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## Materials Today: Proceedings

journal homepage: [www.elsevier.com/locate/matpr](http://www.elsevier.com/locate/matpr)

## Fabrication of aluminium carbon nano tube silicon carbide particles based hybrid nano-composite by spark plasma sintering

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## ARTICLE INFO

## Article history:

Received 9 September 2019

Received in revised form 22 November 2019

Accepted 25 November 2019

Available online xxx

## Keywords:

Aluminium

Multiwall carbon nano tubes

Silicon carbide

Mechanical alloying

Spark plasma sintering

Microhardness

## ABSTRACT

The present research paper focuses on fabrication of, aluminum (Al) – carbon nano tubes (CNTs)-silicon carbide particles (SiC<sub>p</sub>) hybrid nano-composites using element-alloying of Al, CNTs, and SiC<sub>p</sub> powder particles into homogenous powder mixture and sintering the mixture of powders using Spark Plasma Sintering (SPS). The effect of CNTs wt. percentage (1, 3, and 5 wt%) and SiC<sub>p</sub> as a reinforcement on the mechanical properties and microstructural characteristics were analyzed. The surface topography, microstructure and element composition of the Al-CNTs-SiC<sub>p</sub> hybrid nano-composites were investigated by field-emission scanning electron microscopy (FESEM), optical microscope and energy-dispersive X-ray spectroscopy (EDAX). The Vickers hardness tester is used for measure the micro-hardness of the specimens. Microstructure and scanning electron microscopy (SEM) micrographs conformed that the sintered Al-CNTs-SiC<sub>p</sub> composite has a good reinforcement of CNTs and SiC<sub>p</sub> into the grain boundaries of Al-matrix and reduces the dislocation defects thus enhances the microstructure and strengthening metallic bond. As a result, the hybrid composite (Al-5%CNTs-10%SiC) exhibits the micro-hardness 2-fold higher than that of pure-Al.

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Selection and peer-review under responsibility of the scientific committee of the International Conference on Mechanical and Energy Technologies.

## 1. Introduction

Due to increased global competition and growing concern for the environment, automobile and aviation industries are anticipated to invent new intelligent materials having light weight along with improved hardness and strength [1]. The various researchers are working on developing new material having fiber and particle reinforcements which can produce enriched mechanical and tribological properties [2–4].

Owing to this, metal matrix composites (MMCs) has been widely used due to their improved behavior in terms of wear resistance, mechanical properties and retaining their strength at upgraded temperatures as equaled to conventional materials [5].

The aluminum metal matrix composites (AMMCs) which are reinforced with particles are widely used material in the industries because of better mechanical and wear properties as compared with conventional aluminium (Al) alloys [6,7]. The mechanical properties of particle-reinforced AMMCs are dependents to accumulation of reinforcement and have been extensively studied in previous research [8].

In the literature, silicon carbide (SiC), silica sand, boron, titanium carbide (TiC), magnesium oxide (MgO) and silicon nitride (Si<sub>3</sub>N<sub>4</sub>), titanium oxide (TiO<sub>2</sub>) and alumina (Al<sub>2</sub>O<sub>3</sub>) are most commonly used reinforcements [9,10]. In comparison, the accumulation of SiC<sub>p</sub> to Al alloys has been widely used to attain rise in strength and modulus [11]. Recently, the research community has been used carbon nano tubes (CNTs) as dispersion and reinforcement agent to aluminum matrix to enhance its properties [12]. It implies that CNT-AMMCs are estimated to play crucial part in structural materials in the coming years [13,14].

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Still, there is limited research on CNT-reinforced AMMCs have been reported to focus on the difficulties arises during dispersion of CNTs into matrix [15–17]. In general, stir casting, extrusion and forging techniques were used for fabrication of AMMCs, but for CNT-AMMCs the powder metallurgy (PM) is considered as an effective method [18]. As compared to the melting methods, processing at low temperature is a significant advantage of PM [19]. Apart from this, this method provides proper dispersal of reinforcement in matrix and produces proximate net-shape parts at low cost [20–21].

