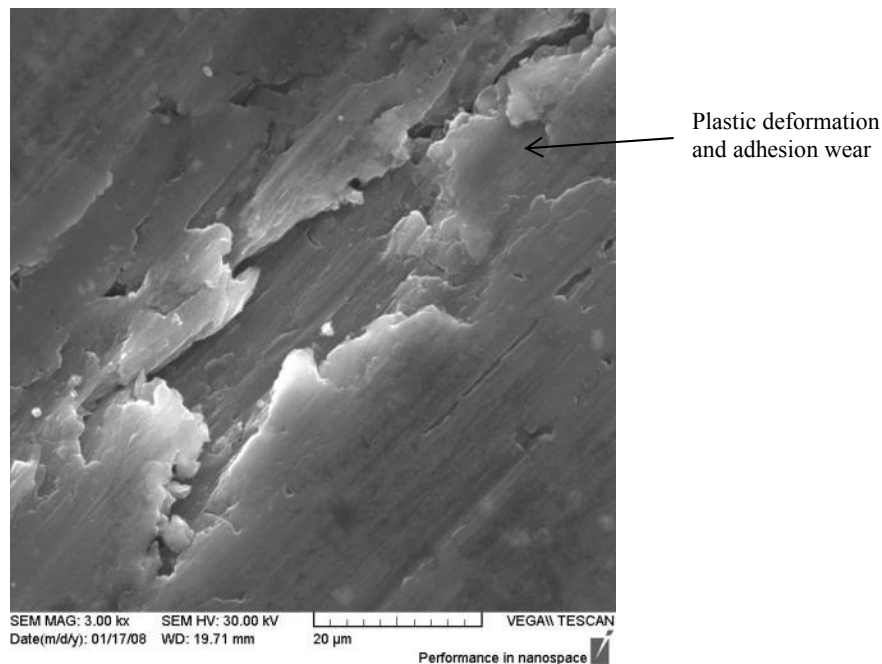


Fig. 9 SEM micrograph of worn surface for base alloy (Al-12wt.%Si) under a normal load 12.5 N at 3,000x.

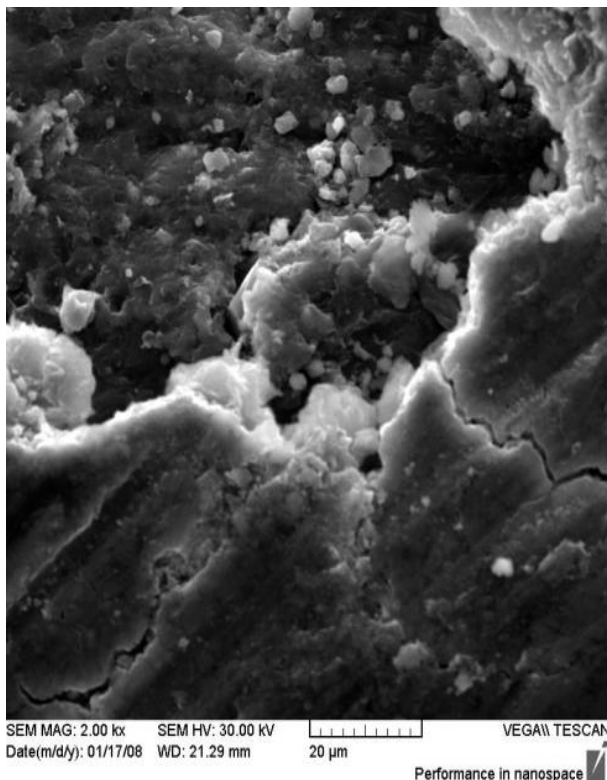


Fig. 10 SEM micrograph of worn surface for hybrid nanocomposite reinforced with 2wt.% ($\text{Al}_2\text{O}_3 + \text{TiO}_2$) nanoparticles under a normal load (12.5 N) at 2,000x.

no longer stay on the worn surface. The hard nanoparticles of (Alumina + Titania) exist between the pin (sample) and disk surfaces. Therefore, the wear mechanism for this sample is a combination of delamination and abrasion.

The effect of sliding time on wear rate for base alloy and hybrid nanocomposites with various percentages of ($\text{Al}_2\text{O}_3 + \text{TiO}_2$) nanoparticles is shown in Fig. 13 at constant load of 7.5 N, and sliding distance of 7 cm.

