

# ELECTROSPUN NANOFIBERS REINFORCED ALUMINIUM MATRIX COMPOSITES, A TRIAL TO IMPROVE THE MECHANICAL PROPERTIES

Hany S. Abdo <sup>1,2,3,\*</sup>, Khalil Abdelrazek Khalil <sup>2,4,\*</sup>, Magdy M. El-Rayes <sup>5</sup>,  
Wagih W. Marzouk <sup>3</sup>, A.M. Hashem <sup>6</sup> and G.T. Abdel-Jaber <sup>6</sup>

<sup>1</sup>Center of Excellence for Research in Engineering Materials (CEREM),  
Deanship of scientific research, King Saud University, P.O. Box 800,  
Al-Riyadh 11421, Saudi Arabia.

<sup>2</sup>Mechanical Design and Materials Department, Faculty of Energy  
Engineering, Aswan University, Aswan 81521, Egypt.

<sup>3</sup>Production Engineering and Design Department, Faculty of Engineering,  
Minia Universities, Minia 61111, Egypt.

<sup>4</sup>Department of Mechanical Engineering, College of Engineering,  
University of Sharjah, P.O. Box 27272, Sharjah, UAE

<sup>5</sup>Mechanical Engineering Department, College of Engineering, King Saud  
University, P O Box 800, Riyadh 11421, Saudi Arabia.

<sup>6</sup>Department of Engineering Materials and Mechanical Design, Faculty of  
Engineering, South Valley of University, Qena 83523, Egypt.

## ***ABSTRACT***

*A comparison between TiO<sub>2</sub> nanofibers and carbon nanofibers as fibers reinforced metal matrix composites with respect to mechanical properties improvements have been made in this paper. Al and Mg have been chosen as metal matrices. The used carbon and ceramic nanofibers (Titanium Oxide) were successfully synthesized using electrospinning technique. Various weight percentage of calcined electrospun TiO<sub>2</sub> and carbon nanofibers (1, 3, 5 and 10%) were mixed with metal matrix and fabricated by route of powder metallurgy using High Frequency Induction heat Sintering (HFIHS). Mechanical properties of the sintered composites have been investigated. The manufactured pellets were tested for compression test, hardness and microstructures by the field emission scanning electron microscopes (FESEM), which reveals the homogeneous distribution of nanofibers in the Al/Mg matrices. In addition, energy-dispersive X-ray spectroscopy (EDS) was employed to obtain the chemical analysis of each composite. The result shows that, the ultimate compressive strength increased to 415 MPa at 5% TiO<sub>2</sub>, which is 13.5% more than the pure Al. The hardness increased up to 64% in case of using the ceramic nanofibers as reinforcement. While using CNFs as reinforcement to the Al matrix deteriorates the mechanical properties.*

## ***KEYWORDS:***

*Aluminium Matrix; TiO<sub>2</sub> Nanofibers; Carbon Nanofibers; Mechanical Properties*

## **1. INTRODUCTION**

Metal matrix composites (MMCs) have been used in engineering applications in different ways because of their mechanical and physical properties [1-5]. In the previous ten years, aluminum matrix composites (AMCs) are used in hi-tech purposeful and structural applications such as automotive, aerospace and defence also as in sports and light industries [6, 7]. AMCs indicate to the category of light weight excessive efficiency Al central systems. Reinforcement in AMCs would be in the type of platelets, tubes, particulates, or fibres, in volume fractions starting from a few percentage up to 65% [7]. The mechanical properties of AMCs can be custom fitted to the requests of various modern applications by playing with the reinforcement combinations and the preparing technique.

In traditional MMCs, ceramics, like SiC or Al<sub>2</sub>O<sub>3</sub>, in the type of fibers, flakes or particulates are the most commonly used as reinforcements [6–9]. Nonetheless, the interference between the metallic matrix and the ceramic reinforcements are generally not perfect, which produce incredibly porous composites with less mechanical properties and higher corrosion sensibility [10]. As a way to resolve this drawback, metal-glasses had been proposed as a novel form of reinforcement in metal matrices composites [11–17]. The mechanical properties of aluminium composites are efficiently improved in case of using nano-fibers as a reinforcement. Accordingly, the high surface to volume ratio of nanofibers effectively enhance the strength and stiffness of Al composites compared to micro-fibers due to the good interface between the ceramic reinforcement (nano-fibers) and the metal matrix.

This work is mainly concerned with the evaluation of light-metal nanocomposites strength. The main objective of the current work is to studying the effect of adding two kind of nanofibers, TiO<sub>2</sub> and CNF [in various contents ranging from 1:10 wt.%] on the mechanical properties of light metal matrix, namely; Aluminium. In order to understand the trends observed in the properties of nanofiber reinforced, a comparison between both kinds of nanofibers as a reinforcement for metal matrix composites with respect to mechanical properties improvements have been performed.

Another objective from this study is to introduce a new nanocomposite material by using nanofiber as reinforcement and to improving the mechanical properties of the light metal matrix.

