

Study of Wear Behavior of Aluminum Alloy Matrix Nanocomposites Fabricated by Powder Technology

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Received on: 26/11/2013 & Accepted on: 6/3/2014

ABSTRACT

In the present work, the dry sliding wear behavior of Al-12wt%Si matrix nanocomposites reinforced with single addition of 4wt.% Al₂O₃ or 4wt.%TiO₂ nanoparticles, and with hybrid addition of 4wt% (Al₂O₃ +TiO₂) nano particles is investigated. All nanocomposites samples were fabricated by powder technology by mechanical milling of the base alloy (Al-12wt%Si) powder and nanopowders of Al₂O₃ and TiO₂, followed by cold pressing at 100bar and sintering at 520 °C for 90min. Vickers hardness test was done by using Vickers hardness tester. Archimedes technique was used to measure the density of sintered samples and porosity calculated as physical tests of sintered samples. Also AFM, SEM were used to investigate the morphology of mixed powders and nanocomposites samples.

Pin – on Disc 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 nanocomposite with 4wt% Al₂O₃ nanoparticles shows the highest hardness than other nanocomposites. It was observed that the wear rate or weight loss of the base alloy and nanocomposite samples increases with the increase in applied load and sliding time. But the nanocomposites samples showed lower wear rate than the base alloy within the same conditions.

Keywords: Nano composites; Wear; Mechanical milling; Powder technology

دراسة سلوك البلى لمواد متراكبة نانوية ذات أساس المنيوم مصنعة بتكنولوجيا المساحيق

الخلاصة

يُدرس البحث الحالي خواص البلى الانزلاقي الجاف لمواد متراكبة نانوية ذات اساس من سبيكة الألمنيوم-12% سليكون مقواة بدقائق نانوية من 4% الومينا او التيتانيا او (كإضافة مشتركة). وقد تم تصنيع السبيكة الأساس والمواد المتراكبة النانوية بطريقة تكنولوجيا المساحيق حيث أجريت عملية الخلط الميكانيكي للمساحيق المختلفة من مسحوق سبيكة الألمنيوم-12% سليكون ومسحوق الالومينا و التيتانيا ثم

أجريت عملية الكبس على البارد بضغط 100 بار والتلييد عند درجة حرارة 520°م لمدة 90 دقيقة. وقد تم قياس الصلادة الفيكيرية وقياس الكثافة والمسامية للنماذج المليدة . كذلك تم استعمال مجهر القوة الذرية والمجهر الإلكتروني الماسح لدراسة مورفولوجي وفحص المساحيق المخلوطة وبعض المواد المترابطة النانوية الهجينة المصنعة. وقد اوضحت الصور المأخوذة بمجهر القوة الذرية لخليط المساحيق من السبيكة الاساس والدقائق النانوية المشتركة من الالومينا و التيتانيا حصول خلط جيد بين المساحيق المختلفة وتوزيع متجانس للدقائق النانوية في ارضية السبيكة . وتم اجراء اختبار البلى من نوع المسمار على القرص تحت ظروف الانزلاق الجاف مع تغيير الاحمال المسلطة و ازمان الانزلاق لكل النماذج المحضرة . وتم استعمال المجهر الضوئي والمجهر الإلكتروني الماسح لدراسة طوبوغرافية سطوح البلى وملاحظة خطوط البلى وحطام البلى. وان المواد المترابطة النانوية المقواة بإضافة 4 % الومينا فقط أعطت أعلى صلادة مقارنة مع المواد المترابطة النانوية الاخرى. وقد لوحظ ان معدل البلى او الوزن المفقود يزداد مع زيادة الحمل المسلط وزمن الانزلاق. ولكن المواد المترابطة النانوية اظهرت معدل بلى اقل من السبيكة الاساس عند نفس ظروف التجربة.

INTRODUCTION

Nano composites are composite materials with at least one phase of a size of less than 100nm. The other components can be larger sized so that it is possible to work with matrices based on submicron sized powders of 100-500 nm and provide the desired functionality by adding a nano scale powder of <100 nm[1]. Nanostructure or Nanocomposite materials include (metallic, ceramic, and polymeric types). There are some of applications; as super hard, tribological coatings for aerospace application, components for electronic and optoelectronic devices, wear-resistant coatings for biomedical applications, in surface engineering technologies and for improving properties of conventional powders [2]. Conventional powder metallurgy (PM) and solution chemical processes like sol-gel methods have been used to prepare composite powders. It has been reported that with a small fraction of nano-sized reinforcements, metal matrix nano composites (MMNCs) could obtain comparable or even far superior mechanical properties than metal matrix composites (MMCs).

