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GRAIN REFINEMENT OF ALUMINUM-BASED NANOCOMPOSITES

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ABSTRACT

Grain refinement of an aluminum alloy was carried out by adding 0.6 wt.% Al-Ti5-B1 master alloy to the aluminum alloy melt at 720 °C for a holding time of 10 minutes. 2 wt.% Al_2O_3 nanoparticles were added to the base melt and mechanically stirred to produce the nanocomposite. The two types of additions were used in case of the combined treatment. A considerable grain refinement of the aluminum alloy was obtained by the addition of Al-Ti5-B1 to its melt. A nanocomposite of fine matrix grains was also produced. A modified nanocomposite of more refined matrix grains and homogeneous microstructure was produced by application of the combined treatment. Both hardness and wear resistance of the aluminum alloy were increased by grain refinement. Greater increases in these properties were obtained in case of nanocomposite. Remarkable improvements in these properties were achieved in the case of grain refined nanocomposite.

KEYWORDS

Aluminum grain refinement; Aluminum matrix nanocomposites; Al_2O_3 nanoparticles; Modification of nanocomposites; Wear resistance.

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INTRODUCTION

In the normal casting conditions, the metals and alloys are usually solidified with a coarse and columnar grain structure [1-3]. The cast structure affects the mechanical properties [4]. Grain refinement plays an important role in improving properties of cast and wrought aluminium alloys [1,5]. The chemical grain refinement can be carried out using Al-Ti5-B1 master alloy. However, the grain refiners based on Al–Ti–B suffer from poisoning in the presence of Zr, and some other elements [1,2,6]. To solve this problem, Al–Ti–C-based grain refiners can be used [1,2,6]. In addition, the performance of metals and alloys can be enhanced by adding suitable reinforcing materials [7,8]. The produced materials are metal matrix composites (MMCs). More outstanding properties over MMCs can be achieved by using reinforcement of nanosize. MMCs with reinforcing particles in the range of 10 nm – 1 µm are termed "Metal Matrix Nano-composites" (MMNCs) [7]. It was reported that, MMNCs overcome the shortcoming of MMCs (such as poor ductility, low fracture toughness and low machinability) [7]. The present study aims to investigate the combined effect of grain refinement (which can be produced by adding Al-Ti5-B1 master alloy) and reinforcement (using Al_2O_3 nanoparticles) on the microstructure and some properties and behavior (such as microhardness and wear resistance) of the produced nanocomposites.

EXPERIMENTAL WORK

The experimental work was planned to carray out three treatments: chemical grain refinement, reinforcement and a combined treatment (addition of both grain refiner and reinforcing particles). In all experiments, aluminum alloy of chemical composition listed in Table 1 was used as a matrix (or base) material. The chemical grain refinement was carried out by adding 0.6 wt.% Al-Ti5-B1 master alloy to the aluminum alloy melt at 720 °C, mechanically stirred and holded at this temperature for 10 minutes after which the treated melt was poured inside a steel mold. The grain refiner was used in the form of chips produced by turning the Al-Ti5-B1 master alloy rod.

The reinforcement treatment was carried out by adding 2 wt.% Al_2O_3 nanoparticles to the aluminum alloy melt and application of mechanical stirring at the same temperature of the grain refinement treatment. The mechanical stirring aids in reducing the agglomeration of nanoparticles. Finally, in the third treatment, the melt of aluminum alloy was subjected to the combined treatment. This treatment was carried out by adding the two additives: (a) 0.6 wt.% Al-Ti5-B1 master alloy to the matrix (aluminum alloy) melt at 720 °C for a holding time of 10 minutes and (b) 2 wt.% Al_2O_3 nanoparticles, after which the mixture of melt and the additives were mechanically stirred. The microstructures of different specimens were investigated using optical and scanning electron microscopes. The different phases and constituents were identified with the aid of EDX and XRD analyses. To evaluate the effect of the different treatments on some properties and behavior of the prepared materials, microhardness and wear tests were conducted. Microhardness of the different materials were measured using microhardness tester with load of 200 g. Wear tests were conducted at load of 544.5 g for sliding distance of 602.88 m using a pin on disc wear tester.

Table 1. Chemical analysis of matrix (base) alloy (wt. %):

Si	Fe	Cu	Mn	Mg	Zn	Ni	Cr	Ti	Pb	Sn	V	Al
1.04	0.245	0.041	0.54	0.62	0.021	0.021	0.012	0.035	0.024	0.028	0.013	Bal.