The best wear resistance (less wear rate) is with hybrid nanocomposites reinforced with of 6wt.% ($\text{Al}_2\text{O}_3 + \text{TiO}_2$) hybrid nanoparticles for different periods of time.

The low load will not effect on hybrid nanocomposite as high load with increased time, as shown from comparison between curves in Figs. 8 and 13 and SEM images as shown in Figs. 11 and 14 for wear rate with effect of maximum load and effect of maximum time respectively for same hybrid nanocomposite.

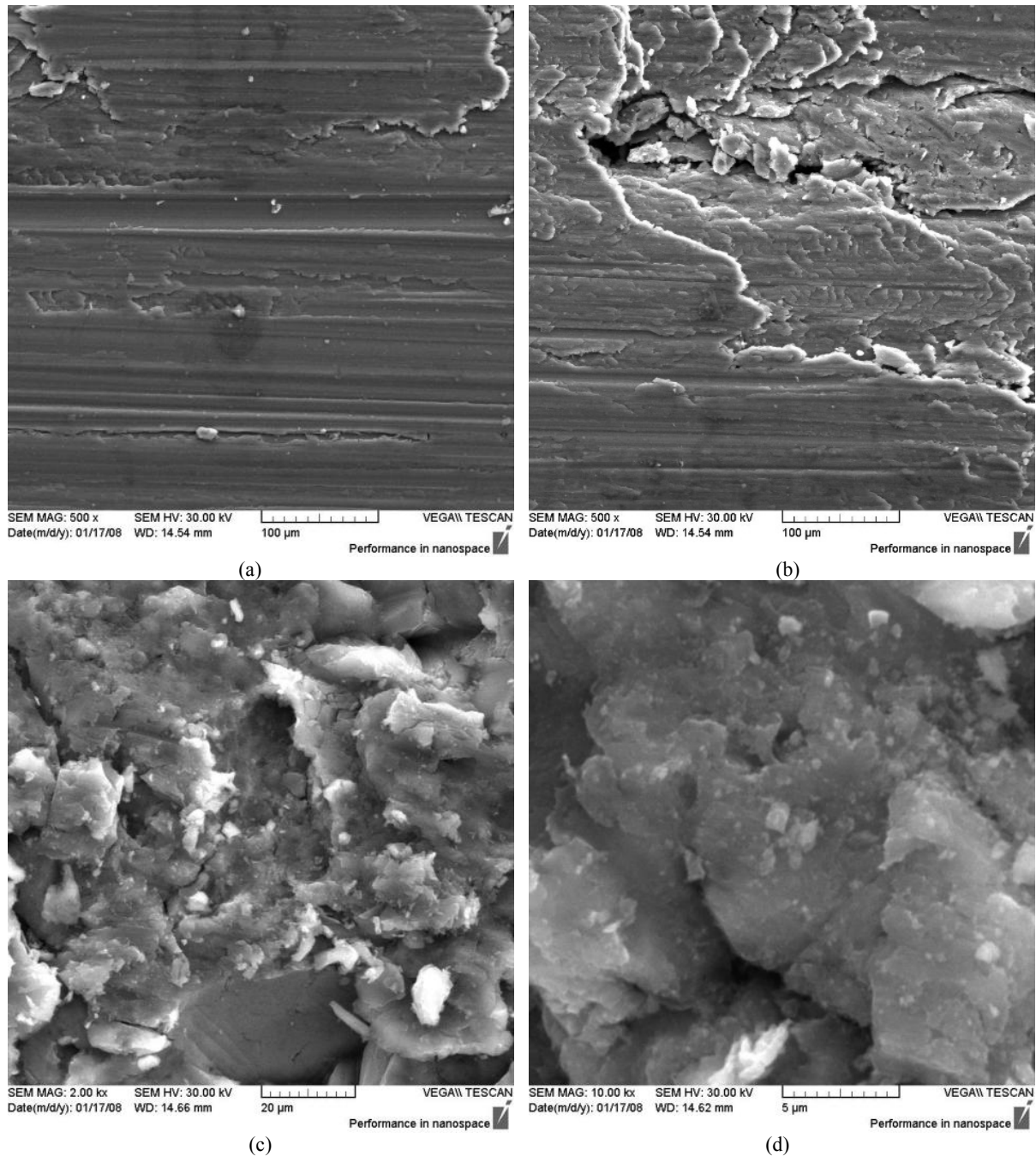


Fig. 11 SEM micrographs of worn surface for hybrid nanocomposite reinforced with 4 wt.% ($\text{Al}_2\text{O}_3 + \text{TiO}_2$) nanoparticles under a normal load (12.5 N): (a) and (b) at 500x, (c) at 2,000x and (d) at 10,000x.

