

Study of distribution of Carbon nanotube in Al-CNT nanocomposite synthesized via Spark-Plasma sintering

A.Maiti¹, and T. Laha²

¹Department of Mechanical Engineering, C V Raman Collage of Engineering, Bhubaneswar-752054, Orissa, India

²Department of Metallurgical & Materials Engineering, Indian Institute of Technology Kharagpur, Kharagpur- 721302, West Bengal, India

Corresponding author Email : amaiti@cvrce.edu.in

Abstract: In the present study, first ever attempt has been made to develop physically functionalized multiwalled carbon nanotube (MWCNT) reinforced Al-11.5Si alloy nanocomposites synthesized via novel consolidation technique viz spark plasma sintering (SPS). There is a recent trend in employing carbon nanotubes (CNTs), an allotrope of carbon, as reinforcement for high strength structural metallic composite materials, as these cylindrical nano-fibers poses extremely unique mechanical properties such as very high elastic modulus (~ 300 GPa to 1.5 TPa) as well as tensile strength (~150 GPa). However, it has remained as an ever-existing problem to achieve a porosity-free nanocrystalline matrix with homogenously dispersed CNTs, owing to the very high coagulation tendency of CNTs. The gas-atomized, spherical Al-11.5Si alloy powders (1-8 μm) were subjected to high energy ball milling for the purpose of achieving nanocrystallinity in the powders. The improvement in MWCNT dispersion was effort by treating the MWCNTs with a physical surfactant, sodium dodecyl sulfate (SDS). The nano-grained ball-milled Al-Si powders with varying MWCNT content (0.5 and 1 wt%) were consolidated via spark plasma sintering in order to retain the nano-sized grains in the Al-Si matrix, attributed to the faster and highly effective sintering kinetics of the sintering techniques. FESEM study shows problem of MWCNT agglomeration persists by addition of non-SDS treated as pristine MWCNT in the composite. After treated with SDS, MWCNTs are well separated out from each other and as a result of that good morphological and mechanical property such as high hardness value obtained after analysis. Detailed TEM study of the 0.5wt% MWCNT reinforced SPS nanocomposite revealed that the distribution of CNTs in the matrix. Mechanical analysis study of the nanocomposite attributes higher hardness in case of SDS treated CNT reinforced nanocomposite owing to less agglomeration problem of the CNT in the matrix. Nano-tribological data attributed variation of surface roughness after consolidated by SPS.

1. Introduction

In automobile and aerospace industries different components are made of various grades of Aluminum based alloys owing to its lighter in weight and easy deformability. However, some of the application areas where strength of the material is prime concern, there Al alloys are failed to serve satisfactory result due to its very low load bearing capacity. To overcome this issue, various types of hard reinforcing elements such as SiC, alumina etc. is added with different proportion to the Al matrix in order to increase the strength of the composite. However, further study reveals that not only proportion but final size as well as distribution of the reinforcing elements play a vital role in the final strength of the composites. By reducing the size of the reinforcing elements, dislocation movements can block in nanometer level which ultimately increases the strength of the composite. Interestingly, density of the consolidated composite does not alter even after incorporating reinforcement elements in different proportion, sometimes it lower the density of the composite but obtained superior strength after addition. Nanometer size hard particles such as Nano-SiC, Nano-Alumina are being used as reinforcement for synthesizing nanocomposite. But, among all the hard nanoparticles, Carbon nanotubes (CNT) are chosen for the present research work as reinforcement elements due its



tremendous high tensile strength and elastic module [1-5]. However, major problem with CNTs is that it has very high tendency of agglomeration owing to their very high surface energy [6]. Agglomerated CNTs are undesirable as they are hindered the proper consolidation during sintering by forming a barrier between two individual particle. As a result of that catastrophic failure of the material would be occurred from that agglomerated region of CNTs. Therefore, separation of CNTs is vital for obtaining a good consolidate microstructure as well as high strength. To meet the objective, in the present research work, Multi-walled CNTs are added in two different proportions (0.5 & 1 wt %) after treating with surfactant SDS. For comparison purpose, another two different samples with same proportion of non SDS treated CNTs (i.e. .0.5 and 1 wt%) are added with the Al alloys powder and finally consolidated via spark plasma sintering (SPS) process. Technique of SPS process is novel as here simultaneously thermal as well as mechanical energy are applied for consolidation. Due to this very good consolidated samples could be obtained after sintering. Sintered nanocomposites were analyzed through optical, SEM and finally TEM in order to study the distribution of MWCNT before and after SDS treatment. Mechanical property evaluations of the sintered samples are carried out to obtain the effect of distributed MWCNT on the composite.