Recent investigations have revealed that further improvements in the wear resistance of aluminum matrix composites (AMCs) can be achieved by synthesizing Nano composites where hard nano particles are embedded in aluminum matrix.

Aluminum oxide (Al_2O_3) the most useful oxide ceramics, as it has been used in many fields of engineering such as coatings, heat-resistant materials, abrasive grains, cutting materials and advanced ceramics. This is because alumina is hard, highly resistant towards bases and acids, allows very high temperature applications and has excellent wear resistance [3].

Fathabadi [2007] [4] investigated nanocomposite coatings fabricated by thermal sprayed $Al_2O_3-xTiO_2$ ($x = 0, 3, 13$ and 20 wt%). Al_2O_3 has been chosen as a base for a preparation of the nanocomposite coatings, due to its superior structural properties such as high hardness, good wear resistance. TiO_2 itself is known as a good wear resistance material and can act as an effective toughening phase, therefore it was added to the matrix 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 [4].

Ahamed and Senthilkumar[2010] [5] employed the high-energy wet ball milling to synthesize nano-crystalline Al6063 alloy powders reinforced with 1.3 vol.% Al₂O₃ , 1.3 vol.% Y₂O₃ and 0.65 vol.% Al₂O₃ , 0.65 vol.% Y₂O₃ at nano-size level. The crystallite size of the matrix of powder particle was affected by the addition of different types of nanoceramic particles separately and in combination keeping total volume percentages of such addition as constant.

Alizadeh et al. [2011] [6] investigated the wear properties of a nanostructured matrix of Al prepared via mechanical milling and hot extrusion before and after incorporation of B₄C nanoparticles. Mechanical milling was used to prepare the nanocomposite samples by addition of 2 and 4 wt % of B₄C 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 nanostructured matrix of Al in contrast to a commercial coarse grained Al matrix. They concluded that wear behavior of coarse grained Al sample is adhesion while for the base nanostructured Al matrix and Al-B₄C nanocomposites is delamination and combination of abrasion and delamination respectively.

Mazahery et al. [2012] [7] used zircon particles reinforced aluminum matrix composite which was fabricated by a powder metallurgy process. In the first stage, different volume percentages of zircon (2.5, 5, 7.5, 10, 12.5 & 15) with average particle size of 20 μm were added to aluminum powders (33 μm) by means of a planetary ball mill without protective atmosphere and with 1% zinc stearate applied as lubricant. The milling time and speed were 20 min and 600 rpm, respectively. In the next stage, the samples were compacted at cold isostatic press (CIP) 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 against case hardened steel disk of hardness 63 HRC. It was noted that the weight loss of the composites is less than that of unreinforced alloy, increases with increase in sliding distance, and has a declining trend with increasing the particles volume fraction.

The present study was undertaken with the effect of Al₂O₃ and / or TiO₂ nanoparticles addition on wear behavior of Al-12wt%Si matrix composites and microstructural characteristics of worn surfaces by using optical and SEM were also investigated.

Experimental Work

Materials used:

The materials used for preparation the metal matrix nanocomposites (MMNCs) are 25μm of Al powder, 25μm of Si powder have 99.98% and 98.5% of purity respectively, two types of nano particles of 50nm of gamma-Al₂O₃ and 30nm of rutile-TiO₂ have 99.98% and 99.8% of purity respectively. Dry mixing method was used to mix the powder of alloy (Al-12%Si) with nano particles of (Al₂O₃ and TiO₂). 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 both (nano Al₂O₃ and nano TiO₂) or with single (nano Al₂O₃ or nano

TiO₂). 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 10Mpa (1500psi) pressure followed by sintering process at 520°C for 90min by using electric tube furnace with argon flow rate 2L/min. 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, and AFM 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 equation (1) and the porosity is given by equation (2) respectively [8].

$$\text{Sp.Gr for sample} = [W1 / (W1 - W2)] \times \text{Sp. Gr. of liquid} \quad \dots\dots\dots (1)$$

where:-

Sp.Gr for sample is the specific gravity of material (g/cm³)

W1 :- The weight of material in air (g)

W2 :- The weight of material suspended in liquid(g)

$$P = (W3 - W1) / \{(W3 - W2) \times \text{Sp.Gr. of liq.}\} \quad \dots\dots\dots (2)$$

where:-

P :- The porosity of material

W1 :- The weight of material in air

W2 :- The weight of material suspended in liquid

W3 :- 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 equation (3) [9].

