Mechanical and metallurgical studies of multi-walled carbon nanotube–reinforced aluminium metal matrix surface composite by friction stir processing

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Abstract
The aluminum alloy 5083 was reinforced with multi-walled carbon nanotubes (MWCNTs) by using friction stir processing (FSP). Different MWCNT volume percentages of 0, 6.06, 12.12, and 18.18% were reinforced into the 5083 aluminium metal matrix. Tensile tests were carried out, and a 24% increase was achieved with 12.12% MWCNTs. Hardness was obtained within the stir zone with the 6.06% of MWCNTs. Scanning Electron Microscopy (SEM) analysis revealed the breakdown of MWCNTs, thus leading to uniform dispersion. Wear rate decreased by 18% with an increase in MWCNTs.

Keywords
Multi-walled carbon nanotubes, SEM, Wear rate, Tensile strength, FSP.

1. Introduction
Aluminum alloys play a major role in the aircraft, shipbuilding, and automotive industries because of their superior strength-to-weight ratio. The aluminum alloy 5083 0 has been used in the shipbuilding industry due to its corrosion resistance properties. It is a nanoparticle that has unique properties based on its structure. Moreover, the presence of carbon results in a very high wear resistance. Its high tensile strength along with its good electrical properties makes it a desirable reinforcement for aluminum alloys. Friction stir processing is a solid-state processing technology that has acquired significant attention in recent years because of the solid-state processing wherein the mixing of materials takes place in the plastic state. This removes the disadvantages of liquid-state processing such as casting, which occurs in high temperatures and leads to various defects, in turn reducing the strength of the composites. These were the motivation behind the current study. In the present study, MWCNTs were reinforced in the magnesium-based aluminum alloy 5083 0. This study exploits the presence of carbon as a self-lubricating feature.

The following are the objectives of this study:
• To reinforce multiwalled carbon nanotubes in an aluminum matrix by friction stir processing.
• To test the fabricated surface aluminum nanocomposites for various mechanical and wear properties.
• To evaluate the microstructure of the fabricated composite.

2. Literature review
Presently, a significant attention is being to the influence of the mixing technique on carbon nanotubes (CNTs). The chemical and thermal stability properties of CNTs and their high yield strength and elastic modulus make them the best option for reinforcement. The AZ31 aluminum alloy reinforced with CNTs exhibited a significant increase in wear compared to the currently available alloys [1]. Addition of 2.0 wt% of CNTs to the 2024 aluminum alloy led to a considerable grain refinement, but shortening of CNTs occurred during the ball-milling process [2]. Independently reinforced n-Al2O3 composites showed a lower wear rate than independently reinforced CNTs; however, its coefficient was higher [3]. In additive metallurgy, dispersion led to a better and uniform dispersion than in ball milling [4]. The maximum tensile strength was achieved with a 75/25 volume ratio of CNTs and cerium oxide [5]. The CNTs...
embedded uniformly in the stir zone and were not affected by the thermomechanical processing due to their multiwall structure [6]. It was observed that aluminum metal matrix nanocomposites are very sensitive to the directional behavior of CNTs, nonstraight shape, and aspect ratio [7]. Damaged carbonaceous products were found in the stir zone due to severe plastic deformation and shear stress [8]. Compact bonding led to uniform dispersion of CNTs in the Mg–6Zn matrix [9]. The distribution of reinforcement particles was uniform in the stir zone of AA6063 and was refined due to the thermomechanical effect of the FSP process [10]. The tangled multi-walled carbon nanotubes (MWCNTs) could not bear the thermomechanical conditions imposed during friction stir processing (FSP) [11]. The flow stress and the Zener–Hollomon parameter improved with a decreasing temperature an and increasing strain rate. The addition of CNTs led to a significant increase in the activation energy of plastic deformation, corroborating the enhanced resistance of the nanocomposite to hot deformation [12] Wh. The flow stress and the Zener–Hollomon parameter improved with a decreasing temperature an and increasing strain rate. The addition of CNTs led to a significant increase in the activation energy of plastic deformation, corroborating the enhanced resistance of the nanocomposite to hot deformation [12]. When the RHA reinforcement is more than 50%, hardness values are not affected by the presence of graphite. Residual stresses without reinforcement depend on process parameters such as traverse speed, rotational speed, and tool pin profile. But the hardness was higher in the stir zone of the FSP Al-A2O3 nanocomposite than in alloys without particle materials [13]. Commercially pure aluminum powders (99.9% with 30 nm size) and alumina powders were cold-compacted and friction stir-processed [14]. The resulting ultrafine grains significant increased during hardening, thus improving the mechanical performance [15]. AA5052/A2O3 surface composites friction stir-processed with 3 or 4 passes exhibited superior tensile and yield strengths [16]. When A356 was reinforced with A2O3 using FSP, the surface composite obtained showed a low friction coefficient and wear rates compared to the nonreinforced material [17]. So far, based on the literature, a substantial amount of work has been carried out on FSP of MWCNTs. But FSP on Aluminum Alloy 5083 o is yet to be explored. In this work, a detailed analysis of the effects of MWCNTs has been carried out and reported.