Kuzumaki is a first explorer in the fabrication of Al/CNT MMCs to achieve escalation in strength by 84 MPa using 5 vol% addition of CNT through mechanical mixing and hot extrusion [22]. In recent research,  $Al_4C_3$  was developed and stabilized on the surface of the CNT tip, which further contributed to the transfer of stress from the Al matrix to the CNTs [23]. Recently, spark plasma sintering (SPS) technique has been used for the sintering using simultaneous heating and pressing the mechanically alloyed powders. Kwon et al. fabricated Al-CNTs composite using SPS method and it has been found that the addition of 5 wt% of CNTs remarkably increased the strength about three times [24]. Very recently, Liao et al. fabricated Al-CNTs composites using SPS method and used 0–2 wt% of MWCNTs as reinforced through mixing using roller mill [25].

From the available literature, it is evident that there is no research available on fabrication of hybrid Al-CNTs-SiCp composite using spark plasma sintering method. In the current research work, the CNTs (1, 3, and 5 wt%) and SiCp (10 wt%) has been used as reinforcements in the aluminium matrix. The developed hybrid composites are catheterized to analyze microstructure, elemental composition, and mechanical properties.

## 2. Materials and methods

Aluminum powder (Purity 99.9%, average particle size 5  $\mu\text{m}$ ) as matrix, multi-walled carbon nanotubes (MWCNTs) with Purity 99.5%, diameter 20 nm, length 15–50  $\mu\text{m}$  and silicon carbide particles (SiCp) with Purity 99.9%, average particle size 25  $\mu\text{m}$  were used to fabricate hybrid Al-CNTs-SiCp composite. Fig. 1 represent the SEM micrograph of powder particles and TEM micrograph of MWCNTs. The Al powder particles are irregular in shape and fine powder particles and SiC powder particle had also irregular shape.

The powder composition was adjusted (1, 3, and 5 wt% CNT) and 10 wt% SiCp and rest is Al powder. The mechanical solid state alloying of elemental powders of Al, CNTs, and SiCp was carried out in a high energy planetary ball mill (Fritsch, Pulverisette-7) of stainless steel balls spinning at 250 rpm for 8–10 h. The ball to powder ration was kept 5:1. A predetermined mass of the powders was calculated according to the die of sintering (30 mm dia. and 4 mm thick) and weigh using high precision weighing balance machine. In argon atmosphere, the mixed powder was preheated at 200  $^{\circ}\text{C}$  for 2 h to dissipate the humidity. Then SPS process (by using SPS-5000 machine; model: Dr. Sinter SPS-625, Japan) was executed. The SPS was conducted under vacuum and at various sintering temperatures and pressure conditions at a heating level of 50 K/min (for a holding time of 5 min). For sintering, a graphite die was used and the solid 20 mm diameter and 4 mm thickness compacts were synthesized [24,25]. A circular compact was prepared with a diameter 30 mm and a thickness of 4 mm. Fig. 2 shows the experimental setup of spark plasma sintering (SPS) machine and consolidated sample. Samples for Scanning Electron Microscopy (SEM), Scanning