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

where:-

D :- sliding circle diameter (cm)

t :- sliding time (min)

N :- steel disc speed (rpm)

Wear Test variables were used as following:

Variable loads were: 5, 7.5, 10 and 12.5 N with fixed time of 15 minutes and the distance of (7 cm) with constant speed. The hardness of steel disc was 63HRC.

Wear surfaces topography and microstructure were investigated by using optical and SEM microscope type VEGA II TESCAN

Results and Discussion

AFM results for powders of Al₂O₃ (50nm), TiO₂(30nm) and base alloy (Al-12%Si) +4wt % (Al₂O₃+TiO₂) as shown in figures 1,2 and 3.

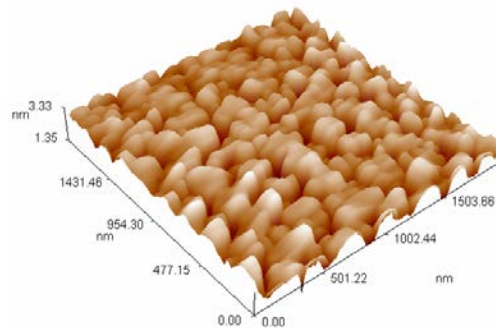


Figure. (1) AFM results for nano powder Al_2O_3

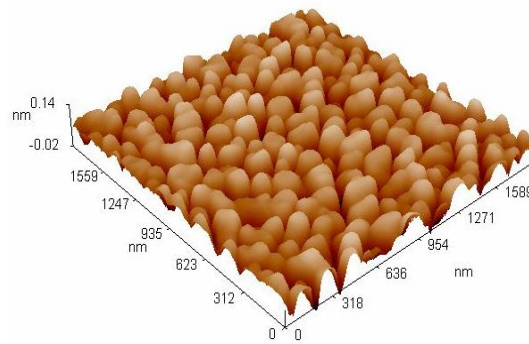


Figure.(2) AFM results for nano powder TiO_2

50nm of nano powder Al_2O_3 is used as shown in Figure (1) which indicates the topography, distribution of particles and average particles size is 78.46 nm.

30nm TiO_2 is used as shown in Figure (2) while the AFM images for topography, distribution of particles and the average particles size was (30.95 nm). AFM images for mixed powders of base alloy (Al-12%Si) and 4wt % ($Al_2O_3+TiO_2$) are shown in Figure (3) which indicates the topography in 3-D image. The average particles size for mixed powders was 100.69 nm.

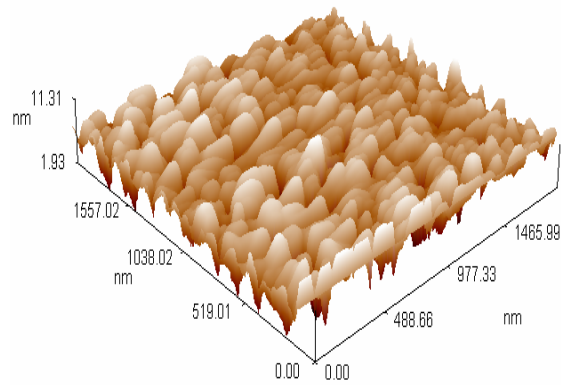


Figure. (3) AFM results for mixed powders of hybrid nanocomposite of base alloy (Al-12%Si) +4wt %($\text{Al}_2\text{O}_3+\text{TiO}_2$).

Some agglomerations were occurred leading to formation of large particles. This is due to insufficient mixing time for powders preparation by using ethanol only without sonication method. If used last method, the results were obtained the good dispersion and less agglomerations. The results from AFM topography for mixing powders (micro and nano) as in Figure (3) shows a good dispersion of nano particles resulted from mechanical alloying and the average particles size was 100.69 nm for mixed powders. SEM Also for mixed powders of hybrid nanocomposite of base alloy (Al-12%Si) +4wt %($\text{Al}_2\text{O}_3+\text{TiO}_2$) shown in Figure (4) .