The study also focuses on the relationships between effective properties and properties of constituents (metal matrix and reinforcement), weight fraction of components, shape and arrangement of reinforcement, and the interaction between matrix and reinforcement.

## **2. METHODOLOGY AND EXPERIMENTAL PROCEDURES**

### **2.1. METHODOLOGY**

To achieve our objective, the methodologies are as follows:

- Electrospinning of ceramic nanofibers,
- Calcinations of obtained nanofibers, to convert it into ceramic/carbon nanofibers,
- Use the ceramic/carbon nanofibers as reinforcement for Aluminium matrix,
- Sintering that nanocomposites by high-frequency induction heat sintering furnace HFIHS with different compositions and temperatures

- Characterizing the sintered pallets (SEM, XRD, ...etc),
- Studying the mechanical properties for the reinforced metal matrix nanocomposites.

## 2.2. MATERIALS

In this study, Titanium isopropoxide (C<sub>12</sub> H<sub>28</sub> O<sub>4</sub> Ti), PVP (M<sub>w</sub> = 1,300,000), Dimethylformamide (DMF) and Polyacrylonitrile (PAN) were obtained from Sigma– Aldrich, USA, and Ethanol, Acetic Acid, Aluminium Fine Powder was obtained from different sources as presented in table 1. All these chemicals and solvents were used as received without further purification.

Table 1: Compounds, Formula, purity and sources used for all experimental work

Materials or Chemicals	Linear Formula	Mw (g/mol)	Purity (%)	Source
Titanium isopropoxide	C <sub>12</sub> H <sub>28</sub> O <sub>4</sub> Ti	284.22	99.999	Sigma-Aldrich
Polyacrylonitrile (PAN)	C <sub>3</sub> H <sub>3</sub> N	150,000	---	Sigma-Aldrich
Ethanol Absolute	H <sub>3</sub> CCH <sub>2</sub> OH	46.07	96	AVONCHEM
Dimethylformamide (DMF)	C <sub>3</sub> H <sub>7</sub> NO	73.09	99.8	Sigma-Aldrich
Acetic Acid Glacial	CH <sub>3</sub> COOH	1.05	99.7	Qualikems, UK
Polyvinylpyrrolidone (PVP)	(C <sub>6</sub> H <sub>9</sub> NO) <sub>n</sub>	1,300,000	95	Sigma-Aldrich
Aluminum Fine Powder	Al	26.98	99	Merck, Germany

## 2.3. PREPARATION OF THE ELECTROSPUN NANOFIBER

### 2.3.1. TiO<sub>2</sub> / PVP Solution

In this step, titanium dioxide was prepared via sol–gel by adding 4.5 gm of Ti(IV)-isopropoxide (C<sub>12</sub> H<sub>28</sub> O<sub>4</sub> Ti), and 9 ml of acetic acid as a solution to gelation containing 30 gm of ethanol and 1.5 gm of polyvinylpyrrolidone (PVP, Aldrich, M<sub>w</sub> 1,300,000). The mixture was vigorously stirred at room temperature for two hours to obtain 45 grams of a homogeneous viscous solution.

### 2.3.2. Pan / Dmf Solution

Polyacrylonitrile (PAN) (M<sub>w</sub> 150,000) 7 wt% solution was prepared in N,N-dimethylformamide (DMF) with vigorous stirring for at least 2 hours to obtain a transparent polymer solution.

### 2.3.3. Electrospinning Device

A schematic diagram of the used electrospinning device used for producing polymer nanofibers is shown in Fig. (1). In a typical electrospinning setup, a high-voltage source is connected to a

metallic needle, which is attached to a solution reservoir (syringe). The needle has a relatively small orifice that concentrates the electric charge density on a small pendant drop of solution.

There are basically three components to fulfil the process: a high voltage supplier, a syringe with needle of small diameter, and a collecting drum. In the electrospinning process a high voltage (20 kV) is used to create an electrically charged jet of polymer solution. Before reaching the collecting drum, the solution jet evaporates or solidifies, and is collected as an interconnected mat of small fibers.

One electrode is placed into the spinning solution/needle and the other attached to the drum collector. In most cases, the collector is simply grounded, as indicated in Fig. (1). The electric field affects the end of the needle that contains the solution fluid held by its surface tension

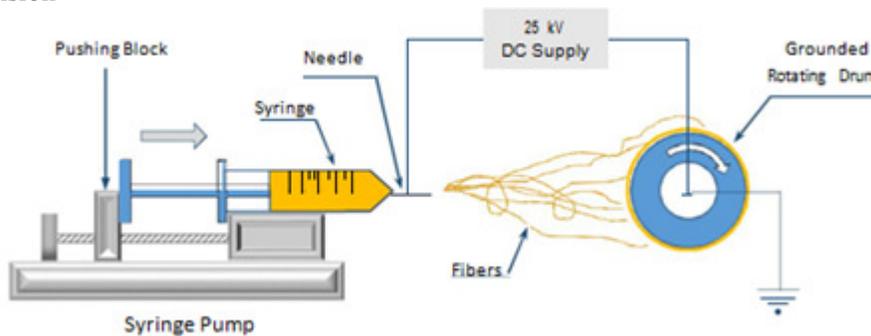


Figure 1: Schematic Layout For The Electrospinning Process

#### 2.3.4. Calcination process

Calcination is the process in which the nanofiber is burned in air or inert atmosphere to produce CNF or metal-oxide nanofiber respectively. Usually it is performed at temperatures above the thermal decomposition temperature and below the melting point. In this study, a tube furnace was used to perform calcination (CARBOLITE Type 3216CC up to 1600 °C).