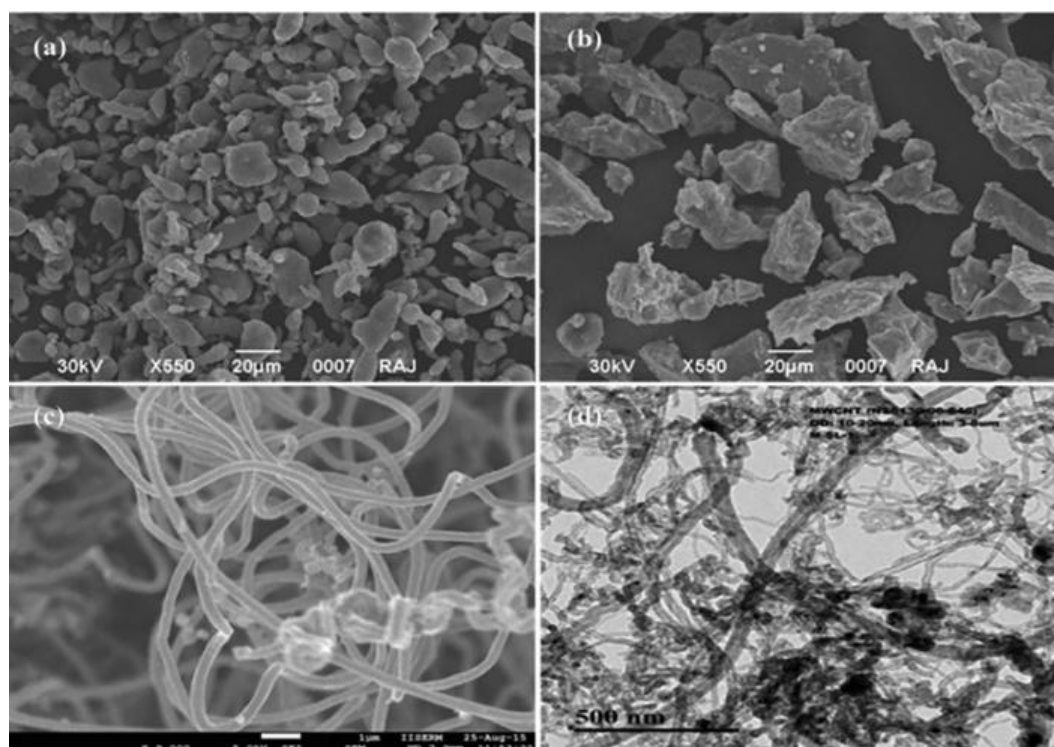
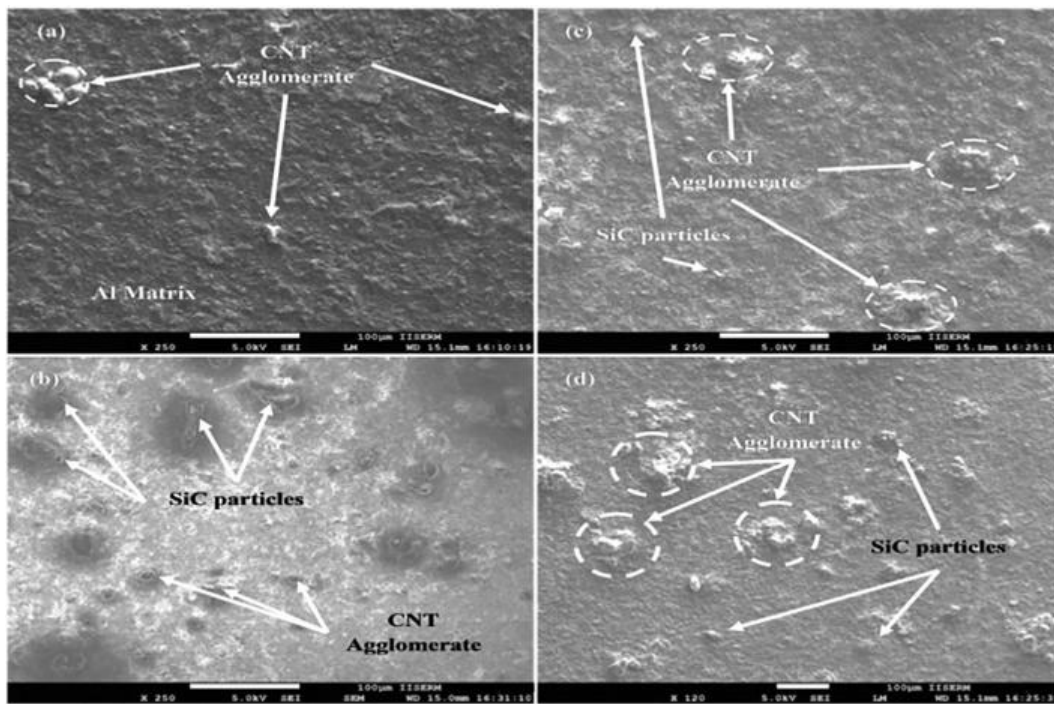


Fig. 1. (a–b) SEM micrograph of Aluminum and Silicon carbide; (c–d) SEM and TEM micrograph of Multi-wall carbon nano tube (MWCNTs).



**Fig. 2.** (a) Spark Plasma Sintering machine (S-5000), (b) red hot sample under vacuum during the process, (a) and as-consolidated sample. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)



**Fig. 3.** SEM micrograph of (a) Al-5% CNTs, (b) Al-1% CNTs-10% SiCp, (c) Al-3% CNTs 10% SiCp, (d) Al-5% CNTs-10% SiCp composites.

Electron Microscopy-Energy Dispersive (EDS) and X-ray diffraction (XRD) examinations were cut, finished, and then polished up to  $R_a \sim 0.5 \mu\text{m}$  [26–27]. Vickers hardness tester (HMV-G21ST, SHIMADZU, Japan) measured the micro-hardness of the as-synthesized composite according to the technique adopted in previous research [28,29].