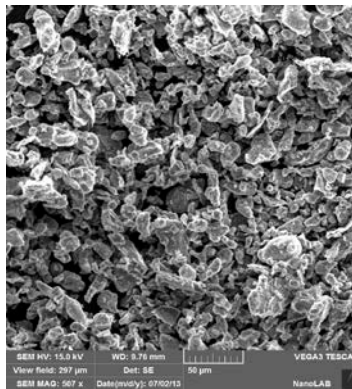


Figure. (4) SEM for mixed powders of hybrid nanocomposite of base alloy (Al-12%Si) +4wt %($\text{Al}_2\text{O}_3+\text{TiO}_2$).

Mechanical milling, which has been used in this study, produces uniform dispersion of the reinforcement particles in the matrix through a repeated process of cold welding,

fracturing, and rewelding, giving rise to the reinforcement particles being well embedded into the matrix particles.

It is noticed that clear different in microstructure in shape and size of Si-phase and eutectic (Al+Si) between nanocomposites (MMNC) reinforced with 4wt% Al_2O_3 and MMNC reinforced with 4wt% ($\text{Al}_2\text{O}_3+\text{TiO}_2$). It is seen that the microstructure of hybrid nanocomposite is finer than that of MMNC reinforced with 4wt% (TiO_2) only. This is due to (TiO_2) nanoparticles addition that acts as refiner and modifier for Al-matrix and Si phase respectively. While it was found that the finer microstructure of Si phase when 4wt% (TiO_2) nanoparticles added only and it is also finer microstructure than that of nanocomposite reinforced with 4wt% Al_2O_3 nanoparticles addition only as shown in Figures (5),(6) and (7).

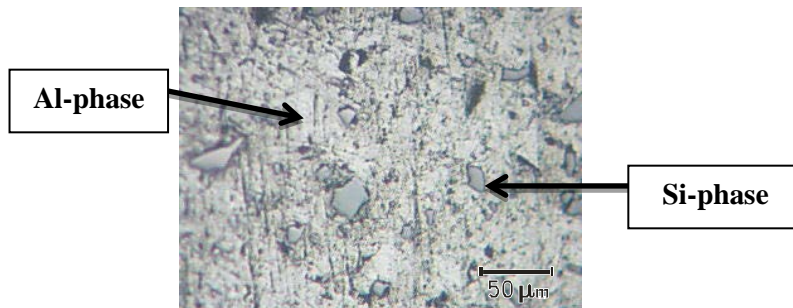


Figure.(5) Microstructure of MMNC with 4wt% ($\text{Al}_2\text{O}_3+\text{TiO}_2$)

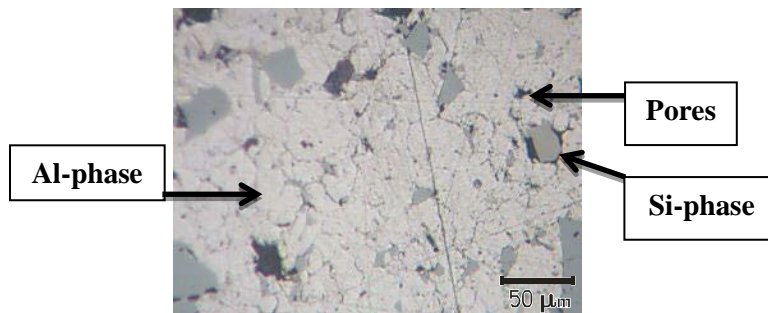


Figure.(6)Microstructure of MMNC with 4wt% (Al_2O_3) nanoparticles

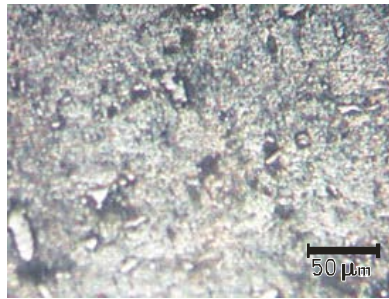


Figure. (7) Microstructures of MMNC with 4wt% (TiO₂) nanoparticles

Figure 8 shows Al-Si matrix hybrid nanocomposite reinforced with 4wt% (Al₂O₃+TiO₂) by SEM.