TiO<sub>2</sub> nanofiber was calcined at 600 °C on air for 3 hrs. with a heating rate of 10 °C/min, while PAN nanofiber mats were stabilized on air atmosphere at 270 °C for 2 hrs (with a heating rate of 2 °C/min) followed by carbonization at 1000 °C for 1 hr (with a heating rate of 4 °C/min) on nitrogen atmosphere to produce carbon nanofiber (CNF).

#### 2.4. PREPARATION OF THE COMPOSITE POWDER

In order to mix the produced ceramic and carbon nanofibers with an Aluminium matrix, we used the high-energy ball milling technique (HEBM). A Desktop 220V High Energy Vibratory Ball Mill with 80ml Jar from Across International Company, USA, was used in this study.

Aluminium Fine Powder (Al) was first de-agglomerated in the HEBM for 1 hour, then TiO<sub>2</sub> Ceramic Nanofibers and Carbon Nanofibers were added separately to the slurry for a certain time as mentioned in table 2 just for mixing. The following table summarizes the different samples and concentrations used:

Base matrix	Reinforcement	Reinforcement percentage %	Mixing time
Aluminum Fine Powder (Al)	TiO <sub>2</sub> Ceramic Nanofibers	1, 3, 5 and 10	15 min
	Carbon Nanofibers		12 min

Table2:Samples Composition and milling time

## 2.5. HIGH FREQUENCY INDUCTION HEAT SINTERING PROCEDURE

The composite powders were densified by using HFIHS process (HF, ELTek CO., Korea). 5 g of powder was putted into a graphite mold of 10 mm diameter. The sintering process was done under 40 MPa pressure at temperature of 580°C, for 5 min. The compaction and sintering processes were done simultaneously at vacuum level of  $2 \times 10^{-3}$  Torr to prevent the oxidation of the surface of the composites. The sintered specimens were finally kept to cool down to reach room temperature. The heating rate from room temperature to 580°C was 250°C/min with a holding time of 5 min in order to prevent the grain growth of the composite particles.

## 2.6. CHARACTERIZATION

The density of the samples was measured using Archimedes' principle using (DAHOMETER, DH 300L). Phase composition of the composites was analyzed by X-ray diffraction system (D-8 Discover, Bruker, Germany) and using CuK $\alpha$  monochromatic radiation. The microstructures and chemical composition of polished and thermal etched surfaces were characterized by field-emission scanning electron microscope (FE-SEM) (JEOL; JSM7600F) equipped with energy dispersive X-ray spectroscopy (EDS). The hardness test was carried out using a Vickers micro-hardness tester (Buehler-micro-met 5114, Akashi Corporation, Japan) under an applied load of 500g with an indentation time for 10 s. The density and hardness reported in this work are the average values of six testing result

## 2.7.COMPRESSION TEST

In accordance with ASTM:E9-89a, the samples were determined at ambient temperature, using INSTRON testing machine with a strain rate of  $8 \times 10^{-5} \text{ s}^{-1}$ . The test specimens of 10 mm diameter and length to diameter ratio  $L/d \sim 1$  were used. For each composition, 3 samples were tested to ensure repeatable values.

## 3.RESULTS AND DISCUSSION

### 3.1.SYNTHESIS AND MICROSTRUCTURE

Fig. 2 (b) shows the SEM images of the TiO<sub>2</sub> nanofibers (5wt% Titanium Isopropoxide) after calcination process at 600 °C for three hours in air with different magnifications. It can be seen from this figure that nanofibers was partly twisted and arcuate. The diameters of nanofibers after calcination almost the same as before, no significant difference as shown in Fig.2(a)

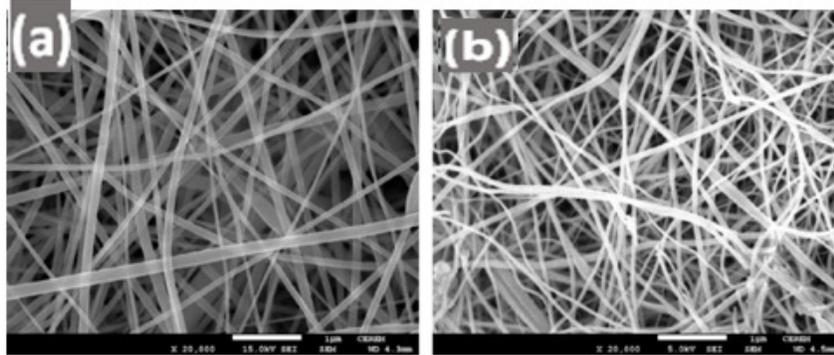


Figure 2: SEM images for TiO<sub>2</sub> nanofiber mat (a) before and (b) after calcination

As shown in Fig. 3 (a), the approximate diameters of PAN nanofibers were 400 nm. It looks homogeneous and uniform without beads or beaded nanofibers. The only difference in morphologies between the fiber bundles before and after calcination is the fiber diameter, which is reduced by a few nanometers as shown in Fig. 3 (b).

