Effect of multiple reinforcements (CNT/Si$_3$N$_4$) on hardness, electrical conductivity and friction coefficient of aluminium hybrid composites

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Abstract: Present investigation reports on the development of aluminium hybrid composites using powder metallurgy technique. Two different reinforcements namely silicon nitride (Si$_3$N$_4$) and carbon nanotubes (CNT) were used to reinforce pure aluminium matrix. Ball milled powder morphology and microstructure of hybrid composites were studied using scanning electron microscope to study the dispersion of Si$_3$N$_4$ and CNT. Microstructure studies showed uniform dispersion of both the reinforcements in aluminium matrix. Microhardness was obtained by applying a load of 50 N for dwell period of 20 seconds. Electrical conductivity in terms of %IACS was obtained using Eddy current tester. Coefficient of friction was obtained by conducting scratch test by employing a load of 10 N for a stroke length of 10 mm and scratch velocity of 0.2 mm/sec. Microhardness was found to increase with increasing content CNT content while electrical conductivity was found to decrease. Finally coefficient of friction was found to decrease with increase in CNT content owing to its lubricating nature.

Keywords: Carbon nanotubes; Powder Metallurgy; Hardness; Coefficient of friction.

1. Introduction

Aluminium based hybrid composites with multiple reinforcements have been gaining huge attention owing to their multifunctional properties. These composites not only have good mechanical properties but also possess improved wear and thermal properties. Improvement in these properties largely depends on choosing right reinforcements as their addition is expected to benefit not only in strengthening but also on thermal and wear properties. At present most commonly used reinforcements are based on oxides, carbides and borides such as TiO$_2$, ZrO$_2$, Al$_2$O$_3$, TiB$_2$, ZrB$_2$, B$_4$C, SiC and TiC. Latest addition to reinforcement list is carbon materials like carbon nanotubes, graphene, nanodiamond and fullerene [1-5]. Most of these reinforcements have very high hardness, high Young’s modulus, high compressive strength, low coefficient of thermal expansion and elevated service temperature. The reinforcement combination used in hybrid composites can have different size like for example both of them can be of micron or nano size or can be combination of both. For instance Prakash et al [6] used micron size graphite and nano size Al$_2$O$_3$ particles to reinforce Al6061 alloy. The hybrid nanocomposite with varying graphite content was developed using friction stir processing technique. In another work, Reddy et al [7] developed Al6061 hybrid nanocomposites reinforced with SiC and graphite nanoparticles. The composites were developed using ultrasonically assisted casting technique where molten metal was stirred for 10 minutes to mix nanoparticles with matrix. Dispersion of reinforcements in the aluminium matrix is very crucial since most of the properties are highly dependent on it. Especially liquid metallurgy technique can lead to clustering of nanoparticles as well as micron size particles owing to their poor wettability [8,9].

Selection of right blending and processing techniques has significant influence on properties of hybrid composites. For instance in the development of composites with nanoparticles or multiple reinforcements powder metallurgy technique is often preferred over casting technique. Primary concerns related to casting techniques are homogenous dispersion and wettability of reinforcement with the metal matrix. In addition to this near net shapes with little or no machining can be produced using powder metallurgy. In powder metallurgy technique, blending metallic powders with reinforcement particles in ball mill or ultrasonication method ensures good dispersion. In their work, Farhadinia et al [10] used...
planetary ball mill to mix the raw materials followed by hot pressing at 500°C. The reinforcements, $\text{ZrB}_2$, $\text{TiB}_2$, and $\text{Al}_2\text{O}_3$ particles were found to be uniformly dispersed in the aluminium matrix. In their work, Megahed et al. [11] used powder metallurgy technique to develop $\text{SiC}$ and $\text{Al}_2\text{O}_3$ particles reinforced aluminium hybrid composites. Dispersion of both the reinforcements was found to be uniform throughout the matrix leading to an increase in yield strength and wear resistance. Likewise, most of the research works are focussed on improving the dispersion of reinforcements in the metal matrices so that the possibility of clustering is avoided which otherwise can have detrimental effects on mechanical and thermal properties. Taking a cue from this, in this work effect of multiple reinforcements like carbon nanotubes (CNT) and silicon nitride ($\text{Si}_3\text{N}_4$) on hardness, electrical conductivity and coefficient of friction of aluminium hybrid composites is studied.

2. Experimentation

To synthesize aluminium hybrid nanocomposites, multiwalled carbon nanotubes (Outer diameter: 15-40nm, purity: 95.5%, density: 2.6g/cm³) was used as a primary reinforcement and silicon-nitride (average particle size: 2-3μm, purity: 98%, density: 3.2g/cm³) was chosen as secondary reinforcement material. Matrix chosen was commercial purityaluminium powder (average particle size: 20-45μm, purity: 98%, density: 2.71g/cm³). The hybrid composite powders with different CNT content (0 - 3%) and fixed $\text{Si}_3\text{N}_4$ content (1%) were prepared by adopting two step blending process. Using pure ethanol solution as a dispersion medium, the reinforcement’s powders were stirred for about 60 minutes at 600rpm. Then powder samples were allowed to settle down, separated from the ethanol solution by filtering it and dried in heating furnace till it dries completely. Horizontal type ball mill of 500g capacity was used for blending the aluminium and both the reinforcements. The parameters used for blending were, ball to powder ratio of 5:1, speed of about 300rpm and milling duration of 7 hours. The blended composite powders were subjected to cold compaction by applying a pressure of 300MPa using single acting hydraulic press. The compacted specimens were then sintered in alumina tube furnace under argon atmosphere for about 3 hours at 570°C. Morphology of ball milled powders, microstructure of sintered specimens and scratch surface were observed using scanning electron microscope (SEM, Make: VEGA3 TESCAN). Vicker microhardness test was performed by applying a load of 50 N for a dwell time of 15 seconds. Average of nine microhardness readings is reported in upcoming section. The electrical conductivity was measured by using eddy current tester (Make: AUTOSIGMA-3000, G.E. Technologies). Scratch test was performed using Scratch Tester (Make: Ducom Instruments, India) to obtain coefficient of friction. Tests were conducted by applying a normal load of 10 N over a scratch length of 10 mm with a scratch velocity of 0.2 mm/sec.