2. Materials and methods

In the Present research work following are the materials obtained externally in as received condition for synthesizing nano-composite :

- (i) Gas atomized, spherical Al-Si (11.5 wt %) alloy powder with 2-10 μm diameter which was used as matrix material in the nanocomposite.
- (ii) 0.5 wt% & 1 wt% multi-walled carbon nanotubes (MWCNTs) of purity >95%, 40–70 nm in diameter and 0.2–0.5 μm in length were used to reinforce the Al-Si alloy matrix.

At first, Al-Si alloy powders of desired quantity was measured separately and then poured inside a planetary ball mill (Retsch GmbH, Germany, Model: PM 100) in order to convert micron size powder to nano-meter size Al powder with continuous milling operation. Ball milling was continued for 8 h using stainless steel balls of 10 mm diameter with ball to powder ratio of 20:1 at a milling speed of 180 rpm. In order to separate out the individual MWCNT from the agglomerated as received MWCNT bundle in powder form; they were dissolved in acetone with SDS by maintaining 2:3 weight ratios. Solutions of SDS and MWCNTs mixtures were then ultra-sonicated for 20 min. Sonicated mixture was poured drop-wise via a burette in a beaker which already contained ball milled Al-Si powders dissolved in acetone, which was being continuously ultra-sonicated. Solutions were allowed for drying in air & eventually dry powder mixture (ball milled Al-Si powder and MWCNT) was consolidated via SPS (Fuji Electronic Industrial Co. Ltd, Japan, Model: SPS 515S) in a 20 mm diameter graphite die. Sintering was performed at 500°C for 3 min with heating rate of 163°C/min at an applied uniaxial loading of 50 MPa.

3. Result and Discussion

3.1 MWCNT distribution in the nanocomposites: FEG-SEM result

Figure 1 shows the FEG-SEM micrographs of SPS nanocomposite with 0.5wt% and 1wt% *SDS treated and non SDS treated* MWCNT. Dispersed CNTs are observed in the Figure1a and c where it is cleared that after SDS treatment CNTs are separated out. Surfactant has ability to disperse the CNT by covering the surface as a form of micelle and subsequently lowering the surface energy of CNT [7]. Interestingly, In Figure 1b and d, it has been observed that MWCNTs were agglomerated because

CNTs are added without treated with surfactant. Besides, it is clearly observed from Figure 1b and d is that agglomerated CNTs restrict the bonding between the particles by forming a barrier.

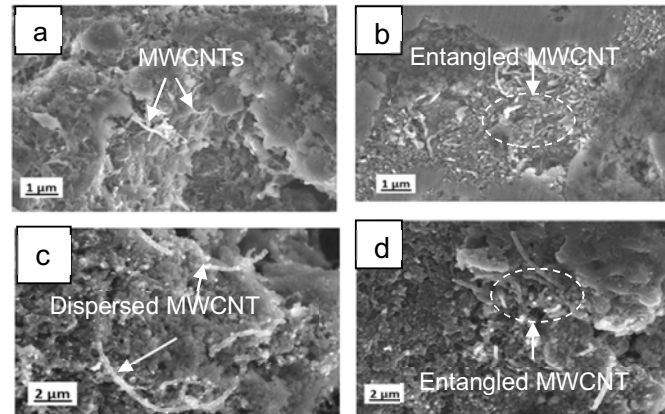


Figure 1: Distribution of (a) SDS treated 0.5 wt% MWCNT (b) non-SDS treated 0.5 wt% (c) SDS treated 1 wt% (d) non-SDS treated 1 wt% MWCNT reinforced SPS nanocomposite

3.2 The distribution of MWCNT in the Al-CNT Nanocomposite: TEM study

0.5wt%MWCNT reinforced SPS NC are shown in the HRTEM micrographs (Figure 2). MWCNTs agglomerated in bundle form and squeezed in between the grains. These tightly compacted MWCNTs would restrict the grain growth (Figure 2a). High magnification HRTEM micrograph of MWCNTs bundle show that MWCNTs are twisted and overlapped due to surrounding pressure of the grains.