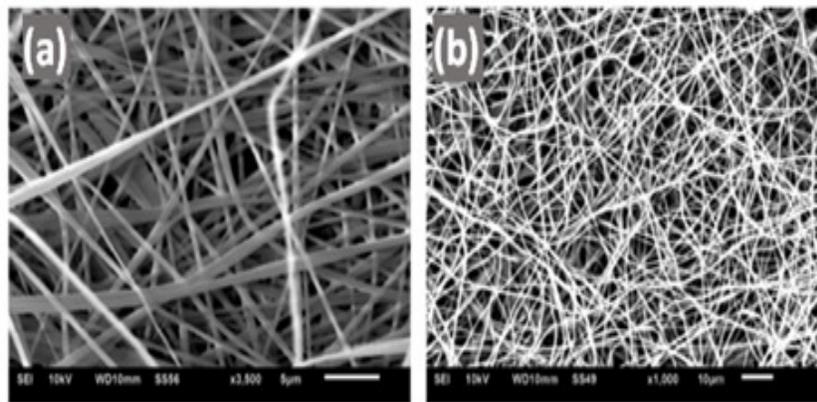


Figure 3: SEM Micrographs for PAN nano fiber (a) before and (b) CNF calcination

Fabrication of Al/TiO<sub>2</sub> and Al/CNF nanocomposites were successfully completed by using powder metallurgy technique followed by high frequency induction heat sintering process.

Fig. 4 showed TiO<sub>2</sub> nanofibers and CNFs (seen as light gray phase) were longitudinal in shape and homogeneously distributed in Al powder. It also shows how the mixing and embedding of nanofibers into powder to form composite.