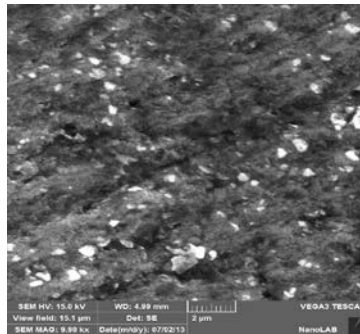


Figure.(8) SEM micrographs for sintered MMNC sample of Al-12%Si +4wt% (Al₂O₃+TiO₂) ;at 10,000 x.

Hardness Results

Vickers hardness measurements for all (MMNCs) samples are listed in Table (1).

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

Samples	HV kg/mm ²	Density g/cm ³	Porosity %
Base alloy (Al-12%Si)	87	2.6079	0.12
Base +4wt% Al ₂ O ₃ +TiO ₂	123	2.6523	0.093
4wt% Al ₂ O ₃	148	2.68	0.09
4wt%TiO ₂	132	2.62	0.094

As shown in Table (1) the (MMNCs) samples have significantly higher hardness than that of base alloy (Al-12%Si). Also, the hardness of hybrid (MMNCs) increases with increasing the percentage of both nanopowders ($\text{Al}_2\text{O}_3+\text{TiO}_2$) in Al-matrix.

It is seen that the (MMNC) sample with 4wt% Al_2O_3 only have the highest hardness as compare with other (MMNCs), this is because of nano Al_2O_3 have higher hardness than that of TiO_2 . Many researchers confirmed these results in their works, as in [10]. The highest hardness results from one addition of nano Al_2O_3 and moderate hardness for addition of nano TiO_2 . The increased in density is connect with decreasing of porosity for nanocomposites, but also with the nano powders additions.

Effect of Nanoparticles Addition on Wear Rate

Figures (9, 10, 11) and (12) were show the influence of the applied load on the wear rate of the base alloy (Al-12wt%Si) and nanocomposite samples. For all of the samples, the wear rate increases with an increasing in the applied load which is more significant in case of base alloy in comparison to the other samples of nanocomposites with single addition (Al_2O_3 or TiO_2) and hybrid addition of ($\text{Al}_2\text{O}_3+\text{TiO}_2$) nanoparticles.

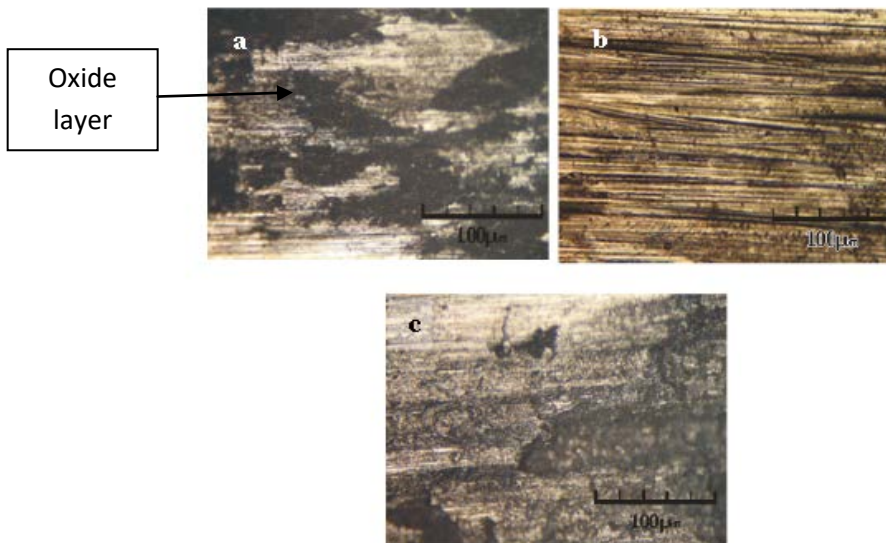


Figure.(9)Optical micrographs of worn surfaces for base alloy (Al-12%Si) under different loads at fixed sliding time and speed: a) at 7.5 N , b) at 10 N and c) at 12.5 N.