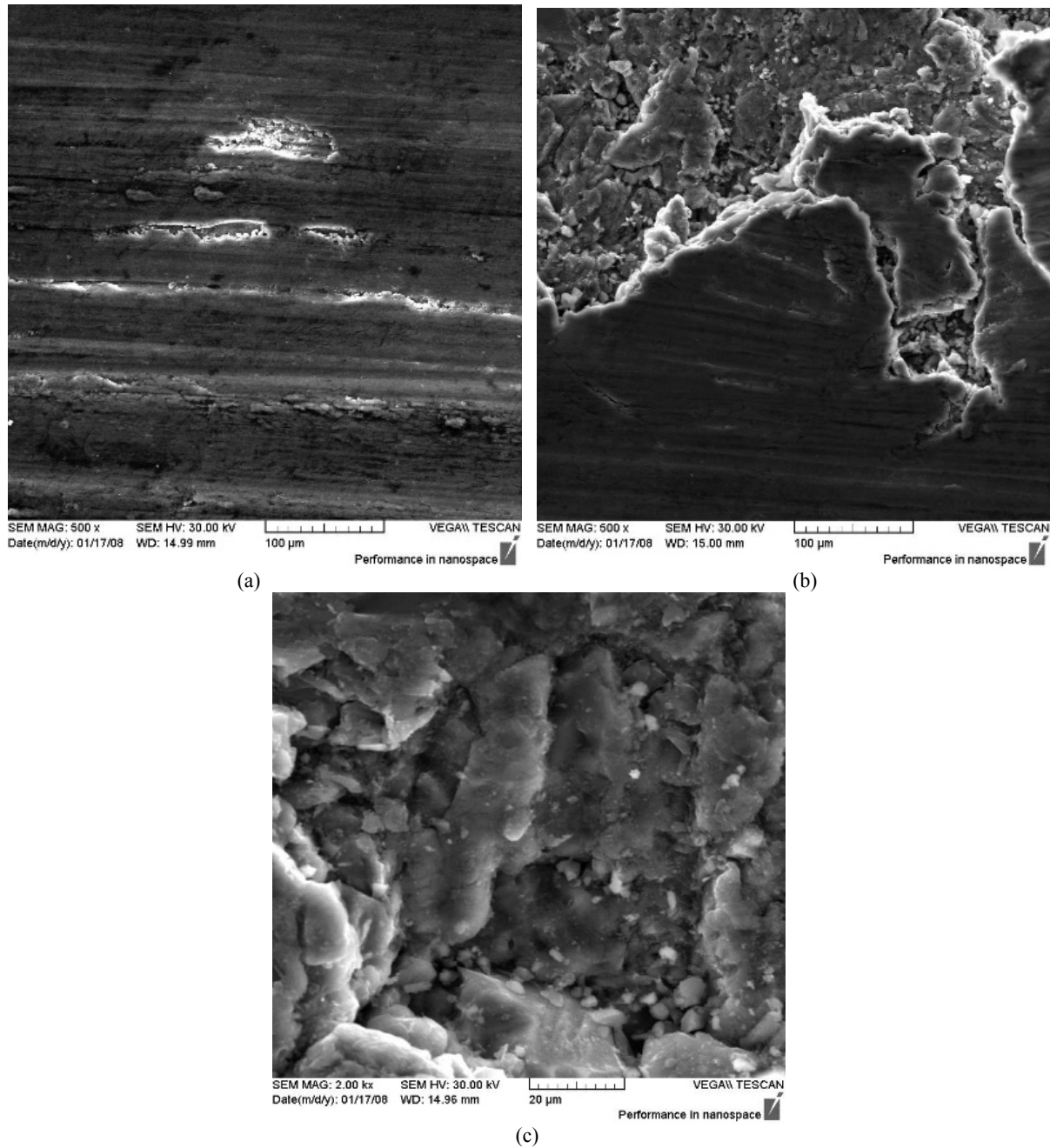


Fig. 12 SEM micrograph of worn surface of hybrid nano composite reinforced with 6 wt.% ($\text{Al}_2\text{O}_3 + \text{TiO}_2$) nanoparticles under a normal load (12.5 N): (a) and (b) at 500x and (c) at 2,000x.