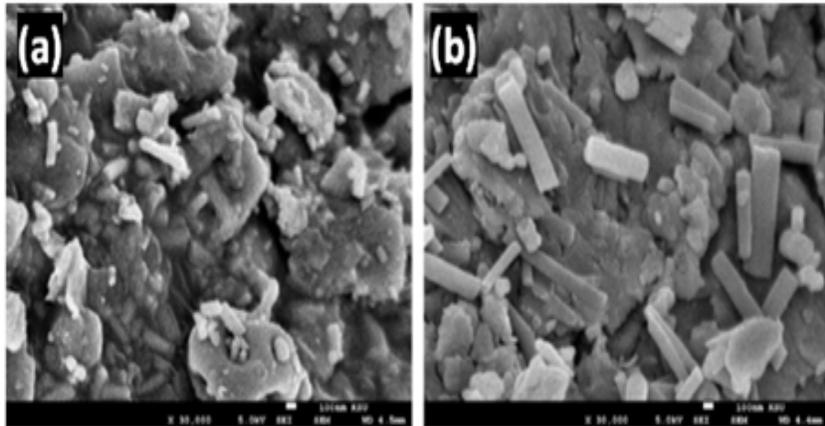


Figure 4: SEM images for (a) Al/TiO<sub>2</sub> (b)Al / CNF

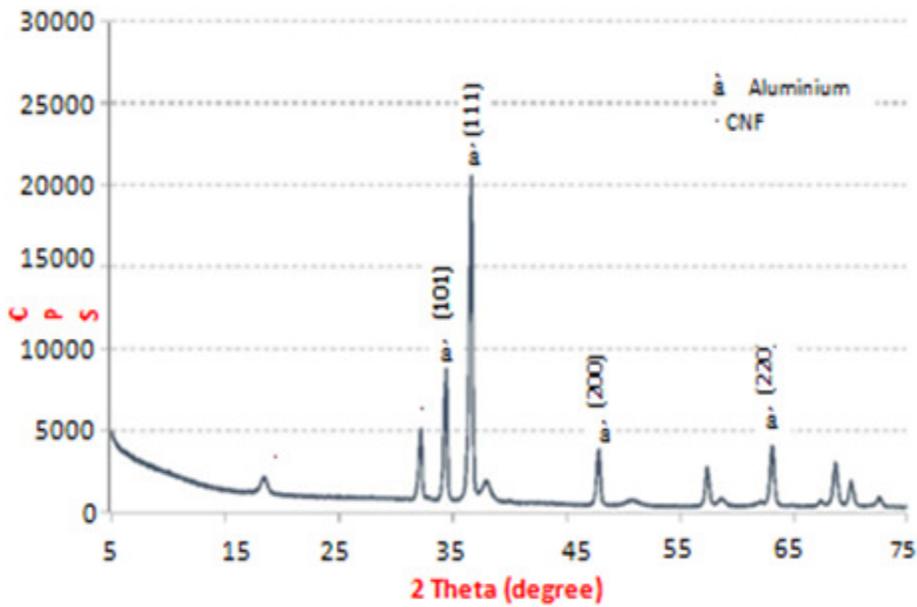


Figure5:XRD Scanning for Al/CNF powder after mixing

Fig. 5 shows the XRD patterns of Al/CNF powder after mixing. It can be observed that the main peaks is corresponds to Aluminium crystals which present at  $2\theta$  equal to 38, 46 and 63, in addition to the carbon fiber main peak at  $2\theta = 16.88^\circ$  corresponds to a spacing of  $d = 5.25 \text{ \AA}$ .

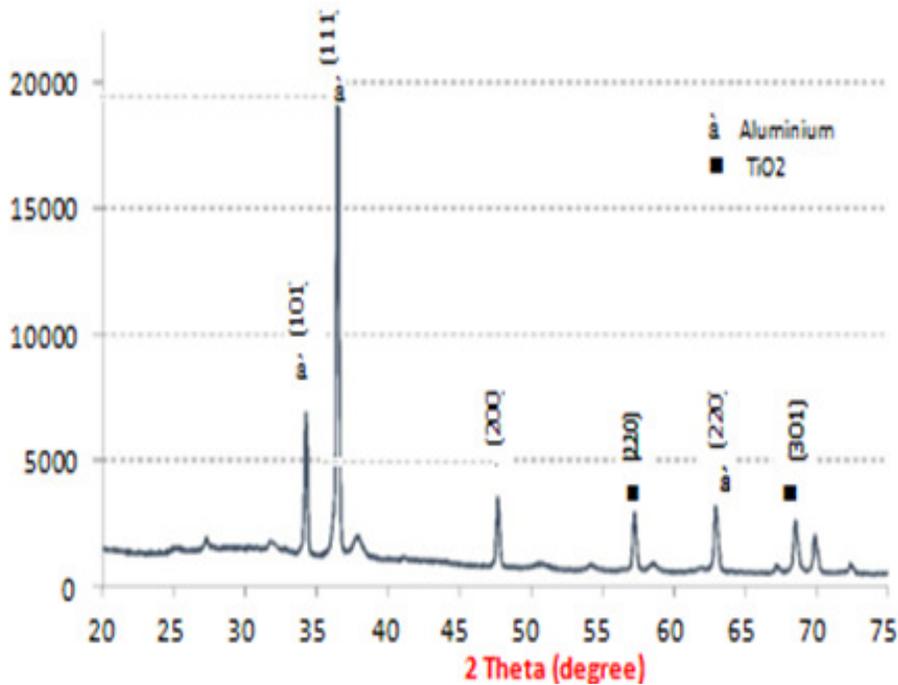


Figure6:XRD Scanning for Al/TiO<sub>2</sub> powder after mixing

Fig. 6 shows the XRD patterns of Al/TiO<sub>2</sub> powder after mixing. The results indicated that, the XRD configurations are similar to those found in Al/CNF except the replacement of CNF by TiO<sub>2</sub>. The peaks of Aluminium which present at  $2\theta$  equal to 38, 46 and 63, in addition to the TiO<sub>2</sub> fiber main peaks at  $2\theta = 32.3^\circ$  and  $57.16^\circ$

### 3.2.DENSITY AND HARDNESS MEASUREMENTS

Density and hardness results of the synthesized nanocomposites measured for different three samples from each composition are shown in Table 3. From the density measurements, marginal increase in the density values by increasing the reinforcements percentage was observed and thereby ensuring the suitability of the fabricated Al composites for weight critical applications. A maximum hardness of  $\sim 100.72$  was observed in the case of Al and 10% TiO<sub>2</sub> nanocomposites. From the results of the density and hardness measurements, it is observed that by utilizing the fabrication methodology, near dense Al based materials can be fabricated.

Table 3: Density and hardness measurements of Al nano composites.

SI No.	Material	Reinforcement		Density measurements		Hardness measurements HV
		Morphology	(Wt %)	Density (gm/Cm <sup>3</sup> )	Theoretical Density	
1	Pure Al		0 %	2.67	2.70	76
2	Al	TiO <sub>2</sub>	1 %	2.66	2.71	91.03
3			3 %	2.62	2.73	95.67
4			5 %	2.64	2.75	97.68
5			10 %	2.62	2.80	100.72
6	Al	CNF	1 %	2.54	2.69	77.95
7			3 %	2.48	2.68	76.08
8			5 %	2.36	2.66	74.92
9			10 %	2.29	2.63	74.13