3. Materials and methods

Aluminum alloy 5083 with magnesium and traces of manganese and chromium were identified for this study due to its high corrosion properties and most upper strength in the category of non-heat-treatable alloys. MWCNTS were used due to their high strength, aspect ratio, and modulus. The specifications of the MWCNTs used and the chemical composition of the AI5083 aluminum alloy are shown in Table 1 and Table 2, respectively.

The schematic of our work is shown in Figure 1. A groove was cut and compacted with MWCNT particles. FSP was carried out using a tool. The dimensions of the tool are shown in Figure 2. The process parameters identified were a force of 20KN, a feed of 30mm/min, and an rpm of 1300. Four specimens of dimensions 130×75×8 were made by the wire-cut EDM method. These grooves were filled with 6.06, 12.02, and 18.08 vol% vol of nanoparticles, respectively. The composition is used based on groove depth and dimensions of the workpiece.

The geometry of the FSP tool is mainly related to the dimension of the base material as well as its material flow characteristics. The specimens were machined to a dimension of 6 mm ×6 mm ×30 mm. Height loss was monitored using a computer-aided data acquisition system. The friction stir-processed plates are as shown in Figure 3. The surface of the specimen was slid on a hardened chromium steel disc. The parameters used for the test are a sliding velocity of 15 m/s, a normal force of 25 N, and a sliding distance of 2500 m.

| Table 1 | Chemical composition of Al5083 alloy |
|----------|--------------------------------|
| Elements | Si | Fe | Cu | Mn | Mg | Zn | Ti | Cr | Al |
| Percentage composition | 0.4 | 0.4 | 0.1 | 0.4-0.1 | 4-4.9 | 0.25 | 0.15 | .05-0.25 | Bal |

| Table 2 | Specifications of MWCNT TYPE 5 |
|---------|--------------------------------|
| Appearance | Black |
| Appearance (form) | Powder |
| Assay | Mn 95% |
| Length | 10 - 30 μm |
| OD | 30 – 50 nm |
Figure 1 Schematic flow of work done

Figure 2 Tool dimensions used for the FSP process

Figure 3 Crown appearance of the FSPed sample
4. Results and discussion
4.1 Tensile behaviour

Figure 4 shows an increase in the ultimate tensile strength (MPa), which might be due to the Orowan looping mechanism, leading to the formation of back stress and an increase in dislocation density. This forms a barrier for further movement of dislocations and increased lattice strain. Shear lag is due to the load transfer from the matrix to the CNTs. The strain misfit between the elastic reinforcement and the plastic matrix might also contribute to the increased tensile strength [18, 19]. Orowan strengthening may be the mechanism behind the increase in strength. This strengthening is primarily due to the interaction between the lattice dislocations and CNTs. The critical interparticle distance is achieved by fragmentation of the CNTs [20]. The stirring action of the pin might be the reason for the uniform distribution of the CNTs, resulting in superior mechanical properties [21]. MWCNTs are present as nucleation sites for void formation at a reinforcement percentage of more than 5%. This nucleation might start recrystallization at an appropriate high temperature [22]. Formation of Al4C3 may not be possible since the operating temperature for this process is only around 8000°C and a higher operating temperature is needed. This could also have increased the tensile strength since it is reported that the formation of Al4C3 in the aluminum/CNT interface would increase the load-bearing capacity of the composite [23]. The mechanisms mentioned above may be further validated by the composite strength model. The higher the aspect ratio, the higher the strength of the composite, thus having a notable effect on strength [24]. However, the effect of aspect ratio for randomly distributed MWCNTs is just half the actual aspect ratio [25]. The refined microstructure due to FSP leads to severe plastic deformation and a hindrance to crack growth is established, thus increasing elongation [26]. The dispersion of MWCNTs is better in 6 and 12 percentage, thus strengthening the composite. This might be due to the reinforcement filling the voids present in the matrix. But a further increase in MWCNTs results in deterioration of the strength, which might be due to the reduction in the densification process and thus the decrease in the density of the composites. The critical length of the MWCNTs also plays a major role in the strength of the composite fabricated. The critical length equation validates this for fiber reinforcements can be applied for MWCNTs also for load transfer strengthening mechanism. The significant length is given by Equation 1 [27]:

\[
l_c = \frac{\sigma_{c} \cdot \sigma_{CNT}}{2 \cdot \tau_{m}}
\]

where the multiple mechanism theory is given by Equation 2:

\[
\sigma_{c} = \sigma_{m} + \Delta \sigma_{LT} + \Delta \sigma_{LM} + \Delta \sigma_{OROWAN}
\]

where \(\sigma_{c}\) is the strength of the composite, \(\sigma_{m}\) is the strength of the matrix, \(\Delta \sigma_{LT}\) is the improved strength due to load transfer, \(\Delta \sigma_{LM}\) is the improved strength due to thermal mismatch and \(\Delta \sigma_{OROWAN}\) is the improved strength due to Orowan looping. The above theory is based on the dislocation theory and thus plastic deformation should be considered [28].