### 3. Results and discussions

#### 3.1. Microstructural characterization

Fig. 3(a–d) represent the SEM micrograph of the Al-5%CNTs, Al-1%CNTs-10%SiC, Al-3%CNTs-10%SiC, and Al-5%CNTs-10%SiC, respectively. The SEM micrograph clear seen that CNTs and SiC particles were homogeneously dispersed in Al-matrix. The CNTs corresponds to white dots/area and presents in the form of agglomerates, whereas SiC corresponds to black patches. As, the CNTs concentration increases the distribution of CNTs agglomerates increases into the matrix. Fig. 4(a–c) shows the microstructure of the transverse cross sections of the sintered compact of Al-CNTs composite. Microstructure showed that CNTs are reinforced into grain interface of the Al matrix and agglomerates some places. In the previous research, it was seen that the CNTs were agglomerates more than 93% and appears similar pattern as claimed in this research work [30]. The CNTs corresponds to white dots/area. Fig. 4(b–d) shows the microstructure of the transverse cross sections of the sintered condensed hybrid Al-CNTs-SiCp composite. Microstructure showed that SiC particles along with CNTs are reinforced into grain interface of the Al matrix. The SiC particles corresponds the black irregular shapes and found uniformly distributed in the matrix. Balani et al. [31] reported the absence of any interface between SiC<sub>p</sub> and Al-Si matrix. Kwon et al. [32] confirms the formation of grain growth and presence of CNTs in the interface of grain boundary after spark plasma sintering. This is because the rapid heating and pressure applied by SPS process can help to overpower grain growth [25].

#### 3.2. Elemental composition

Fig. 5(a–b) represent the EDS spectrum of the Al-5%CNTs composite. It can be clearly seen that elements Al, C, O are present in the EDS spectrum of the Al-5%CNTs composite. The weight percentage of Al, C, and O is 94.82%, 4.76%, and 0.42%, respectively. The presence of Al, C, and O elements possibly form  $\text{Al}_2\text{O}_3$ , AlC, which is a particularly important factor to emphasize in the increase of micro-hardness. Fig. 5(c–d) shows the EDS spectrum of the Al-5%CNTs-10% SiC composite. The weight percentage of Al, Si, C, and O is 89.89%, 3.87%, 5.67%, and 0.57%, respectively. The element O and C were present along with Al and Si because due heating the carbide and oxides will form. The presence of Al, Si along with C and O elements possibly form SiC,  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ -SiC,  $\text{Al}_2\text{O}_3$ , AlC, which is a particularly important factor to emphasize in the increase of micro-hardness of hybrid composite.

#### 3.3. Hardness values

Fig. 6 shows the hardness of Al-5%CNTs, Al-1%CNTs-10%SiC, Al-3%CNTs-10%SiC, and Al-5%CNTs-10%SiC composites. The micro-hardness of the Al with any reinforcement is 58 HV [25]. As predicted, with the addition of reinforcement (SiC and CNTs), Al hardness increases. As compared to unreinforced Al, the Al-5% CNTs composite shows 68.96%, as reported in another study [25,32], while in the case of Al-1%CNTs-10%SiC, Al-3%CNTs-10% SiC, and Al-5%CNTs-10%SiC composite the hardness is increased 148%, 160%, and 172%, respectively. The improvement in the micro-hardness for Al-5%CNTs composite is due to the fact that CNTs reinforced into the interface of grain boundary and creates a barrier to dislocation thus boost the strain hardening in the plastic deformation [25,33]. In the case of hybrid composites (Al-CNTs-SiC) the micro-hardness increased due to the strengthening effects of both reinforcements SiCp and CNTs [34]. These results are in accordance with the other studies]. The micro-hardness increased in the case of hybrid composites (Al-CNTs SiC) due to the reinforc-

