Microstructural and tribological properties of nanostructured Al6061-CNT produced by mechanical milling and extrusion

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Wear properties of Al6061-CNT nanocomposites have been studied using dry sliding method. Nanostructured Al6061 powder has been prepared using 30 h mechanical milling of atomized powder, and mixed with different percent of CNT. The yielded mixture was subjected to mechanical milling for 0.5 and 4 h. Mixtures were cold compacted, homogenized and extruded to achieve 13 mm diameter round cross section bars. Relative density and hardness of specimens were determined for extruded samples. Maximum relative density was achieved for specimen without CNT which was about 99.848%, while maximum hardness of 236.1 HV was for specimen with 1.25 wt% CNT. Pin on disk method wear test results indicated that weight loss and wear mechanism strongly depends on normal load. At lower normal load, minimum weight loss achieved for Al6061-1.25 wt% CNT nanocomposite at abrasive and delamination mechanisms, while by increasing of normal load, minimum weight loss achieved for specimen without CNT that its wear mechanism was adhesive and delamination.

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1. Introduction

Nanocomposites are composed of two or more separate elements, one or more of which have dimensions smaller than 100 nm. These elements consist of a matrix and a reinforcing material. Presence of particles or short and long reinforcing fibers usually contributes to strengthen the matrix and provides uniform distribution of the forces applied to the composite. Because of their high strength-to-weight ratio, various applications are considered for Al matrix composites such as automotive, aerospace, and military industries. Carbon nanotubes (CNT), due to their high length-to-diameter ratio, low weight, desirable mechanical, physical, and thermal properties, thermal stability up to 2700 °C in argon atmosphere as well as the self-lubricating property, are the promising candidates for reinforcing composites [1–3]. Since mechanical and physical properties of CNT are conveyed to the matrix, they are employed to produce ceramic [4], metal [5,6] and polymer [7,8] matrix composites. In addition to the potential of CNT in improving the properties of composites, production of nanostructured matrix enhances the composite efficiency more than coarse-grained matrix composites in terms of thermal stability, hardness, and wear resistance.

In recent years, CNT have attracted great attentions due to their excellent mechanical properties such as tensile strength of ~150 GPa and Young’s modulus of ~1.2 TPa [1]. However, because of their severe agglomeration, distribution of CNT in the metal matrix composite by conventional methods is difficult. Also, due to the possibility of chemical reaction between CNT and Al matrix and formation of Al4C3, preparation of these nanocomposites through melting methods is problematic. In contrast, Al-CNT nanocomposite powder has been produced by mechanical milling resulting in the quick formation of nanostructured and nanocomposite materials [9].

Al-Qutub et al. investigated the applied load of 1wt.%CNT reinforced Al6061 composite produced by ball milling and spark plasma sintering (SPS) and compared to Al6061 monolithic alloy. Their finding revealed that, the wear rate increased linearly with the applied load. At lower loads of 5–15 N, the composite displayed better wear resistance. At higher loads of 20–30 N, the wear resistance of monolithic alloy was better than the composite. These results show that addition of 1 wt% CNT improves wear resistance of the Al6061 alloy at lower loads only [10]. Abdullahi et al. [11] investigated the wear behavior of Al-CNT nanocomposite prepared by mechanical milling and hot isostatic pressing. Their findings revealed that the wear rate is decreased as the CNT content is
Table 1
Chemical composition (wt\%) of Al6061 powder.

<table>
<thead>
<tr>
<th>Element</th>
<th>Al</th>
<th>Cu</th>
<th>Mg</th>
<th>Si</th>
<th>Fe</th>
<th>Mn</th>
<th>Na</th>
<th>S</th>
<th>Cr</th>
<th>Zn</th>
</tr>
</thead>
<tbody>
<tr>
<td>Content</td>
<td>Balance</td>
<td>0.39</td>
<td>0.82</td>
<td>0.33</td>
<td>0.50</td>
<td>0.12</td>
<td>0.10</td>
<td>0.023</td>
<td>0.17</td>
<td>0.063</td>
</tr>
</tbody>
</table>

Fig. 1. SEM micrographs of Al6061 powder particles (a) without milling or after milling for (b) 4, (c) 12, and (d) 30 h.