4.2 SEM (Scanning electron microscopy) analysis
From Figure 5, it may be observed that the CNTs are deformed and fractured to form a homogenous distribution. This might be due to the severe plastic deformation and shear strain developed in a short
duration. Grain refinement transformed from being discontinuous to continuous dynamic recrystallization due to the hard inclusions. The CNTs were sheared due to the load applied through the tool pin during FSP. There is a reduction in grain size from the average size. The MWCNTs in the grain boundaries of the aluminum matrix impeded grain growth and further resulted in enhanced mechanical properties due to the microstructure. Notable bonding was observed between the matrix and CNTs. Since aluminum has 12 slip systems, grain refinement can be conveniently obtained by plastic deformation and thus an increase in hardness [29, 30].

![Microhardness test](image)

**Figure 5** The effect weight percentage of MWCNTs on the MicroHardness

5. **Microhardness test**

The increase in hardness in the stir zone might be attributed to either of these mechanisms: (i) Orowan strengthening, also known as dislocation bowing around particles, and (ii) grain size refinement. In the former, the dislocations form a loop around the particle, called the Orowan loop [31]. Grain or substructure strengthening is shown in *Figure 6*. Grain size refinement can increase hardness based on the Hall–Petch equation. The coefficient of thermal expansion, being different for both the matrix and the reinforcement, also contributed to the hardness upshot [32]. The nanosize of the reinforcement particle leads to the pinning of dislocations, leading to the reduction in grain growth. This pinning effect stops the dislocations from further sliding due to grain boundary [33]. Also, the hindrance of the movement of dislocations can be attributed to the presence of MWCNTs due to which elastic modulus mismatch and thermal expansion mismatch occur between the MWCNTs and the aluminum matrix. At 5% and more of MWCNTs, the strain hardening effect can also be attributed to the increase in strength compared to the base metal [34]. In nano-sized reinforcements, the dislocation density evolved by athermal storage of dislocations due to dislocation pinning and annihilation of these dislocations due to dynamic recovery [35]. This might lead to plastic deformation from conventional dislocation slip to grain boundary migration. Strengthening due to carbon atoms is not possible since the solubility of carbon atoms in aluminum atoms is very low at low temperatures [36]. Higher restriction to localized matrix deformation can also be attributed to an increase in hardness [37].

**Wear behaviour**

The inclusion of MWCNTs reduces wear loss as they hinder the crack propagation. Also, MWCNTs can remain between the two contacting surfaces and diminish the direct contact effect. Furthermore, the frictional temperature developed can be reduced by the presence of MWCNTs. Thus at optimum concentration, MWCNTs can provide good tribological properties (*Figure 7*).

Moreover, the aspect ratio, orientation, and interaction with the matrix contribute to an improvement in these properties [38]. Formation of a graphene layer might be one of the factors that decrease in wear rate since there will be an increase in layers formed with an increase in the quantity of MWCNTs reinforced in the matrix [39]. Oxidation between the matrix and MWCNTs is inhibited and does not occur. The existence of the peeling surface of the MWCNTs on the surface of the composite also contributes to the reduction in wear 12.02 percentage by avoiding plowing [40]. Also, the increase in hardness decreases wear rate. A great reduction in direct contact between the nanocomposite surface and the disc also reduces wear rate. The primary mechanism is the load-bearing
ability of the MWCNTs in the aluminum matrix-reinforced interface [41]. The MWCNTs mainly improve the strength and hardness of the matrix, and thus better resistance to plastic deformation is achieved, leading to the reduction in the plowing effect of the reinforcement from the matrix. This can also be attributed to a decrease in weight loss with an increase in MWCNTs [42, 43].

![Figure 6 Effect of weight percentage of MWCNT on the wear rate](image)

**Figure 6** Effect of weight percentage of MWCNT on the wear rate

![Figure 7 SEM Results](image)

**Figure 7** SEM Results for (a) FSP of (b) Al5083 with 6% vol CNT (c) Al5083 with 12% vol CNT (d) Al5083 with 18% vol CNT

### 5.1 Limitations

FSP could produce acceptable results in the current study. But there were a few limitations to the severe plastic deformation due to the process. This could be overcome by employing a pre-heating system or a post-heating system to avoid generation of stress.

### 6. Conclusion and future work

The tensile strength followed a downward trend with the increase in the volume percentage of reinforcement. This clearly shows that the new composites formed exhibited a lower tensile strength when compared to AA 5083 under FSP. The distribution of reinforcements, microstructure, and bonding has been observed using SEM photographs. The wear rate decreases with the inclusion of MWCNTs but an increase in MWCNT concentration from 12.02% to 18.08% leads to a surge in the wear rate. Wear Rate is closely associated with the hardness of the composite: the harder the surface, the less the wear rate. The microhardness value graph clearly shows an increase in hardness when MWCNTs are added.

Wear mechanisms and fracture mechanisms could be further studied by employing a new methodology to avoid the damage to the structure of the MWCNTs.

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### Conflicts of interest

The authors have no conflicts of interest to declare.

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