RESULTS AND DISCUSSIONS

Effect of Treatments on Microstructure

The microstructures of untreated Al alloy, grain refined alloy (Al alloy + 0.6 wt.% Al-Ti5-B1), nanocomposite (Al alloy/2 wt.% Al_2O_3 nanoparticles), and grain refined nanocomposite are shown in Fig. 1 (a, b, c and d respectively). In case of the untreated alloy; Fig. 1(a); a microstructure of large grains was obtained. Coarse columnar α-Al dendritic grains are clearly revealed in this figure. With the addition of 0.6 wt.% of Al-Ti5-B1 master alloy, the coarse dendritic structure was transformed into one of fine and equiaxed grains; Fig. 1(b). In this case and as described in many papers, the TiB_2 and $TiAl_3$ (which represent main phases of Al-Ti5-B1) act as heterogeneous nucleation sites for the refined phase. These phases were identified by XRD analysis as shown in Fig. 2. The microstructure of the aluminum alloy-based nanocomposite is shown in Fig. 1(c). As shown in this figure, some grains are refined. Grain refinement of aluminum matrix due to the addition of Al_2O_3 nanoparticles was also obtained by other investigators [9-11]. In these cases, the Al_2O_3 nanoparticles can act as heterogeneous nucleation sites during solidification. To observe and characterize the features of Al_2O_3 nanoparticles (size, distribution and agglomeration) and the bonding between them and the matrix, investigations using scanning electron microscope were done. Figures 3 and 4 represent typical SEM images of this composite and EDX analyses for Al_2O_3 nanoparticles. As shown in the images, some Al_2O_3 particles are uniformly distributed through the matrix alloy. The EDX analyses show that, some particles are found at the grain boundaries; Fig. 3 and other ones are found at the grain centers; Fig. 4. The particles that are near the grain boundaries are pushed by the solidification front, they seem to be also nucleating sites. The nanoparticles show a rounded agglomerated structure of about 200 to 300 nm. The presence of Al_2O_3 can be indicated by O and part of Al peaks shown in the EDX analyses; Figs. 3 and 4.The detection zone shown in Fig. 4 is bigger than the analaysed Al_2O_3 nanoparticles and as a result the peaks include compositional information of Al matrix near particles.

The combined effect of the two additives (Al-Ti5-B1 and Al_2O_3) on the microstructure for the produced material is demonstrated in Fig. 1(d). More refined and homogenized microstructure was produced. This can be attributed to the presence of the different heterogeneous nucleating sites (TiB_2, $TiAl_3$ and Al_2O_3) and hindering of grain growth which can be resulted from the presence of Al_2O_3 particles at the grain boundaries. Some Al_2O_3 nanoparticles are agglomerated in some zones as shown in Figs 5(a) and 6(a). These particles are confirmed by the EDX analyses; Figs 5 (b) and 6 (b). During solidification processing, nano particulates tend to agglomerate. The agglomeration and non-homogeneous distribution of the reinforcement with particle debonding were also observed by other investigators [7,12]. The agglomeration and non-homogeneous distribution of the reinforcing nanoparticles reduces the direct contact and bonding between them and the matrix and consequently the improvement in properties (specially strength) is reduced.

An α-Al grain solidified on $TiAl_3$ blocky in the case of grain refined aluminum alloy-based nanocomposite is shown in Fig. 7(a). The presence of $TiAl_3$ phase is confirmed by EDX analysis; Fig. 7(b). As described in [5], borides nucleate α-Al. However the effectiveness in nucleation of borides is lower than that of $TiAl_3$ [5].

In all EDX analyses; Figs. 3-7; a part of the Al peak is resulted from excitation of a portion of the matrix and the other part is resulted from other constituents (such as $TiAl_3$ or Al_2O_3). In Fig. 4, portions of both Al and Si of matrix are indicated. The peaks of other matrix elements are too low to detected and the analyses were concentrated on the nucleation and reinforcing agents (TiB_2, $TiAl_3$ and Al_2O_3). The different phases were investigated with the aid of XRD and shown in Fig. 2.

Effect of Treatments on Hardness

Microhardness of the different materials are listed in Fig. 8. An increase in hardness was obtained by refining the grains. In this case, 34 % improvement in hardness was obtained. It is well known that, the reduce in grain size increases the strength and hardness of the material.