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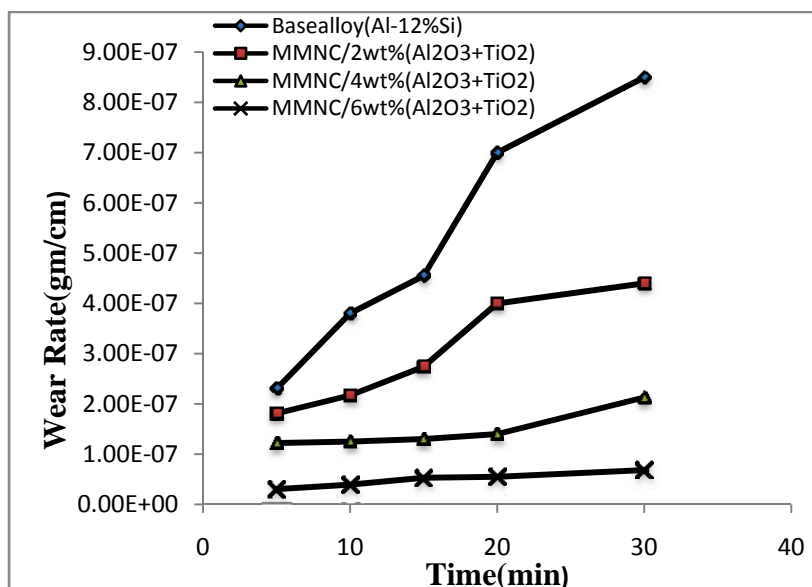


Fig. 13 Effect of sliding time on wear rate for base alloy (Al-12%Si) and hybrid nanocomposites under constant load (7.5 N).

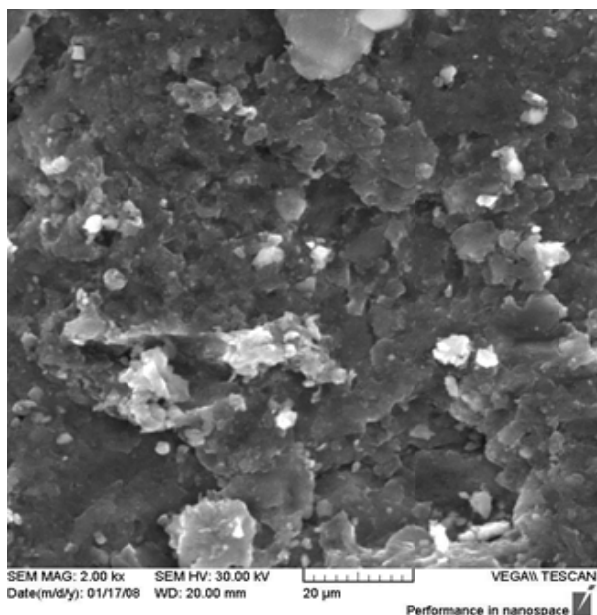


Fig. 14 SEM micrographs of worn surface of nanocomposite reinforced with 4 wt.% ($\text{Al}_2\text{O}_3 + \text{TiO}_2$) nanoparticles under a normal load of 7.5 N and 30 min at 2,000x.

4. Conclusions

Results of AFM images of mixed powders of the base alloy and hybrid nanopowders ($\text{Al}_2\text{O}_3 + \text{TiO}_2$) indicate good mixing between the different powders and homogenous distribution of nanopowders in the Al-12 wt.%Si matrix.

The wear rate or weight loss of the base alloy and nanocomposite samples increases with the increase in the applied load and sliding time. But the hybrid nanocomposites show lower wear rate or better wear resistance than the base alloy under the same loads and sliding time.

The wear rate of the nanocomposites is considerably improved by the addition of the reinforcement nanoparticles and decreases with increasing the weight percentages from 2 to 6 wt.% ($\text{Al}_2\text{O}_3 + \text{TiO}_2$) nanoparticles in matrix of Al-alloy.