Fig. 2. Variation of Al lattice parameter versus milling time for Al6061 powder.
increased from 0 to 1.5 wt%, while slight increase was observed with increasing the CNT content in the range of 1.5–2 wt%. The CNT content of more than 2 wt% rapidly increased the wear rate. In this research, the effect of nanocrystallization on wear properties of bulk nanostructured Al6061 and Al6061-CNT nanocomposites produced by mechanical milling and extrusion has been studied.

### 2. Experimental procedures

Atomized Al6061 powder with about 70% particle size in range of 63–88 μm was used in this research. The chemical composition (Table 1) was identified using X-ray fluorescence.

As-received Al6061 powder particles was subjected to mechanical milling under an attrition mill using a hardened stainless steel with a diameter of 6 mm and argon atmosphere (99.99% purity) for 0–30 h to obtain nanostructured powder. Ball to powder weight ratio and rotational speed were justified to 10:1 and 500 rpm. To reduce the powder particles cold weld rate, 1 wt% stearic acid \( \text{[CH}_3\text{(CH}_2\text{)}_{16}\text{COOH]} \) was added to the powder.

CNT with 80 nm diameter was employed as reinforcement. To remove catalyst impurities including cobalt and nickel, the CNT was immersed into the concentrated nitric acid (60 wt%) for 12 h. It was followed by rinsing with distilled water for several times until pH reached 7 and drying was carried out in an oven at 120 °C. Finally, they underwent ultrasonic operation at the frequency of 40 kHz in ethanol solution for 90 min. After ultrasonic treatment of CNT, the 30 h milled nanostructured Al6061 powder was gradually added, and ultra-sonication of CNT-nanostructured Al6061-ethanol solution lasts for 30 min at 40 kHz. The beaker containing

### Table 2
Grain size and lattice strain of nanostructured Al6061.

<table>
<thead>
<tr>
<th>CNT wt.%</th>
<th>Milling time (h)</th>
<th>Powder</th>
<th>Bulk nanocomposite</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Grain size (nm)</td>
<td>Lattice strain (%)</td>
</tr>
<tr>
<td>1.25</td>
<td>0.5</td>
<td>44</td>
<td>0.5</td>
</tr>
<tr>
<td>1.25</td>
<td>4</td>
<td>35</td>
<td>0.51</td>
</tr>
<tr>
<td>1.75</td>
<td>0.5</td>
<td>52</td>
<td>0.465</td>
</tr>
<tr>
<td>1.75</td>
<td>4</td>
<td>43</td>
<td>0.485</td>
</tr>
</tbody>
</table>
CNT-nanostructured Al6061-ethanol solution was placed on a magnetic stirrer at 130 °C in order to dry the ethanol solution. Subsequently, ball milling of nanocomposite powders was carried out for 0.5 and 4 h with ball-to-powder weight ratio and rotational speed of 10:1 and 350 rpm, respectively [12]. The above-mentioned procedure was employed to prepare nanocomposite powders with 1.25 and 1.75 wt% CNT.

The bulk samples of Al6061, nanostructured Al6061 and nanocomposites containing 1.25 and 1.75 wt% CNT were produced using cold compaction, homogenization followed by extrusion. Above powders were cold compacted in a steel die under a uniaxial pressure of 475 MPa for 30 min. The samples were exposed to wear test up to the sliding distance of 1000 m and their weight loss was recorded with 0.1 mg accuracy at a normal load of 10 N and sliding speed of 1.1 m/s. Variation of friction coefficient with sliding distance was simultaneously recorded for those samples. The wear behavior (weight loss and friction coefficient) was also studied for nanostructured Al6061 and Al6061–1.25 wt% CNT nanocomposite at a normal load of 20 N and sliding speed of 1.1 m/s. After the wear tests, the worn surface morphology and wear debris were studied by SEM.