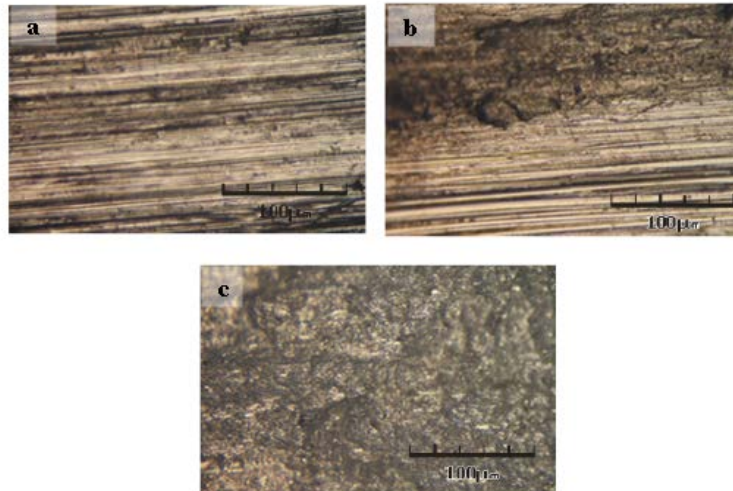


Figure.(10) Optical micrographs of worn surfaces for hybrid nanocomposite of 4wt% ($\text{Al}_2\text{O}_3+\text{TiO}_2$) under different loads with at fixed sliding time and speed; a) 7.5N, b) 10N and c) 12.5N.

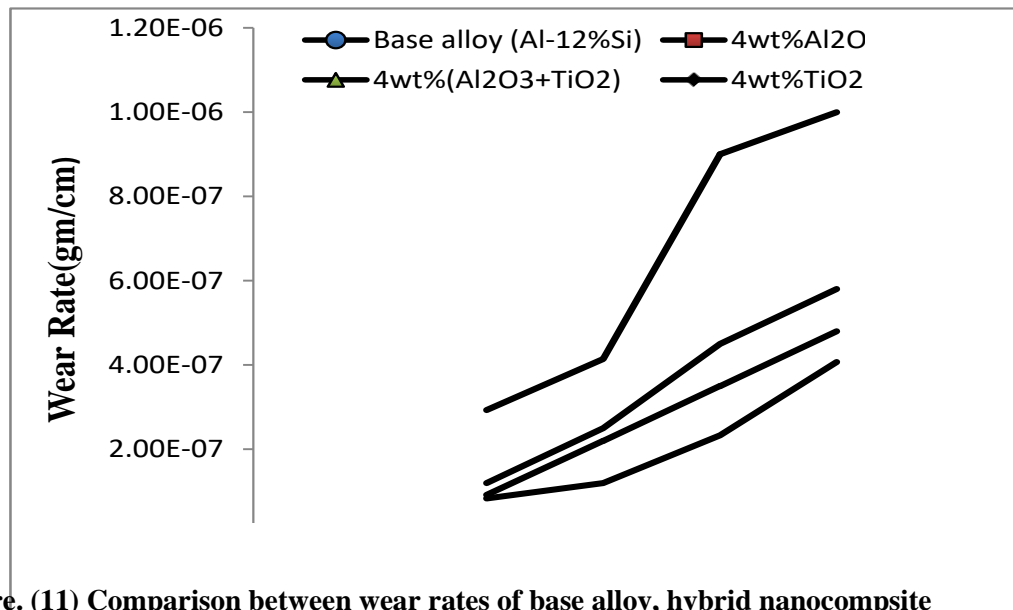


Figure. (11) Comparison between wear rates of base alloy, hybrid nanocompsite with 4wt % ($\text{Al}_2\text{O}_3+\text{TiO}_2$), nanocomposites with single addition 4wt% Al_2O_3 and 4wt% TiO_2 as a function of applied load.

It was noticed that the effect of 4wt % ($\text{Al}_2\text{O}_3+\text{TiO}_2$) hybrid nano particles addition on the wear resistance is the best sample in comparison to the other samples. This is

attributed to the combined effect of nano TiO_2 particles which is modified and refined the microstructure of Al-matrix and Si phase in addition to the clear role of nano Al_2O_3 particles in improving the hardness of the nanocomposite.

The SEM analysis of wear surface was carried out to study the wear mechanisms in the base alloy and nanocomposites. Figure (12) shows SEM micrographs of worn surface of the base alloy which revealed the plastic deformation of the Al-matrix and transfer of the material to counterface during the sliding process and the wear mechanism was adhesion.