By adding the reinforcing particles, a greater increase in hardness (41.8 % improvement) was obtained. This can be attributed to the combined effect of the refined matrix grains and the hardening due to the reinforcement. As described above, Al_2O_3 nanoparticles act as heterogeneous nucleation sites for aluminum which lead to grain refinement. The resulted grain refinement aids in the strenthening of nanocomposite. The grain-refined strengthening effect of Al_2O_3 particulates is also reported by others [10,12]. In addition, the uniform distribution of some Al_2O_3 particles aids in the hardness improvement. No decohesion between the Al_2O_3 particles and the matrix was observed. This indicate that bonding between the reinforcing particles and the matrix (see figures (4-6)) is strong which is an important factor in improving the hardness and strength. Also, The nano size of reinforcing particles plays another important role in the hardness improvement. This is in agreement with the results of other investigators [13,14]. In these cases, and as stated in [15], the high strain energy for the periphery of particles increases the hardness. Moreover, the difference between the coefficient of thermal expansion (CTE) values of matrix and ceramic particles generates thermally induced residual stresses and increases the dislocations density [16]. As a result the hardness and strength are increased. Also, the high hardness of the ceramic particles (~2200 HV for alumina) aids in hardness improvemen [17,18]. The improvement in hardness of aluminum-based nanocomposite due to reinforcing with alumina was also obtained by other investigators [12,19].

A remarkable improvement (57.3 %) was achieved in case of grain refined nanocomposite. This can be attributed to the combined effect of grain refinement (resulted from the addition of Al-Ti5-B1 and Al_2O_3 nanoparticles), microstructure homogenization and reinforcement with Al_2O_3 nanoparticles. This result shows that, the hardening effect of the reinforcing agent is improved by grain refinement of the matrix phase.

Based on the analyses for the effect of Al_2O_3 nanoparticles on the microstructure features and hardness of the produced Al-based nanocomposite, it can be concluded that, these particles can be considered as a grain refiner and reinforcing agent.

Effect of Treatments on Wear Rate

Specimens of the different materials were prepared and tested at 544.5 g and sliding distance of 602.88 m. The wear of the different specimens was evaluated by calculating the weight losses per unit area. Figure 9 show the weight losses for the different specimens. 16.7 % reduction in weight losses per unit area was obtained in case of grain refined material. So, the wear rate decreases with the decrease in the grain size. The decrease in grain size increases the hardness and strength. Consequently, the wear rate is decreased. These results are in agreement with those obtained by other investigators [20].

The hardening of material due to the addition of reinforcing particles increases the wear resistance. The reduction in weight losses per unit area in this case is greater than that in the case of grain refinement. In case of the nanocomposite and as described in the treatments and microstructural analyses, very hard nanoparticles (Al_2O_3 of ~ 200-300 nm) are uniformly dispersed in the matrix with good interfacial bonding between them and the matrix. In addition, the matrix grains are refined due to the action of these nanoparticles as heterogeneous nucleating sites during solidification of α-Al. These microstructural features and the resulted properties (such as high hardness) aid in the wear resistance improvement. These results are in agreement with those obtained by several investigators [7,12,21-28].

A remarkable reduction in the weight losses per unit area was achieved in the case of grain refined nanocomposite. This result can be attributed to the combined effect of grain refinement (resulted due to the addition of conventional grain refiner; Al-Ti5-B1 master alloy; and Al_2O_3 nanoparticles) and the reinforcement. In this case, 66.7 % reduction in weight loss per unit area was obtained.

CONCLUSIONS

The results of the present study lead to the following conclusions:

1. A considerable grain refinement for the investigated aluminum alloy can be obtained by adding 0.6 wt.% Al-Ti5-B1 to the melt at 720 °C for a holding time of 10 minutes.
2. An aluminum matrix nanocomposite of refined matrix grains can be produced by adding 2 wt. % Al_2O_3 nanoparticles to the matrix melt and application of mechanical stirring. Accordingly, the nanoparticles of Al_2O_3 can be considered as grain refiner for aluminum alloys.
3. A modified aluminum matrix nanocomposite of more refined matrix grains and homogeneous microstructure can be produced by application of combined treatment (the two treatments: addition of 0.6 wt.% Al-Ti5-B1 master alloy to the

matrix (aluminum alloy) melt at 720 °C for a holding time of 10 minutes and 2 wt.% Al_2O_3 nanoparticles, after which the mixture of melt and the additives are mechanically stirred).