### 3.3.COMPRESSION PROPERTIES

It can be seen that from Figs. 7 and 8, increasing the percent of ceramic nanofiber content, the ultimate compressive strength increased to 415 MPa at 5% TiO<sub>2</sub>. The increase of fracture strength might be related to the good interfacial bonding between the ceramic NFs clusters and Al matrix. While the opposite happened by increasing CNFs content to the Al matrix. The decrease of strength maybe related to the poor interfacial bonding between the CNFs clusters and Al matrix, which weakened the crack bridging effect of the CNFs consider

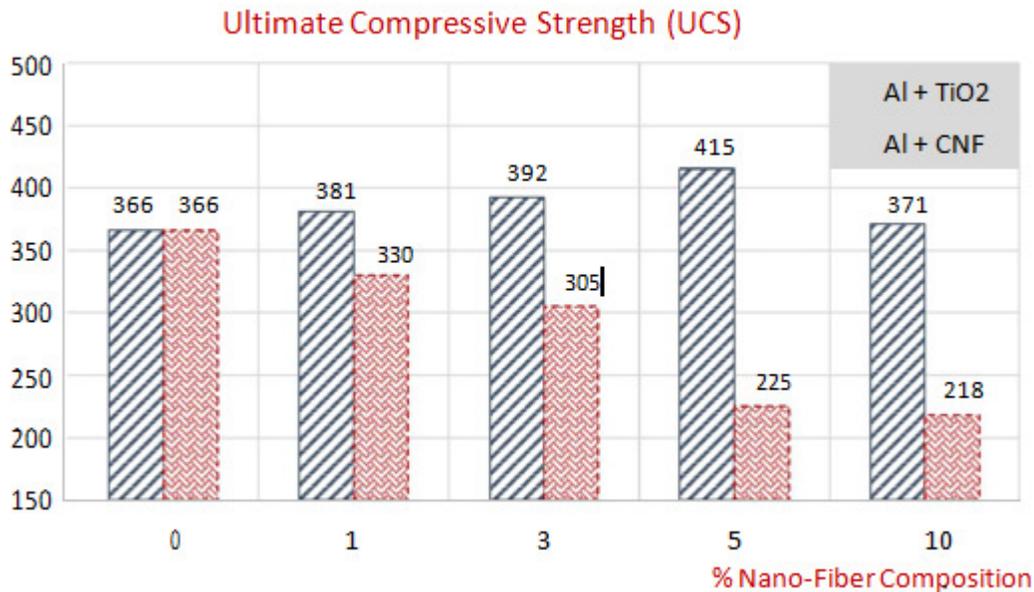


Figure 7: Ultimate compressive stress for Al / TiO<sub>2</sub> and Al / CNF compared by pure Aluminium

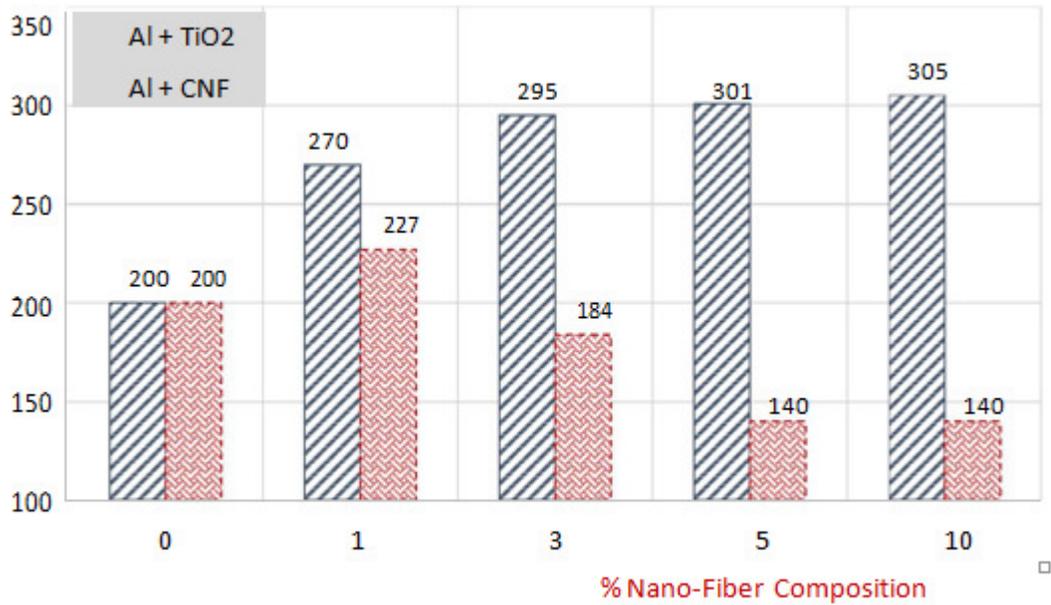


Figure 8: Yield compressive strength for Al / TiO<sub>2</sub> and Al / CNF compared by pure Aluminium

Fracture surfaces of Al composites with TiO<sub>2</sub> and CNF contents are shown in Fig.

