WEAR BEHAVIOUR OF CARBON NANOTUBES REINFORCED NANOCRYSTALLINE AA 4032 COMPOSITES

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Abstract. The present paper emphasizes the friction and wear properties of Carbon Nanotubes reinforced AA 4032 nanocomposites prepared by powder metallurgy technique. CNTs are multi-wall in nature and prepared by electric arc discharge method. Multi-walled CNTs are blended with AA 4032 elemental powders and compaction followed by sintering to get bulk nanocomposites. The strength of the composites has been evaluated by microhardness and the surface contact between the nanocomposites and EN 32 steel has been evaluated by Pin on disk tester. The results are proven that reinforcement of CNTs play a major role in the enhancement of hardness and wear.

1. INTRODUCTION

Aluminum and its alloys, being the most abundantly used non-ferrous structural materials, were the first choice for reinforcement with carbon Nanotubes. Due to their extraordinary properties, CNTs caught the attention of researchers and work on development of CNT composites started at a tremendous pace in the area of structural, automobile and aerospace industries [1, 2]. There are several challenges such as dispersion and non wetting nature in the fabrication of MMCs with CNT reinforcement. Carbon nanotubes proved to have the potential to produce the strongest composites [3,4]. Many applications have been projected for CNT metal matrix composites based on the mechanical and functional properties of CNTs. Much research is still underway for overcoming the challenges and understanding the behavior of these composites [4]. The reinforcement of aluminum alloys with carbon nanotubes leads to a new generation of tailor-made engineering materials with improved properties such as strength to weight ratio for structural applications. Sliding wear or adhesive wear is defined as the transfer of materials from one surface during sliding motion[5-10]. The sliding wear of the aluminum composites which are substitute for the lining and piston materials can be extensively studied to find the possible aluminum based composites. In the present article, the dry sliding wear behavior of aluminum matrix nanocomposites containing different mass fractions of carbon nanotubes was investigated.

2. EXPERIMENTAL PROCEDURES

The constituent elemental powders of AA 4032 alloy were taken in a cylindrical shear mixer of 1000 ml capacity and mixed at 400 rpm for 30 minutes. The matrix and the multi-walled carbon nanotubes (MWNTs) at x wt. % (x = 1, 1.5 and 2) were blended using low energy ball mill for 20 minutes. The blended powders were compacted followed by sintering at 600 °C for 90 minutes to produce bulk composites.

The density was determined using Archimedes’ method and the hardness of the starting surfaces were evaluated using a micro-Vickers pyramid and 200g load with six indents per sample. A Pin on Disc Tribo tester was used in this work for investigating the tribological behavior of the composites prepared. The size of the pin was 6 mm in diameter and the 25 mm in length. The alloy and composites were used as the test material. The counter disc with 55 mm OD and 10 mm thick made of hardened chromium steel (EN 32) was used in these experiments. The test pins were loaded against the disc with dead weight. Specimens were ultrasonically cleaned in acetone before testing. Sliding...
wear tests were conducted at four different normal loads of 5, 10, 15 and 20 N at sliding velocity of 0.1571 m/s. Tests were performed under unlubricated condition at room temperature (30˚C; 60 ± 5% RH). Frictional force and displacement of specimens were measured at periodic intervals. The wear rate was calculated from weight loss measurements. To investigate the wear mechanism, worn surfaces and subsurface regions were observed under SEM.

3. RESULTS AND DISCUSSION

3.1 Microstructure

Fig. 1 shows the TEM picture of the multi wall CNTs synthesized by arc discharge technique [11].

Figure. 1. TEM picture of (a) as received CNTs ; (b) Purified CNTs ; (c) Raman spectra of CNTs

The soot produced from arcing is shown in Fig (a). it is observed that more impurities such as graphitic particles, nanorods and other nano particles are present in sample. All the impurities are removed by physical and chemical purification steps. The TEM microstructure and Raman spectra of purified CNTs are shown in Fig. (b & C). The observation of characteristic multi-peak features around 1339 and 1574 cm⁻¹ provides a signature of carbon nanotubes.

The purification treatment consist of four steps

Step 1
The crushed powder was heated in a closed muffle furnace. All the amorphous carbon materials were burned off by heating the soot at 650 C for 1 hour.

Step 2
The heat treated CNTs were washed in distilled water and treated with toluene for 5 hours. During this step the fullerenes and the soluble impurities were removed. Then the sample was dried in air at 100 C.

Step 3
In this step, the sample was subjected to liquid phase oxidation with 20 % hydrogen peroxide for 2 hours. By this way a large amount of amorphous carbon was eliminated. Then the sample was
was washed with distilled water and dried in air at 100°C. The purification in liquid phase oxidation leads to surface modification of carbon nanotubes.