The wear behavior of base alloy and nanocomposites is similar and can be classified into three regions, first; mild wear which occurs between loads (5-7.5 N), second; transition wear which is between loads (7.5-10 N), the third; severe wear which occurs at higher load of 12.5 N.

The hybrid nanocomposites reinforced with 6 wt.% ($\text{Al}_2\text{O}_3 + \text{TiO}_2$) hybrid nanoparticles give the highest hardness and best wear resistance compared with other hybrid nanocomposites.

From SEM micrographs of the worn surfaces of base alloy and nanocomposites samples the dominant wear mechanism is plastic deformation and adhesion of base alloy, there is a deformation layer at upper part of the

wear surface, while for nanocomposites samples it is delamination and abrasion.

References

- [1] Hemant, K. 2008. "Synthesis of Al-Si-Ni Nanostructured Materials by Mechanical Alloying." M.Sc. Thesis in Metallurgical and Materials Engineering Department, National Institute of Technology Rourkela.
- [2] Tok, A. I. Y., Boey, F. Y. C. and Zhao, X. L. 2006. "Novel Synthesis of Al₂O₃ Nanoparticles by Flame Spray Pyrolysis." *Journal of Materials Processing Technology* 178: 270-3.
- [3] Mohammed, J. F. and Muna, K. A. 2014. "Wear Characterization of Hybrid Aluminum Matrix Nanocomposites." LAP Lambert Academic Publishing.
- [4] Suryanarayana, C. 2001. "Mechanical Alloying and Milling." *Progress in Materials Science* 46: 1-184.
- [5] Hadi, E. F. 2007. "Investigation into the Industrial Use of Nanocomposite Coatings." Petroleum University of Technology, Faculty of Petroleum Engineering, Abadan-Iran, 1-10.
- [6] Jung, H. A., Kim, Y. J. and Chung, H. 2008. "Al-AlN Tri-Modal Composites Prepared By Mechanical Alloying." *Advanced Mater. Sci.* 18: 329-34.
- [7] Hao, Y. 2010. "Processing Routes for Aluminum Based Nanocomposites." Msc. Thesis, Worcester Polytechnic Institute.
- [8] Alizadeh, A. and Taheri-Nassaj, E. 2011. "Wear Behavior of Nanostructured Al and Al-B₄C Nanocomposites Produced by Mechanical Milling and Hot Extrusion." *Tribol Lett* 44: 59-66.
- [9] Ali, M. and Mohsen, O. S. 2012. "Study on Microstructure and Abrasive Wear Behavior of Sintered Al Matrix Composites." *Ceramics International* 38 (5): 4263-9.
- [10] Bian, H. M., Yang, Y., Wang, Y., Tian, W., Jiang H. F. and Hu, Z. J. et al. 2013. "Effect of Microstructure of Composite Powders on Microstructure and Properties of Microwave Sintered Alumina Matrix Ceramics." *Journal of Materials Science & Technology* 29 (5): 429-33.
- [11] Durai, T. G., Das, K. and Das, S. 2008. "Al (Zn)-4Cu/Al₂O₃ *in situ* Metal Matrix Composite Synthesized by Displacement Reactions." *J. Alloys Compd.* 457: 435-9.
- [12] Srinivasa, R. C. and Upadhyaya, G. S. 1995. "6061 Aluminum Alloy-Based Powder Metallurgy Composites Containin Carbide Particles/Fibres." *Mater. Des.* 63: 59-66.
- [13] Francis, E. D., Eswara-Prasad, N., Ratnam, Ch., Sundara-Kumar, P. and Venkata-Kumar, V. 2011. "Synthesis of Nano Alumina Reinforced Magnesium Alloy Composites." *International Journal of Advanced Science and Technology* 27: 35-44.
- [14] Alpas, A. T. and Embury, J. D. 1990. "Sliding and Abrasive Wear Behavior of Aluminum 2024-SiC Particle Reinforced Composite." *Scr. Metall.* 24: 931-5.
- [15] Ma, T., Yamaura, H., Koss, D. A. and Voigt, R. C. 2003. "Dry Sliding Wear Behavior of Cast SiC-Reinforced Al MMCs." *Mater. Sci. Eng. A* 360: 116-25.