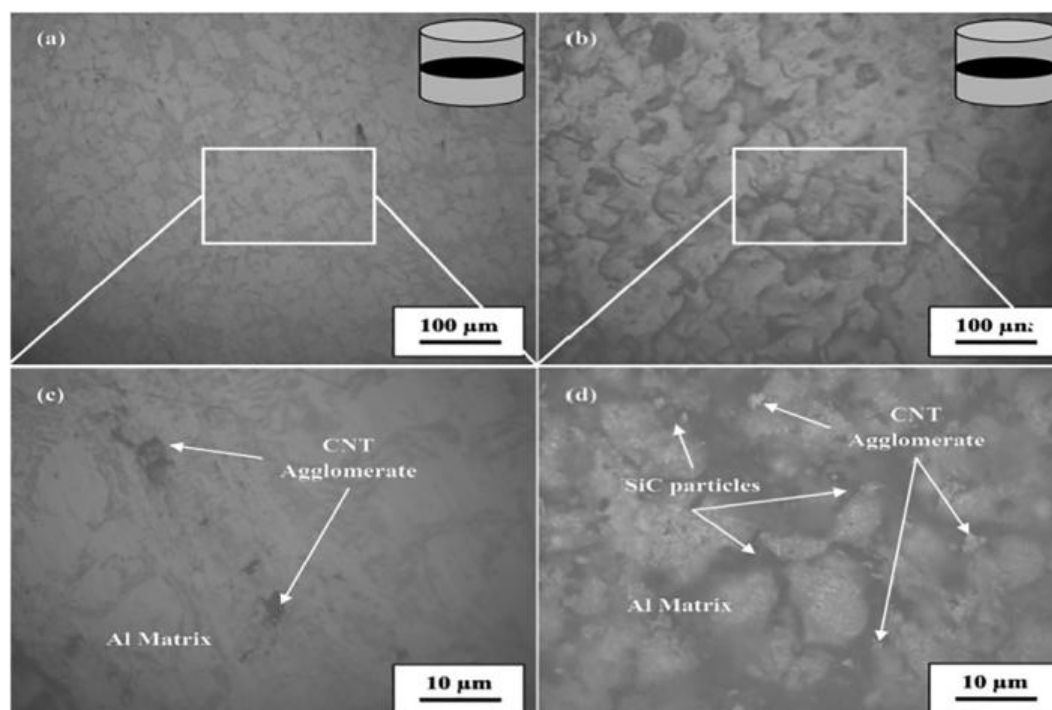


Fig. 4. Optical micrograph of (a–c) Al-5% CNTs; (b–d) Al-5% CNTs-10% SiCp composites.