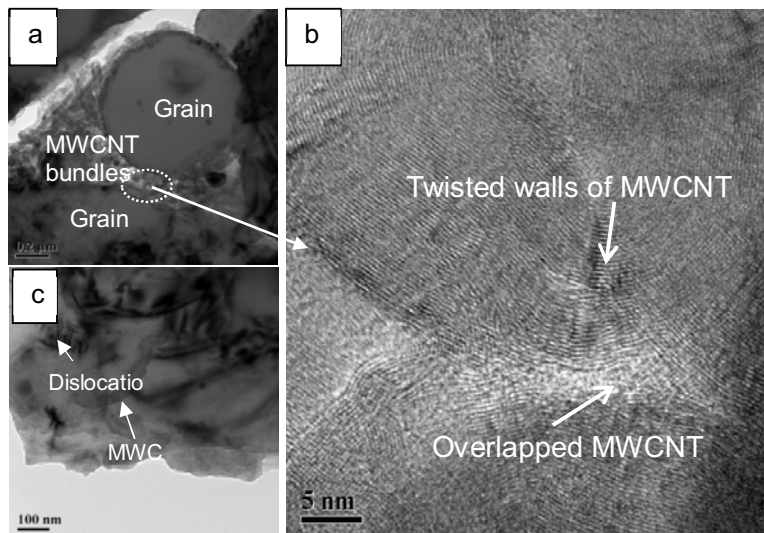


Figure 2: HRTEM micrographs showing (a) MWCNT bundles, (b) twisted and overlapped MWCNTs in between Al-Si grains, and (c) MWCNT – dislocation interaction in SPS nanocomposites with 0.5 wt% MWCNT

MWCNT-dislocation interaction can be observed in Figure 3, which indicates strengthening of the nanocomposite by virtue of dislocation movement restriction by MWCNT nano-fibers. The effect of physical functionalization on the MWCNT dispersion by treating the MWCNTs with Sodium dodecyl

sulfate (SDS) is shown in Figure 3a. Although TEM images revealed only a very small portion of a sample, still it can clearly be observed that the MWCNTs were separated out very well from each other. It is worthy to mentioned here that SPS nanocomposite synthesized with pristine (not SDS treated) MWCNT exhibited agglomerated MWCNTs as shown clearly in Figure 3b.

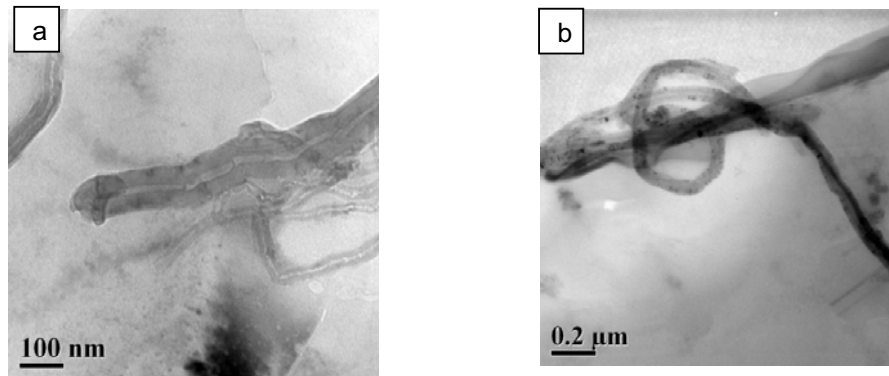


Figure 3: (a) Dispersion of SDS treated MWCNT (b) Agglomerated MWCNT

3.3 Mechanical property evaluation: hardness

Figure 4 shows the microhardness values of the SPS nanocomposite reinforced with SDS treated & non-SDS treated MWCNT. The micro-hardness increased after incorporating SDS treated MWCNT in the nanocomposite. Better distribution of the CNT in the matrix as shown in Figure 1 reason for higher consolidation and porosity attributed higher micro-hardness.