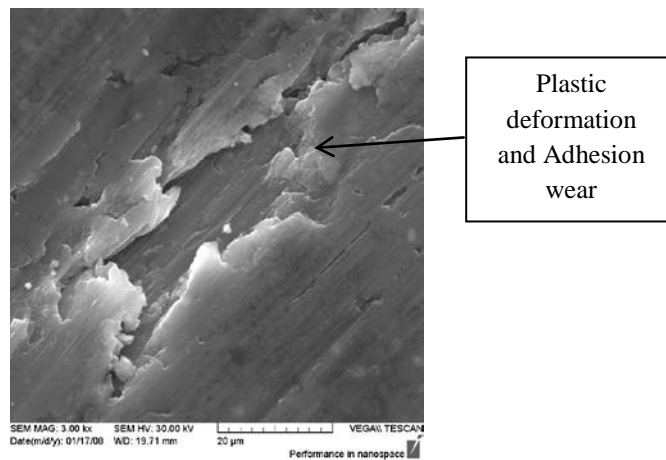


Figure.(12)SEM micrograph of worn surface for base alloy (Al-12wt%Si) after wear test at normal load 12.5N; at 3000x.

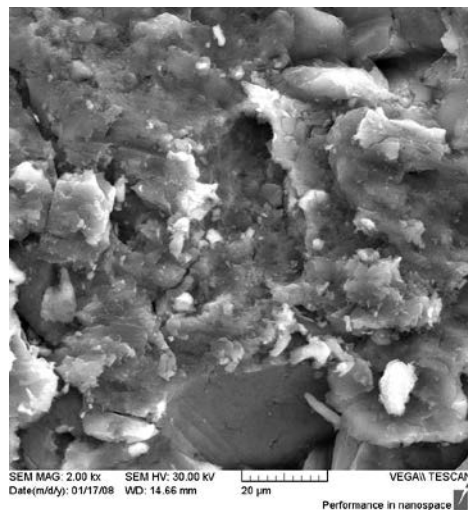


Figure.(13) SEM micrographs of worn surface for hybrid nanocomposite with 4wt% ($\text{Al}_2\text{O}_3 + \text{TiO}_2$) nanoparticles after wear test at normal load (12.5N), at 2000x.

When the load is getting higher plastic deformation and that a few particles separated from the aluminum surface, leaving the surface exposed to friction with the surface of the hard disk. But when a little load as shown in Figure (14) we note that the surface suffers from wear less and fine wear lines and observed the effects of oxide layer on the sample surface where this is called oxidative wear.

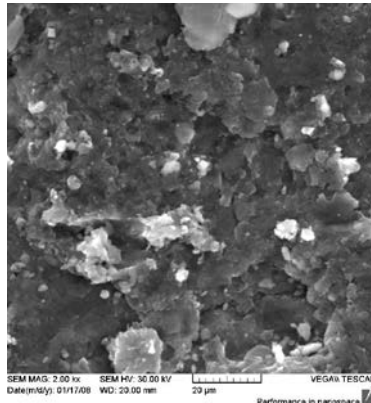


Figure.(14) SEM micrographs of worn surface for nanocomposite with 4wt% ($\text{Al}_2\text{O}_3 + \text{TiO}_2$) nanoparticles after wear at 7.5N normal load and 30min. at 2000x.

The low load will not effect on hybrid nanocomposite as high load with increased time that from comparison between SEM images as shown in Figures (13&14) for wear rate with effect of maximum load and effect of maximum time respectively for same hybrid nanocomposite.

CONCLUSIONS

- 1-Mechanical milling, which has been used in this study, produces uniform dispersion of the reinforcement nanoparticles in the Al-12wt%Si matrix.
- 2-Results AFM images of mixed powders for the base alloy and hybrid nanopowders ($\text{Al}_2\text{O}_3 + \text{TiO}_2$) indicates good mixing between the different powders and homogenous distribution of nanopowders in the Al-Si matrix.
- 3- The Al-Si powders entered the nanometric regime ($< 100\text{nm}$) at around 8h of milling (first 4h mixing of Al-Si and second 4h mixing of Al-Si with nano particles) .
- 4-The wear rate or weight loss of the base alloy and nanocomposite samples increases with the increase of the applied load. But the nanocomposites showed lower wear rate than the base alloy within the same loads.
- 5-The nanocomposites reinforced with single addition of 4wt% Al_2O_3 nanoparticles shown the highest hardness comparing with other nanocomposites and have higher wear resistance when compared with base alloy and other nanocomposites.

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