3. Results and discussion

3.1. Structural changes during ball milling of Al6061 powder

Fig. 1(a–d) shows the morphology of Al6061 powder without milling or after 4, 12, and 30 h milling. After 4 h milling (Fig. 1b), the powder particles have agglomerated morphology, probably due to the high cold weld rate, which leads to the coarsening of the powder particles as compared to just atomized powder without milling (Fig. 1a). With increasing the milling time to 12 h (Fig. 1c), the work hardening produced in powders makes them susceptible to fracture. For longer periods of time (Fig. 1d), as the fracture overcomes the cold weld process, powders will be finer with an almost equiaxed and homogenous particle size distribution. After 30 h of milling, the size of powder particles is reduced to about 20 μm, whereas the morphology is exactly equiaxed because of the refined grains and homogenous distribution of the particles [15]. Particle size is reduced with increasing the milling time, due to the simultaneous effects of work hardening, which causes embrittlement of particles [9,15], and presence of stearic acid, which suppresses the cold welding phenomenon. The latter is done because the stearic acid sticks onto the surface of powder particles and reduces the surface tension and energy required for powder particles size reduction. These factors yield to the faster reduction of particle size during ball milling. At longer milling times, particle size remains almost constant due to the balance between cold weld rate and their fracture [15,16].

As shown in Fig. 2, and in accordance with the Bragg’s equation, the Al lattice parameter is increased from 4.0653 Å for Al6061 powder to 4.0866 Å after milling for 30 h. As the milling progresses, Mg2Si dissolved and Mg atoms enter the Al lattice and increase the lattice parameter, since the atomic radius of Mg is larger than that of Al. In other words, the formation of solid solution is closely related to the crystalline structure due to the prolongation of milling time [17].

There is considerable amount of energy exerted on the powder by the repeated collision of balls, a portion of which is stored in the form of lattice strain (Fig. 3). These results resolute with the results gaining by the other researchers [15,17,18].

Variations of lattice strain and grain size with milling time can be explained by severe plastic deformation introduced into the powder particles. At the early stages of milling, the number of crystal defects such as dislocations in particle structure rises, and the grain size is reduced at a higher rate. During the milling process,
because of the local temperature rising in powder particles, the recovery phenomenon occurs at a higher rate, leading to the fewer crystalline defects and their new arrangement. The dislocations gradually make a cellular structure and result in the size reduction of grains at a slower rate (Fig. 4). Therefore, as the milling process prolongs, and as a result of more plastic deformation, new dislocations are formed. Consequently, subsidiary frontiers followed by main frontiers are formed and also the primary grains are observed to be divided into smaller grains. Within the final stages of milling process, the rate of production and elimination of dislocations becomes equal [17–19].

3.2. Structural properties of nanocomposites

As indicated in Fig. 4, the grain size for nanostructured Al6061 reached to stable condition after 12 h, and after 30 h milling, 35 nm grain size has been attained. These 30 h milled nanostructured Al6061 powder mixed with CNT and milled again for 0.5 and 4 h. As shown in Table 2 for Al6061-1.25 wt% CNT powder after 0.5 h milling grain size increase to 44 nm in compare to CNT free powder. But after 4 h milling grain size decreases again and reaches to 35 nm which is same as Al6061 nanostructure powder without CNT. This behavior in grain size occurs for Al6061-1.75 wt% CNT, too. As indicated in Table 2 by adding 1.75 wt% CNT to Al6061 nanostructure powder in 0.5 h milling time grain size increases to 52 nm, and after 4 h milling, it decreases to 43 nm.

By comparing above data also it can be seen that increasing CNT percent increases the grain size. After 4 h milling, grain size is 35 nm and 43 nm, for 1.25 wt% and 1.75 wt% CNT, respectively. Also as observed in Table 2, lattice strain decreases with increasing CNT percent.

In fact, a majority of researchers have found that the presence of hard particles during the soft powder milling accelerates the grain refinement and increases the lattice strain in the powder mixture [12,18,20,21].