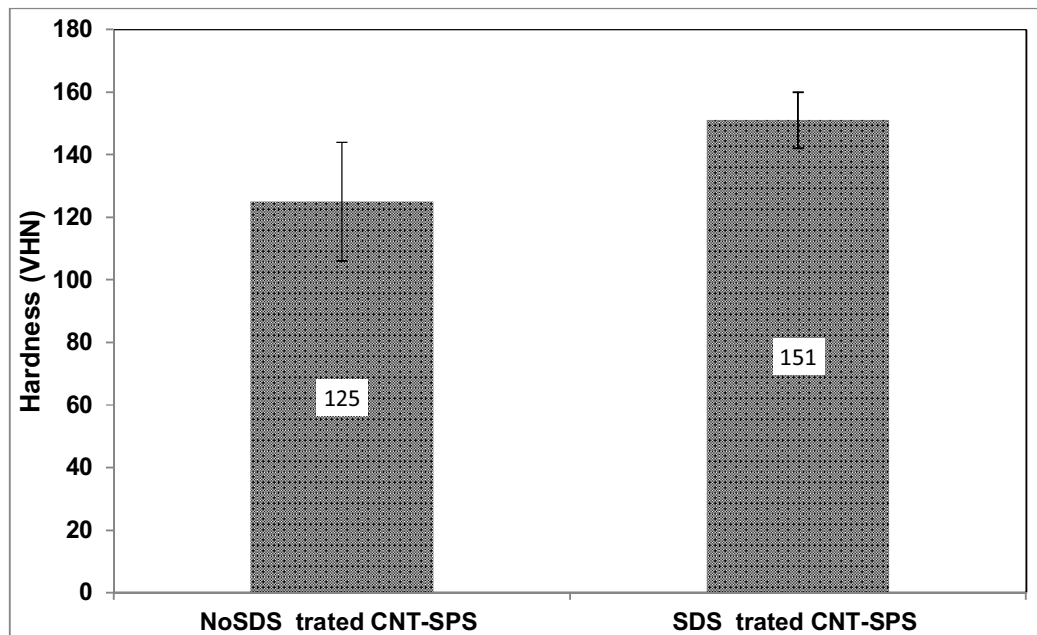


Figure 4: Micro-hardness values of the 0.5 wt% SDS & non-SDS treated MWCNT reinforced nanocomposites sintered SPS

3.4 Mechanical property evaluation: Tribological behaviour analysis by nano-scratch

Nano-scratch technique was carried out to understand the tribological behaviour of the two nanocomposites having SDS treated and non-SDS treated MWCNT as reinforcement. Figure 5 shows the variation of coefficient of friction (COF) values with a scratch distance of 4 μm at a constant load of 2000 μN . COF was measured from the ratio of the instantaneous lateral force and normal force applied during scratching. The variation of COF values along the 4 μm scratch distance in case of 0.5 wt% SDS & non SDS treated MWCNT reinforced composites are shown in Figure 5. Interestingly, variation of COF values were increased in case of non-SDS treated MWCNT reinforced nanocomposite. For example, COF of non SDS treated MWCNT reinforced composite fluctuated in between ~ 0.38 to 0.45 (Figure 5). But, variation is less after adding SDS treated MWCNT in the matrix. Large variation of COF data indicated the non-uniform consolidation in the nanocomposite & it is supported by the FESEM micrographs in Figure 1 where agglomerated CNTs were entangled to form a barrier for consolidation. Surface roughness or irregularities minimized after adding SDS treated MWCNT in the matrix as better consolidation obtained due to MWCNTs were separated out shown in Figure 1a.