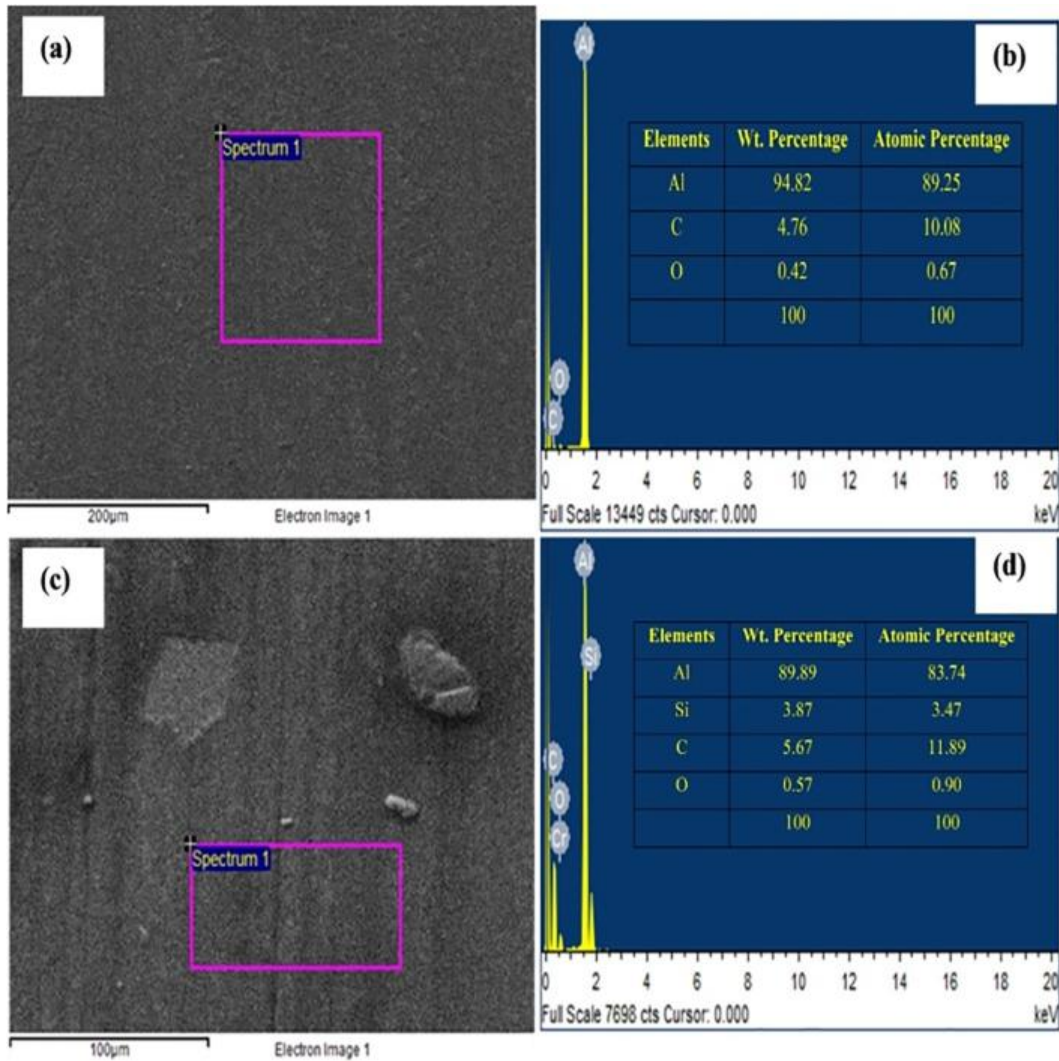


Fig. 5. EDS spectrum of (a-c) Al-5% CNTs; (b-d) Al-5% CNTs-10% SiCp composites.

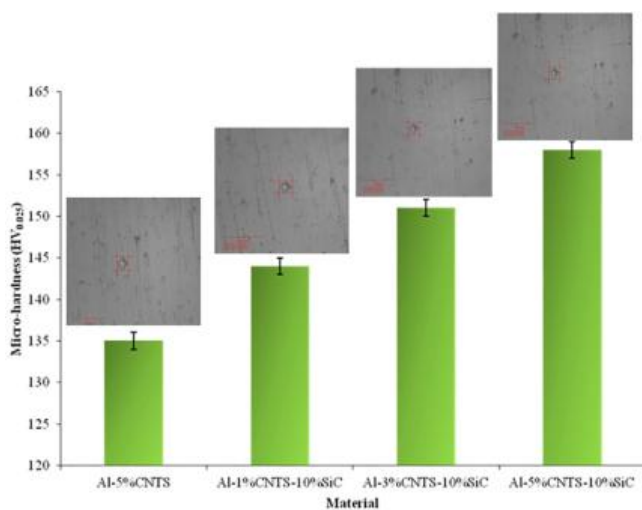


Fig. 6. Micro-hardness values of Al-5%CNTs, Al-1%CNTs-10%SiC, Al-3%CNTs-10%SiC, and Al-5%CNTs-10%SiC composites.

ing effects of both SiCp and CNT reinforcements [34]. These findings are consistent with the other studies [25,32,34].

#### 4. Conclusions

We draw the following conclusions from exploration on the mechanical alloying of Al-CNTs-SiCp powders and consolidation by SPS:

- (1) The mechanical alloying of Al-CNTs-SiCp powders has been successfully achieved and reduces the problems of CNTs dispersion.
- (2) Al-5%CNTs, Al-1%CNTs-10%SiC, Al-3%CNTs-10%SiC, and Al-5%CNTs-10%SiC composites were successfully fabricated by SPS process.
- (3) The reinforcement CNTs and SiCp are found present uniformly distributed and reinforced into grain interface boundaries of Al matrix. The CNTs presents in the form of agglomerates and if CNTs concentration increases the distribution of CNTs agglomerates increases into the matrix.

(4) The micro-hardness of the Al-based composite increased greatly as compared to unreinforced Al. As compared to unreinforced Al, the hardness of Al-5%CNTs, Al-1%CNTs-10%SiC, Al-3%CNTs-10%SiC, and Al-5%CNTs-10%SiC composite is increased by 68.96%, 148%, 160%, and 172%, respectively.

On the basis of these results, it is predicted that the wear resistance of Al-5%CNTs-10%SiC may be enhanced. In the future research work, the wear and corrosion performance of the as-prepared samples is studied.

#### CRediT authorship contribution statement

**Chander Prakash:** Project administration, Conceptualization, Methodology, Investigation. **Sunpreet Singh:** Project administration, Conceptualization, Methodology, Investigation. **Shubham Sharma:** Project administration, Methodology, Validation. **Harish Garg:** Resources, Visualization. **Jujhar Singh:** Data curation and Formal analysis. **Harish Kumar:** Resources, Visualization. **Gursharan Singh:** Visualization.

#### Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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