4. Both hardness and wear resistance of the investigated aluminum alloy can be increased by grain refinement.
5. Greater increases in both hardness and wear resistance can be obtained by producing the nanocomposite.
6. Remarkable improvements in both hardness and wear resistance can be achieved by producing the grain refined nanocomposite.

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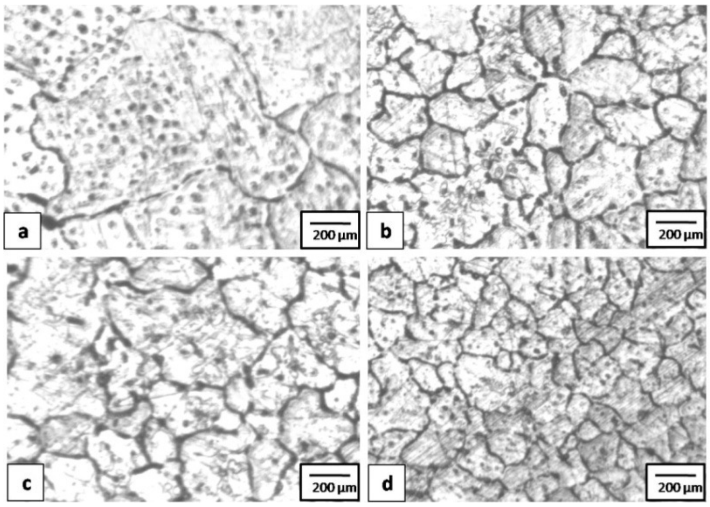


Fig. 1. Optical micrographs for microstructures of untreated and treated material: (a) untreated Al alloy, (b) grain refined alloy (Al alloy + 0.6 wt.% Al-Ti5-B1), (c) nanocomposite (Al alloy/2 wt.% Al_2O_3 nanoparticles), and (d) Grain refined nanocomposite (Al alloy/2 wt.% Al_2O_3 nanoparticles + 0.6 wt.% Al-Ti5-B1).

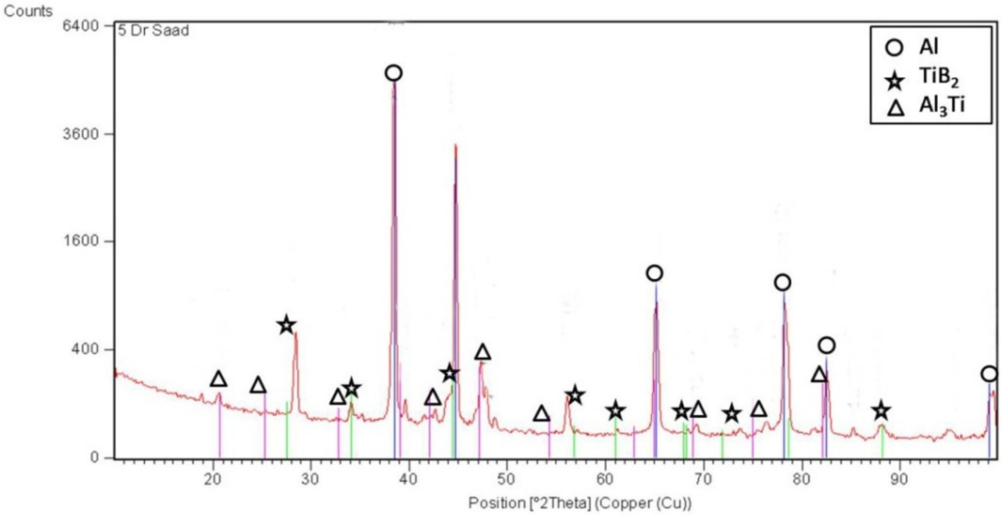
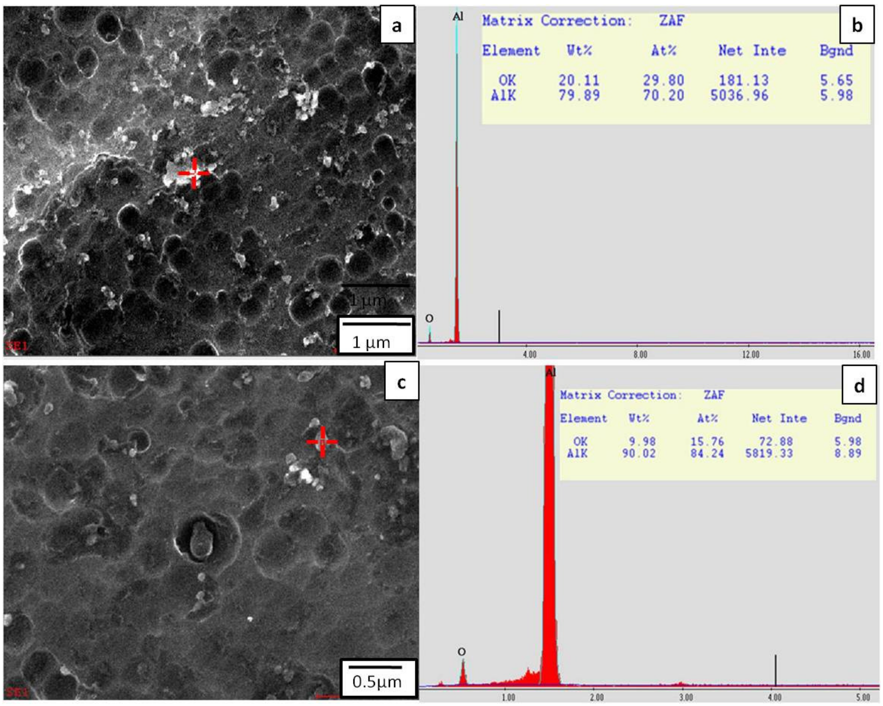