9 (a) and (b). The addition of nanofibers help in improving the solidification

10 process. As shown, the distribution and orientation of nanofiber in the aluminium composites is homogeneous. There is no change observed during the synthesized nanocomposites process according to the XRD results Figs. 10 and 11

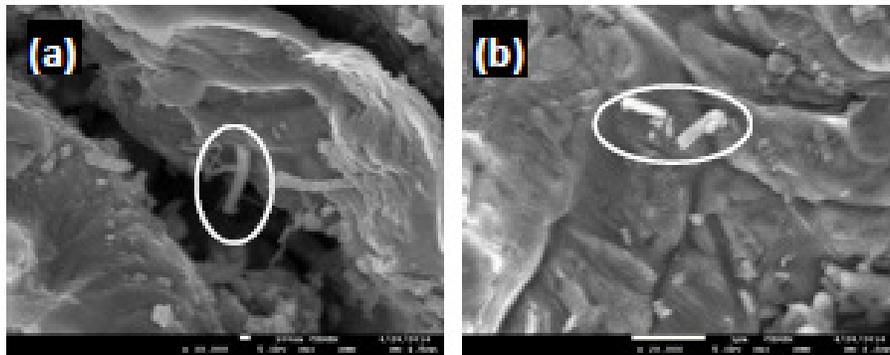


Figure 9: SEM images for the fracture surface after compression test (a) Al / TiO<sub>2</sub> (b) Al / CNF

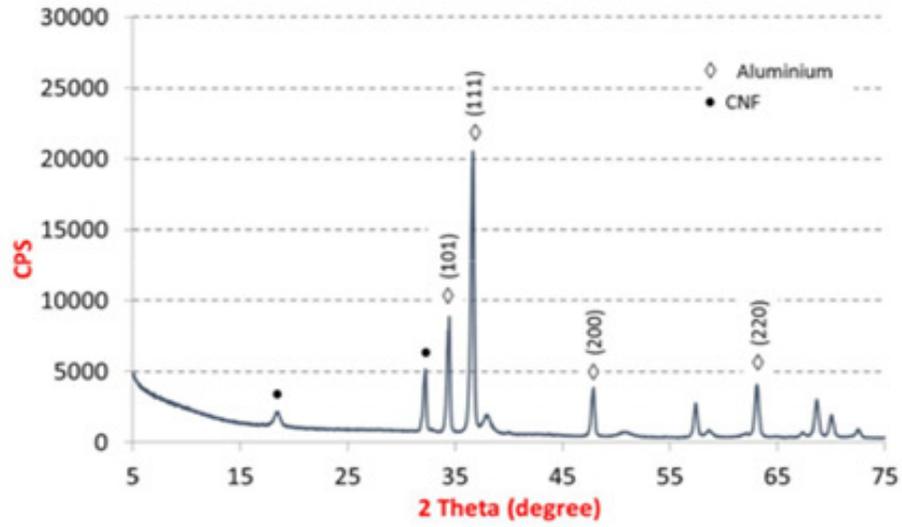


Figure10:XRD Scanning for Al/CNF synthesized nano composites

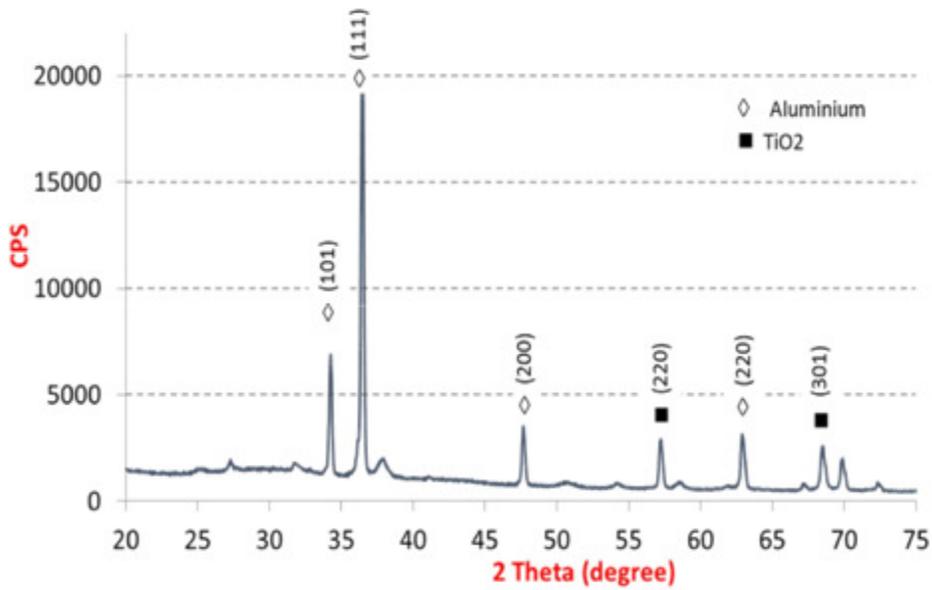


Figure 11: XRD Scanning for Al / TiO<sub>2</sub> synthesized nanocomposites.