![Fig. 1 SEM micrographs of sintered (a) Pure aluminium and (b) Al/1%Si$_3$N$_4$/3%CNT hybrid composite](image)

3. Results and Discussion

3.1 Microstructure and microhardness

SEM micrographs were taken to study the dispersion of both the reinforcements in the aluminium matrix and are presented in Fig. 1. Clear grain boundaries with large grain sizes in the range of 15 to 40 μm were seen for pure aluminium matrix as shown in Fig. 1 (a). On the other hand both CNT and $\text{Si}_3\text{N}_4$ were seen at both inside the grain and at the grain boundaries of Al/1%$\text{Si}_3\text{N}_4$/3%CNT hybrid composite as shown in Fig. 1 (b). The dispersion of $\text{Si}_3\text{N}_4$ particle was found to be uniform throughout
the matrix and can be seen in the micrograph as white particle. Clustering of either of the reinforcement particle was not observed in the Fig. 1 (b) which indicates that the selection of two step blending process to be efficient for dispersion of the reinforcements. Microhardness of unreinforced aluminium and hybrid composites are presented in Table 1. It can be observed that for unreinforced aluminium and Al/1%Si₃N₄/3%CNT hybrid composite the microhardness was about 37.89 and 73.62 VHN respectively. It can be seen that after addition of 3% CNT and 1%Si₃N₄ the increase in microhardness was about 94%. Significant improvement in microhardness can be attributed to high hardness of both the reinforcements and their effect of microstructure. The changes in microstructure include grain refinement and generation of dislocations due to difference in coefficient of thermal expansion mismatch of constituents of hybrid composite [11,12].

![Fig. 2 Snapshot of coefficient of friction during scratch test and SEM micrographs of scratch surface of sintered (a, c) Pure aluminium and (b, d) Al/1%Si₃N₄/3%CNT hybrid composite](image)

Table 1. Microhardness, COF and Conductivity of Al and its hybrid composites

| Composition | Microhardness (VHN) | COF  | Electrical conductivity, %IACS |
|-------------|---------------------|------|-------------------------------|
| Pure Al     | 37.89               | 0.395| 54.90                         |
| Al + 1%Si₃N₄| 47.75               | 0.416| 47.70                         |
| Al + 1%Si₃N₄ + 1%CNT | 54.37 | 0.250 | 36.40 |
| Al + 1%Si₃N₄ + 2%CNT | 62.82 | 0.232 | 29.08 |
| Al + 1%Si₃N₄ + 3%CNT | 73.62 | 0.219 | 23.58 |

3.2 Coefficient of friction

Scratch test was conducted on unreinforced aluminium and hybrid composites and the coefficient of friction (COF) obtained from the test are presented in Table 1. It can be seen that COF value of unreinforced aluminium is 0.395 and for hybrid composites the COF tends to decrease with the increase in CNT content. For 1% and 3% CNT, the COF was 0.232 and 0.219 respectively. The reduction in COF of hybrid composites can be attributed to lubricating nature of CNTs which tend to reduce the
friction during scratch test. Further the snapshots of COF being recorded as function of scratch length is shown in Fig. 2 (a) and (b) for unreinforced aluminium and Al/1%Si$_3$N$_4$/3%CNT hybrid composite. From these two images it can be observed that the fluctuation in unreinforced aluminium is found to be greater than that of Al/1%Si$_3$N$_4$/3%CNT hybrid composite. Minimal fluctuation in scratch test can be attributed to CNTs which act as solid lubricants. Further the SEM images of scratch surface taken for unreinforced aluminium and Al/1%Si$_3$N$_4$/3%CNT hybrid composite is shown in Fig. 2 (c) and (d). Preliminary investigations via SEM micrographs reveal shallow depth and less material loss for hybrid composite when compared to that of unreinforced aluminium. Both the reinforcements pose significant resistance to plastic deformation caused by the indenter leading to shallow depth and less material loss.

3.3 Electrical conductivity

Electrical conductivity test was conducted on unreinforced aluminium and hybrid composites and obtained value from the test in %IACS (International Annealed Copper Standard) are presented in Table 1. One can observe that addition of reinforcements tend to decrease the electrical conductivity of unreinforced aluminium. For unreinforced aluminium and Al/1%Si$_3$N$_4$/3%CNT hybrid composite, the electrical conductivity was 54.90 and 23.58 %IACS. It can be seen that drop in electrical conductivity was about 57.04% and is attributed to grain refinement caused by addition of multiple reinforcements and electron scattering at matrix/reinforcement interface [12,13].

Conclusions

In summary, CNT and Si$_3$N$_4$ reinforced aluminium hybrid composites were successfully synthesized by two step blending process and powder metallurgy route. The developed hybrid composites showed uniform dispersion of reinforcements with no visible clustering. The microhardness of hybrid composites was found to increase with the increase in CNT content while electrical conductivity was found to decrease. Finally the coefficient of friction tested by scratch test revealed significant reduction in friction coefficient due to lubricating nature of CNTs.

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