For Al6061-CNT milled powder after extrusion at 500 °C, nanocomposite has been made. As shown in Table 2 extruded bulk nanocomposites have larger grain size and smaller lattice strain in compare to powders. It can be related to high temperature extrusion, which by restoration mechanisms such as recovery and recrystallization, dislocation density decreases, then internal stress and also strain reduces. Also by grain growth, and joining of grains

![Fig. 6. Variation of friction coefficient for (a) Al6061 (b) nanostructured Al6061 at a normal load of 10 N and sliding speed 1.1 m/s.](image-url)
Fig. 7. Variation of friction coefficient for (a) Al6061–1.25 wt% CNT nanocomposite (b) Al6061–1.75 wt% CNT nanocomposite at a normal load of 10 N and sliding speed 1.1 m/s.

Fig. 8. Weight loss at different conditions as a function of sliding distance at a normal load of 10 N and sliding speed 1.1 m/s.
in high pressure at high temperature, grain size increases. These results are in agreement to another research's results [22,23]. On the other hand Table 2 shows when CNT% increases, grain size increases and lattice strain decreases for bulk extruded nanocomposites. These results can be attributed to agglomeration of CNT which causes nonuniform distribution of nanotubes.

Fig. 5(a and b) shows the morphological variations of nanostructured Al6061 powder mixed with 1.25 wt% CNT and milled again for 0.5 and 4 h. Researchers conducted on nanocomposite powders reveal that the important problem in dispersion of carbon nanotubes with the aluminum matrix is their agglomerating [2,14]. In this study, it seems that ball milling for 0.5 h is not appropriate adequate to detached CNT and diffuse them via the Al6061 particles (Fig. 5a). By increasing milling time to 4 h, carbon nanotubes are restricted by the matrix powder, and the bound up CNT are gradually dispersed and place entrant the Al6061 powders (Fig. 5b).

3.3. Hardness and relative density measurements

As shown in Table 3, the maximum relative density is 99.848%, which is for nanostructured Al6061, probably because of single-phase nature of the sample. Also, as the milling time increase, the sheet-shaped particles break down and the co-axial particles are gradually created. These particles seem has led to the filling of space between coarse particles with fine particles and increasing the density. A similar result has previously been proposed by Jafari et al. [12], Hosseini et al. [20], and Esawi et al. [22]. The mean hardness of this sample was measured to be 203.4 HV. The addition of the reinforcement has been reduced the density due to the two-phase nature of the system and also lower density of nanocomposite. For Al6061–1.25 wt% CNT nanocomposite, the relative density

Fig. 9. SEM micrograph from (a) worn surface (b) wear debris of Al6061 at a normal load of 10 N and sliding speed of 1.1 m/s.

Fig. 10. SEM micrograph from worn surface of nanostructured Al6061 at a normal load of 10 N and sliding speed 1.1 m/s.

Fig. 11. SEM micrograph from (a) worn surface (b) wear debris of Al6061 – 1.25 wt % CNT nanocomposite at a normal load of 10 N and sliding speed 1.1 m/s.
and hardness were 99.645% and 236.1 HV, respectively. The higher hardness of nanocomposite sample can be explained by the dispersed and uniform distribution of CNT and filling the existing micro cavities among the powder particles by CNT [12]. However, due to the formation of a two-phase system, the relative density of this sample is lower than that of nanostructured Al6061. Lower relative density (97.849%) and hardness (193.5 HV) were measured for Al6061–1.75 wt% CNT nanocomposite as compared to other samples. This can be attributed to the existence of agglomerated, entangled and nonuniform CNT at higher weight percentages [22,23].

3.4. Wear properties

According to Fig. 6(a and b), the changes of friction coefficient versus sliding distance for Al6061 and nanostructured Al6061 at a normal load of 10 N and sliding speed 1.1 m/s are within the range of (0.24–0.78) and (0.24–0.53), respectively. The friction coefficient for these samples initially increases and then moves onto a steady state region. This effect is attributed to the removal of sticking impurities of these samples [24]. For nanostructured Al6061, the harder surface indicates a lower pin on steel disk push depth.

By comparing Fig. 7a and b, it is observed that the changes of friction coefficient as a function of sliding distance for nanocomposite containing 1.25 and 1.75 wt% CNT are within the range of (0.2–0.38) and (0.23–0.59), respectively. For Al6061–1.25 wt% CNT nanocomposite, the direct contact between the sample (pin) and the steel disk becomes lower, implying that slip rate of CNT with short array and tube shape between the metallic surfaces of the sample and steel disk increases, leading to a decrease in friction coefficient [13]. Another reason can be the increase in the ratio of yield strength to the Young’s modulus [25] and self-lubrication effect of CNT at this weight percentage [26,27].