Fig. 2. XRD analysis for constituents and phases of grain refined aluminum alloy.



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Matrix Correction:    ZAF

Element    Wt%       At%     Net Inte     Bgnd

  OK      20.11     29.80     181.13      5.65
 AlK      79.89     70.20    5036.96      5.98
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Matrix Correction:    ZAF

Element    Wt%       At%     Net Inte     Bgnd

  OK       9.98     15.76      72.88      5.98
 AlK      90.02     84.24    5819.33      8.89
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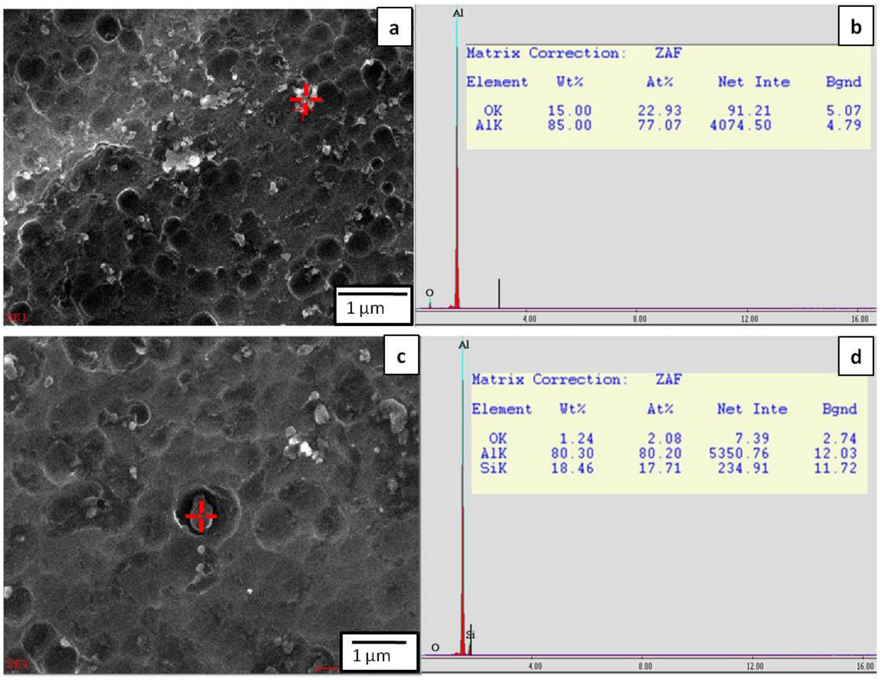
Fig. 3. EDX analysis for elements of Al_2O_3 nanoparticles (marked by red cross) at grain boundaries in case of the nanocomposite.