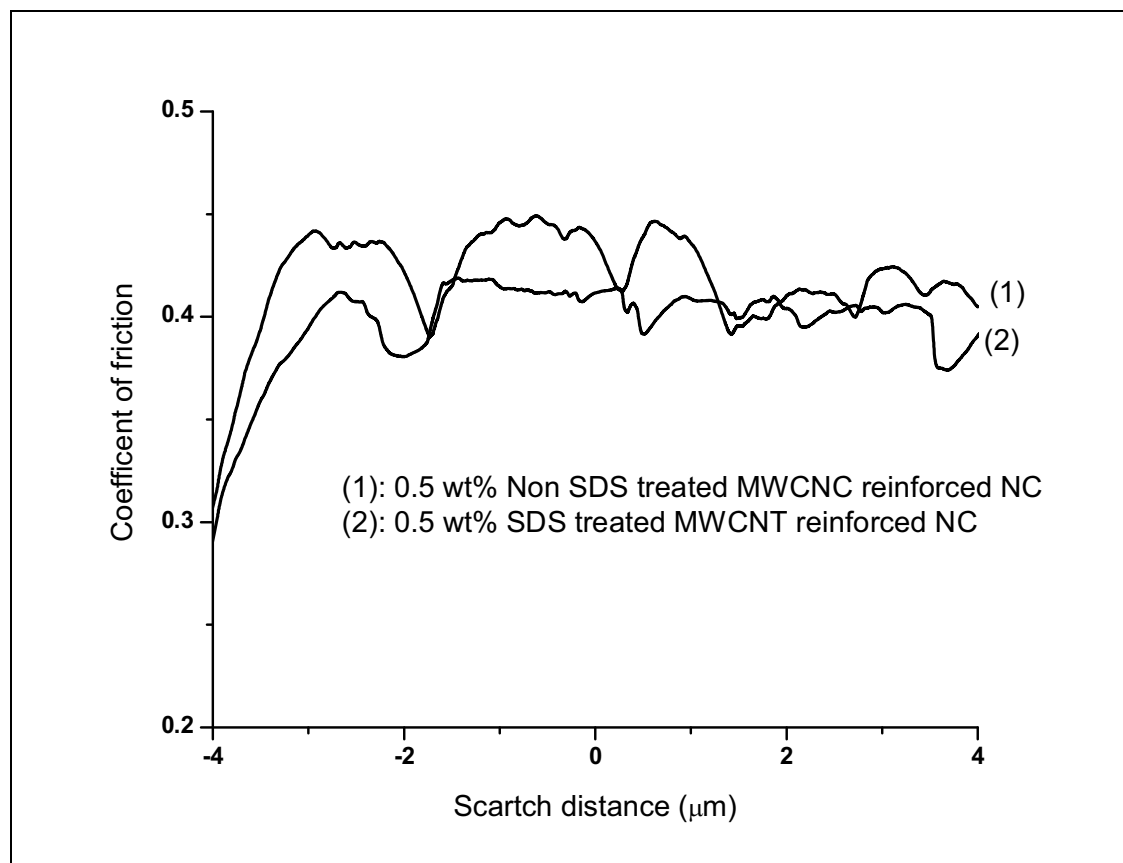


Figure 5: Variation of the coefficient of friction (COF) with scratch distance of SPS nanocomposites having 0.5wt% SDS treated & Non-SDS treated MWCNT as reinforcement

4. Conclusion

Physically functionalization of the MWCNTs improved the dispersion of these nano-fibers in the Al-Si matrix and better dispersion was achieved in the 0.5 wt% MWCNT reinforced nanocomposites, in comparison with that in 1 wt% MWCNT reinforced ones. TEM study showed that the MWCNTs retained their structural integrity without introducing major damages on the structure of the MWCNT. MWCNTs were tightly compacted along the grain boundaries and restricted the grain growth. Good adhesion between matrix and the MWCNT was observed from HRTEM study. There is further scope of densification enhancement of the nanocomposites by further improving the dispersion of MWCNTs in the matrix. Effect of other physical surfactants such as CTAB, Brij76 etc. on the dispersion of MWCNT should be studied in this context. Interestingly, SDS treated MWCNT reinforced nanocomposite elucidated better mechanical properties such as elastic modulus and micro-hardness as compared to non-treated MWCNT reinforced nanocomposite owing to the better dispersion of surfactant treated MWCNTs. Agglomerated CNTs hindered the particle to particle interaction by forming a barrier in between which eventually imparted porosity in the matrix. Increasing porosity contain in the matrix is the prime reason for degrading the mechanical properties of the nanocomposite.

5. References

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