![EDS Spectrums from (a) worn surface and (b) wear debris of Al6061 – 1.25 wt% CNT nanocomposite at a normal load of 10 N and sliding speed 1.1 m/s.](image-url)

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For the nanocomposite containing 1.75 wt% CNT, it seems due to high porosity of this sample, the surface becomes unsmooth and more rough than 1.25 wt% CNT, therefore, the steady state of friction coefficient diagram of former specimen (Fig. 7a) is very different from the latter (Fig. 7b).

According to Fig. 8, weight loss was gradually reduced when the weight percent of CNT rose up to 1.25. In other words, for Al6061, nanostructured Al6061, and nanocomposite containing 1.25 wt% CNT, the weight loss after covering the sliding distance of 1000 m was obtained to be 4, 3.6, and 2.9 mg, respectively. The minimum weight loss of Al6061–1.25 wt%CNT nanocomposite can be explained by the uniform distribution of CNT in the Al matrix. However, Al6061–1.75 wt% CNT nanocomposite showed a weight loss of about 5.5 mg for sliding distance of 1000 m. Hence, for this sample there is no homogeneity on worn surface considering the non-uniform dispersion and fracture of CNT [10,13].

Fig. 9(a) illustrates the signs of metal fluidity and peeling in the worn surface of Al6061. These signs indicate a plastic deformation in the sample and effective material transfer from pin surface to the steel disk during the wear test [10,20,24,28]. This worn surface

![SEM micrograph from worn surface of Al6061 – 1.75 wt% CNT nanocomposite at a normal load of 10 N and sliding speed 1.1 m/s.](image)

![Variation of friction coefficient for (a) nanostructured Al6061 (b) Al6061–1.25 wt% CNT nanocomposite at a normal load of 20 N and sliding speed 1.1 m/s.](image)

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morphology represents the adhesive wear mechanism. The delamination wear mechanism is also observed for this sample. As presented in Fig. 9(b), flake formation and the presence of coarse particles firmly confirm plastic deformation [20].

Fig. 10 shows a cleaner worn surface with less surface roughness for nanostructured Al6061 in comparison to Al6061–1.25 wt% CNT nanocomposite. Signs of adhesive and delamination wear mechanisms are detected in the worn surface of nanostructured Al6061.

SEM micrograph from worn surface of Al6061–1.25 wt% CNT nanocomposite are presented in Fig. 11a. The worn surface morphology indicates the presence of a relatively great band distance, with a combination of shallow and relatively deep grooves.
abrasive and delamination wear mechanisms accompanied by fine wear debris (Fig. 11b) can be observed for this sample. Indeed, the nanocomposite surface is exposed to the surface oxidation during the steady wear process, followed by delamination and oxide film fracture. Accordingly, the oxide particles obtained from the broken oxide film lead to an abrasive wear [10,29].

In the lower loads, due to surface protection by carbon nanotubes, the effective interface between the surface of disk and the surface of the pin (aluminum) decreases, and the carbon nanotubes with the aluminum matrix make the wettability and the common interface more appropriate [14,27].

Fig. 12(a and b) shows the EDS analysis of the worn surface and wear debris of Al6061–1.25 wt% CNT nanocomposite at a normal load of 10 N and sliding speed of 1.1 m/s, verifying the stability of the iron oxide layer in this sample. These findings also confirm the presence of iron and oxygen in the wear debris. It seems that, due to the production of fine fragments consisting of iron and iron oxide transferred from steel disk to the wear track, a lower weight loss value and surface fatigue is obtained. The matrix also sustains gradual abrasion and, due to less friction coefficient of the carbon nanotubes and formation of lubricating carbon layer, the abrasive wear has taken place [10]. Also iron oxide layer formation prevents oxidation wear.