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Matrix Correction:    ZAF

Element    Wt%       At%     Net Inte     Bgnd

  OK      15.00     22.93      91.21      5.07
 AlK      85.00     77.07    4074.50      4.79
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Matrix Correction:    ZAF

Element    Wt%       At%     Net Inte     Bgnd

  OK       1.24      2.08       7.39      2.74
 AlK      80.30     80.20    5350.76     12.03
 SiK      18.46     17.71     234.91     11.72
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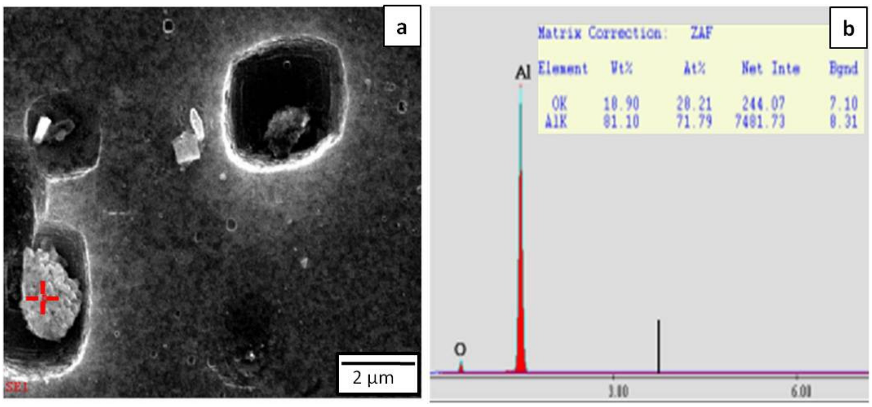
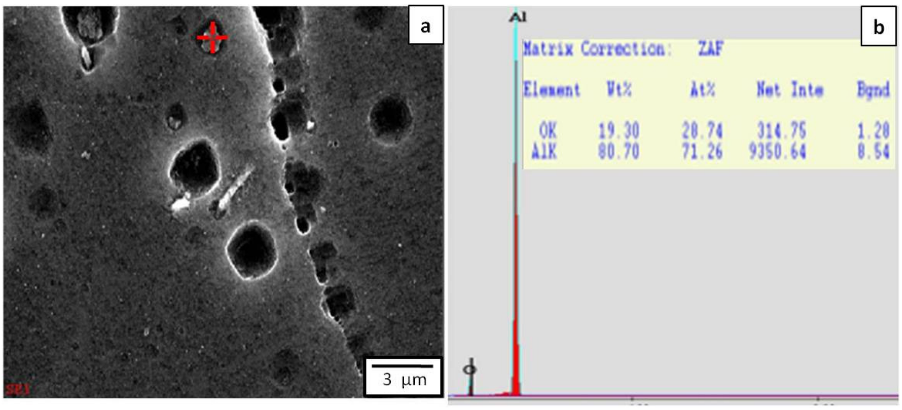
Fig. 4. EDX analysis for elements of Al_2O_3 nanoparticles (marked by red cross) on which a grain is solidified in case of the nanocomposite.



Matrix Correction: ZAF

Element	Wt%	At%	Net Inte	Bgnd
OK	18.90	28.21	244.07	7.10
AlK	81.10	71.79	7481.73	8.31

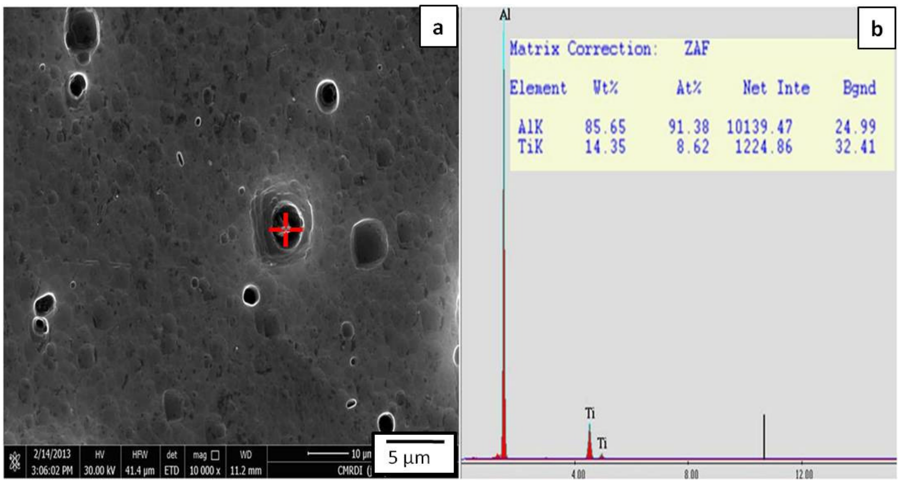
Fig. 5. (a) SEM image for microstructure of grain refined nanocomposite. The figure shows the accumulated Al_2O_3 nanoparticles. (b) EDX analysis for elements of Al_2O_3 nanoparticles.