**Step 4**

Then the sample was ultrasonicated in acetone for about 30 minutes so as to avoid the agglomeration of CNTs and subsequently dried in air. This causes an increase in the isolation of MWNTs. In this technique, the particles are separated due to ultrasonic vibrations. Agglomerates of different nanoparticles were forced to vibrate and become more dispersed.

The TEM microstructure of CNT reinforced AA 4032 is shown in Fig 2. Aluminum particles are uniformly mixed with nanotubes along with other constituent elements. The structure of the CNTs was not damaged or altered after sintering. The structure of CNTs will not change up to 740°C during heat treatment and it was reported by Ajayan et al. [4].

![TEM microstructure of CNT reinforced AA 4032 composite](image)

**Figure 2.** TEM microstructure of CNT reinforced AA 4032 composite

**3.2 Microhardness**

The results of hardness tests are plotted as a function of wt. % of MWNTs shown in Fig. 2. This indicates that the hardness of the composites is increased from 63 to 120 with increasing wt. % of MWNTs. It is evident that the hardness of the composite increases with increasing the mass fraction of CNTs, but then decreases with further increase in the weight fraction. The CNTs retard the grain growth during sintering and strength got improved. The strengthening mechanism for this study is orowan strengthening [12].

![Microhardness of CNT/AA 4032 composites](image)

**Figure 3.** Microhardness of CNT/AA 4032 composites

**3.3 Friction and wear**
The effects of CNT content on the wear rates and coefficient of friction of the composites under 5, 10, 15 and 20 N of loads are discussed. The morphology of worn surface studied through SEM is also shown.

Fig. 3 shows the effects of CNT content on the wear rates and coefficient of friction of the composites under 5, 10, 15 and 20 N of loads. Within the range of CNT content from 0 to 2.0 %, the wear rate of the composites shows a steadily decreasing trend with increasing content of CNTs. The favorable effects of CNT on wear resistance are attributed to their excellent mechanical properties and the efficiency reinforcement to Al matrix [13]. The hardness of the composites increased initially and then start to decrease, but the hardness more than the base alloy and the CNTs act as solid lubricating nature. Hence the wear resistance is increased with the addition of nanotubes.

The coefficient of friction of the composites under 5, 10, 15 and 20 N loads is plotted in Fig. (b). Addition of CNTs lowers the friction coefficient of the composites. The surface contact area is reduced in between the composite and the steel disc due to the presence of CNTs within the matrix. The wear resistance of the composites is explained as follows,

(i) The self lubricating nature of the CNTs decreases the friction coefficient of the composites
(ii) The contact surface proportion of composite and the counterpart disc is a nonlinear function of load because the composite and the steel disc is an elastic-plastic contact
(iii) More carbon film can cover the wear prone surface. The surface of the composites under gone wear test

3.4 Analysis of worn surfaces using SEM

The SEM micrograph of the worn surfaces of AA 4032 alloy and the composite specimen sliding at room temperature are shown in Fig. 4. It can be seen that wear track was covered with debris, cleaning of the specimen in an ultrasonic bath with acetone can remove some of the loose debris. The surface shows a lot of scratches due to wear. The primary appearance suggests abrasion is predominant wear mechanism at dry sliding conditions. Surface projections of asperities present in the contact surface plastically deform and eventually weld together by high local pressure during relative motion between the contact surfaces. As sliding continues these bonds break up producing microcavities which causes tiny particle abrasion.
Figure 5. Worn surfaces of (a) AA 4032; (b) 1% CNT/AA 4032; (c) 1.5% CNT AA 4032; (d) 2% CNT/AA 4032

There is evidence of adhesion and ploughing on the Al worn surface, which shows a distinct characteristic of abrasive and adhesive wear. According to Tu et al [14], oxidation wear mechanism is the predominant in Al/CNT composites. But in the present work, some oxidation wear also occurred due to formation of oxide layer during abrasion. However, according to the SEM images of worn surfaces of the composite, there exist some delamination of surfaces. The cracks extended later and caused break and split of the hardened surface layer by shear deformation of the surface. The oxidation of the worn surfaces along with elemental analysis is shown in Fig. 6.

Figure 6. (a) SEM picture; (b) EDS analysis of worn surface
The same kind of results also reported by Kim et al in the Copper- carbon fiber composites [15]. It is suggested that the abrasive and adhesive wear of the composites is slighter than pure aluminum due to increases of hardness and self-lubrication of carbon nanotube, and the delamination wear becomes important.

4. CONCLUSIONS
There is no linear relationship found in improvement of hardness and strength values with the addition of carbon nanotubes which is attributed to the presence of Vander Waals forces. The strengthening mechanism of the composites is found to as orowan strengthening. The tribological behavior of the microcomposites is improved with the addition of MWNTs to the matrix AA 4032 alloy. The mechanism of wear was found to be predominantly the oxidation and ploughing.

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