By increasing CNT percent, mechanism of wear changes and severe wear is observed for nanocomposites. As shown in Fig. 13, cracks initiation and delamination are wear mechanism for Al6061–1.75 wt% CNT nanocomposite at a normal load of 10 N and sliding speed 1.1 m/s. In other words, cracks initiation has important role in wear of above specimens, and on the other hand significant delamination occurs for this sample which is in distinction to specimens with lower CNT percent or even without CNT. Also as shown in Fig. 8 the most weight loss was observed for 1.75 wt% CNT. Therefore as results indicates, increasing of CNT above the 1.25 wt%, decreases the wear resistance to lower than even specimens which have not any CNT, and as discussed before this can be attached to nonuniform distribution of CNT and also fracture of these hard nanotubes.

Fig. 14(a and b) reveal that the friction coefficient of nanostructured Al6061 and Al6061–1.25 wt%CNT nanocomposite at a normal load of 20 N and sliding speed of 1.1 m/s varies in the ranges of (0.17–0.45) and (0.12–0.5), respectively. For Al6061–1.25 wt%CNT nanocomposite, the friction coefficient profile is accompanied by more significant fluctuations as compared to the load of 10 N (see Fig. 7a). The main elucidation for such mutations in friction coefficient is oxidation on the worn surface threads at higher loads [30].

As shown in Fig. 15a, adhesive and delamination mechanisms are observed for nanostructured Al6061 at a normal load of 20 N and sliding speed of 1.1 m/s. High dislocation density of nanostructured Al6061 stimulates delamination wear mechanism; hence, the work hardening applied on this sample during the wear test yields to dislocations pile up in the subsurface regions. Further increase in dislocations density results in the cracks initiation and its propagation up to the surface layer, bringing forth the surface delamination [10,20,24]. Indications of cracking and delamination are also obvious in the wear debris of nanostructured Al6061 (Fig. 15b).

According to Fig. 15c, for Al6061–1.25 wt% CNT nanocomposite such pores are developed because of the larger part sticking to the other side of the disk and act as a provenance of cracks initiation. Weak bonding between CNT and Al6061 ingredients in sintered specimens could be another inference of cracks initiation and dispersion at higher loads [10,13,31–34]. Also delamination wear mechanism is observed for this sample. The morphology of the wear debris for Al6061–1.25 wt% CNT nanocomposite (Fig. 15d) also indicates the presencement of some cold-welded particles, formed perhaps due to the applied high shear stress to this sample under this load [10,31]. The wear debris of the nanocomposite consists mainly of flake-like similar particles.

At the higher loads, due to higher stress, it is more tolerant of the walls of carbon nanotube cells, the interface of matrix and reinforcement is not strong enough, therefore, segregated parts of carbon nanotubes are transferred to the wear debris and will increase the composite wear. A similar result has previously been proposed by Bastwros et al. [13], Kim et al. [27], and Choi et al. [34].

Fig. 16 shows the changes of weight loss with sliding distance for nanostructured Al6061 and Al6061–1.25 wt%CNT nanocomposite at a normal load of 20 N and sliding speed of 1.1 m/s. The amount of weight loss after covering the sliding distance of 1000 m was obtained to be 4.6 and 4.3 mg for nanostructured Al6061 and Al6061–1.25 wt% CNT nanocomposite, respectively.

4. Conclusions

Al6061-CNT nanocomposites were prepared by mechanical milling, cold pressing, homogenization and extrusion. Structural properties were determined and wear tests were carried out on these samples. Below brief results obtained by experiments:

(1) Minimum weight loss at lower normal load obtained for Al6061–1.25 wt% CNT nanocomposite and the wear mechanism was abrasive and delamination.
(2) With increasing of normal load, minimum weight loss achieved for nanostructured Al6061 without CNT sample and the wear mechanism was adhesive and delamination.
(3) For nanostructured Al6061 without CNT sample by changing the normal load wear mechanism does not change, but for Al6061–1.25 wt% CNT nanocomposite at higher normal load wear mechanism was cracks initiation and delamination.
(4) Maximum relative density was achieved for nanostructured Al6061 without CNT sample was about 99.848%, while maximum hardness of 236.1 HV was obtained for Al6061–1.25 wt% CNT nanocomposite.

Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at https://doi.org/10.1016/j.apt.2017.12.026.

References