Matrix Correction: ZAF

Element	Wt%	At%	Net Inte	Bgnd
OK	19.30	28.74	314.75	1.28
AlK	80.70	71.26	9350.64	8.54

Fig. 6. (a) SEM image for microstructure of grain refined nanocomposite. The figure shows accumulated Al_2O_3 nanoparticles inside the grain. (b) EDX analysis for elements of some accumulated Al_2O_3 nanoparticles (marked by red cross) in the microstructure.



Matrix Correction: ZAF

Element	Wt%	At%	Net Inte	Bgnd
AlK	85.65	91.38	10139.47	24.99
TiK	14.35	8.62	1224.86	32.41

Fig. 7. (a) SEM image for microstructure of grain refined nanocomposite. (b) EDX analysis for elements of $TiAl_3$ on which a matrix grain is solidified.

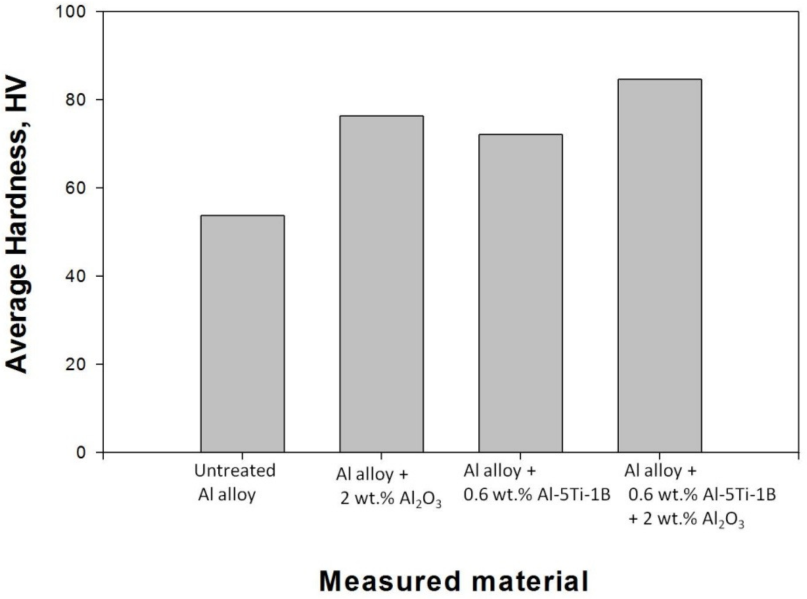


Fig. 8. Microhardness of the different materials, $HV_{0.2}$

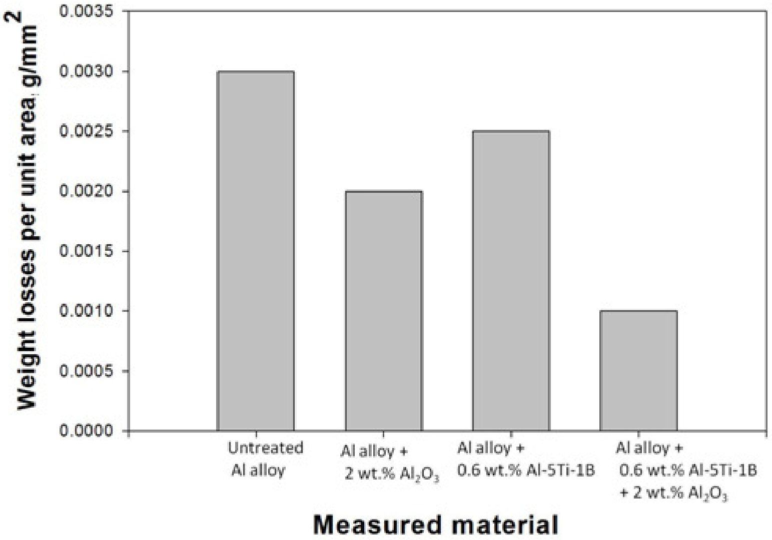


Fig. 9. Weight losses for the different specimens tested at 544.5 g and sliding distance of 602.88 m.