#### 4. CONCLUSION

Carbon nanofibers and ceramic nanofibers (Titanium Oxide) were successfully synthesized and utilized as reinforcement for light metal matrices. Composites were successfully prepared using High Frequency Induction Heat Sintering furnace (HFIHS). The results can be concluded as follows:

1. The addition of ceramic nanofibers into light metal matrices clearly showed improvement in the reinforcement is mainly due to the good interfacial adhesion between fibers and metal matrix, which leads to improvement of the mechanical properties of the composite, high hardness.

2. Furthermore, the mechanical properties of the reinforced composites can be modulated by adjusting the volume fraction of nanofibers.

3. Adding CNFs to the light metal matrices leads to a decrease in strength especially at high percentage (more than 1%).

4. Compressive properties results show that there are simultaneous improvements in hardness, yield and ultimate compressive strengths (UCS) up to 10 wt.% TiO<sub>2</sub> NF. But while using CNFs as a reinforcement, an increase in the yield strength and hardness were observed till 1.0 wt.% of CNFs in Al/Mg, then, more addition of CNFs to the metal matrix will lead to poor yield, UCS and hardness.

#### REFERENCE

- [1] N. Chawla, K.K. Chawla, Metal Matrix Composites, Springer, New York, 2013.
- [2] S. Rawal, Metal–matrix composites for space applications, JOM 53 (4) (2001) 14– 17.
- [3] B. Ratna Sunil, Developing Surface Metal Matrix Composites: A Comparative Survey, International Journal of Advances in Materials Science and Engineering (IJAMSE) Vol.4, No.3, July 2015.
- [4] Wei-Hsio Chen, Hao-Tung Lin, Jianmin Chen, Pramoda K. Nayak, Alex C. Lee, Horng-Hwa Lu, Jow-Lay Huang, Microstructure and wear behavior of spark plasma sintering sintered Al<sub>2</sub>O<sub>3</sub>/WC-based composite, International Journal of Refractory Metals and Hard Materials, 54 (2016) 279-283.
- [5] I. Tsunemichi, M. JianFu, D. Shangli, S. Ichinori, S. Naobumi, L. Gilles, High strain rate superplasticity of TiC particulate reinforced 2014 aluminum alloy composites, Materials Science and Engineering A 364 (2004) 281–286.
- [6] I. Kerti, Production of TiC reinforced-aluminum composites with the addition of elemental carbon, Materials Letters 59 (2005) 3795–3800.
- [7] M K Surappa, Aluminium matrix composites: Challenges and Opportunities. Sādhanā Vol. 28, Parts 1 & 2, February/April 2003, pp. 319–334.
- [8] N. Hansen, Strengthening of aluminium by a three-dimensional network of aluminium-oxide particles, Acta Metall. 17 (1969) 637–642.

- [9] Zhang DL, Koch CC, Scattergood RO. The role of new particle surfaces in synthesizing bulk nanostructured metallic materials by powder metallurgy. *Mater Sci Eng A* 2009;516:270–5.
- [10] Montazeri A, Javadpour J, Khavandi A, Tcharkhtchi A, Mohajeri A. Mechanical properties of multi-walled carbon nanotube/epoxy composites. *Mater Des* 2010;31:4202–8
- [11] Bhaduri A, Gopinathan V, Ramakrishnan P, Miodownik AP. Processing and properties of SiC particulate reinforced Al–6.2Zn–2.5Mg–1.7Cu alloy (7010) matrix composites prepared by mechanical alloying. *Mater Sci Eng A* 1996;221:94–101.
- [12] Sub ZQ, Zhang D, Li GB. Evaluation of dry sliding wear behavior of silicon particles reinforced aluminum matrix composites. *Mater Des* 2005;26:454–8.
- [13] Slipenyuk A, Kuprin V, Milman Y, Goncharuk V, Eckert J. Properties of P/M processed particle reinforced metal matrix composites specified by reinforcement concentration and matrix-to-reinforcement particle size ratio. *Acta Mater* 2006;54:157–66.
- [14] Dudina DV, Georgarakis K, Li Y, Aljerf M, LeMoulec A, Yavari AR, et al. A magnesium alloy matrix composite reinforced with metallic glass. *Compos Sci Technol* 2009;69:2734–6.
- [15] Lee MH, Kim JH, Park JS, Kim JC, Kim WT, Kim DH. Fabrication of Ni–Nb– Ta metallic glass reinforced Al-based alloy matrix composites by infiltration casting process. *Scripta Mater* 2004;50:1367–71.
- [16] Scudino S, Surreddi KB, Sager S, et al. Production and mechanical properties of metallic glass-reinforced Al-based metal matrix composites. *J Mater Sci* 2008;43:4518–26.
- [17] Khalil, K.A.; Sherif, E.-S.M.; Nabawy, A.M.; Abdo, H.S.; Marzouk, W.W.; Alharbi, H.F. Titanium Carbide Nanofibers-Reinforced Aluminum Compacts, a New Strategy to Enhance Mechanical Properties. *Materials* 2016, 9, 399.