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Enhancing the Surface Properties of Aluminium Alloy 6061 by Laser Cladding with DWCNTs and MWCNTs using an Automated Pulsed Nd:YAG Laser

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Abstract
Industrial applications of nanomaterial in particular carbon nanotubes (CNTs) have increased from various viewpoints, such as, high performance, energy saving, cost saving, and environmental concerns. Laser cladding technique has been used successfully to modify the surface properties of materials, i.e., improving their wear and corrosion resistance. This work involves cladding aluminum alloy (Al 60601) with different types of carbon nanotubes (multi-walled carbon nanotubes (MWCNTs) and double walled carbon nanotubes (DWCNTs)) by an automated pulsed Nd:YAG laser. This was achieved by controlling selected cladding parameters which are laser power, pulse duration, and working frequency. The experimental investigation included microhardness, energy dispersive X-ray, corrosion resistance and microstructure topography by scanning electron microscope. The optimum estimated laser parameters for achieving the best results at spot diameter of 3 mm were (peak power of 3.1 kW, pulse duration of 16 msec (3 msec as pre heating and 13 msec as the main action time of the pulse) and working frequency of 1.5 Hz. Results indicated that laser cladding of Al 6061 using DWCNT enhanced three of the most important of surface properties which are hardness, abrasive wear resistance and corrosion resistance more than did the MWCNTs. The distribution of the MWCNTs on the surface of Al 6061 is better than that for the DWCNTs but its penetration is less.
Keywords: Laser cladding, CNTs, DWCNTs, MWCNTs, AL6061.

1. Introduction

Over the past few years laser surface treatment was chosen as one way to overcome premature failure in semi-solid casting by developing amorphous layer on die surface. Rapid development in surface engineering field leads to utilization of advanced heat sources such as plasma, laser, ion, and electron. Tool and die industries pay attention to the technology of laser surface treatment due to its precision of operation, short processing time and localized treatment effects (Fauzun et al. 2013).

Laser cladding has been used successfully to modify surface properties of materials, i.e., improved wear and corrosion resistance. This process is usually classified into either preplaced powder cladding or blown powder cladding (Henry et al. 2001).

Al6061 alloys have been widely used as structural materials in aeronautical industries due to their attractive comprehensive properties such as low density, high strength, ductility, toughness and resistance to fatigue (ManjunathaL.H & P.Dinesh 2013).

Carbon nanotubes (CNTs) got much of interest due to their high properties (Moosa et al. 2014). Its unique properties such as chemical inertness, physical properties such as electrical conductivity, tensile strength, flexibility, elasticity and thermal conductivity enable to be a material that can be used in a variety of applications including energy storage and energy conversion devices; sensors; field emission displays; hydrogen storage media; nanometer-sized semiconductor devices, probes and most important high-strength composites especially metal matrix composites (Moosa et al. 2014; Tripathi et al. 2010). The applications of CNTs in metal matrix composites (MMCs) especially Al matrix composites (AMCs) have been widely reported in recent years (Liao et al. 2011).

Using of (CNTs) to manufacture layers on the metal's surface is a means to obtain materials with unique properties which can find their way in a wide range of technical and medical fields. CNTs deposited on the metal's surface can constitute an attractive material (Fraczek-Szczypta et al. 2013).

2. Experimental materials and methods

2.1 Materials

The substrate materials selected to be cladded by pulsed Nd:YAG laser for this study were Aluminum alloy (Al 6061) and different type of Carbon nanotube (CNTs) Multi-walled carbon nanotube (MWCNTs) & Double-walled carbon nanotube(DWCNTs) as a clad material. (DWCNTs) was supplied by Timesnano company Chengdu Organic Chemicals Co. Ltd., Chinese Academy of Sciences and (MWCNTs) was supplied

by NANOSHEL LLC, a Wilmington, Delaware USA and Intelligent Materials Pvt. Ltd.
Chemical composition and mechanical properties of Al6061 alloy shown in table 1.1 and 1.2.

Table 1.1 Chemical compositions for the used material in Wt%.

Element	AL 6061
Si	0.661
Fe	0.541
Cu	0.30
Mn	0.068
Cr	0.20
Ni	0.014
Zn	0.12
Ti	0.015
Co	0.001
Pb	0.009
V	0.009
Al	Bal.

Table 1.2 Mechanical properties for Al 6061.

Properties	Al6061
Elastic Modulus (Gpa)	70-80 (SURESH & KUMAR 2013; Kumar et al. 2010)
Density (g/cc)	2.7 (SURESH & KUMAR 2013; Kumar et al. 2010)
Poisson's Ratio	0.33 (Kumar et al. 2010)
Hardness (HB500)	30 (Kumar et al. 2010)
Hardness (HV)	107 (SURESH & KUMAR 2013)
Tensile Strength (Mpa)	310 (SURESH & KUMAR 2013)
% Elongation	21.66% (Malhotra et al. 2013)

Specifications of both types of CNTs are shown in Table 1.3 and 1.4 according to the specification certificate issued by the previously mentioned companies.

Table 1.3 specifications of DWCNTs.

Property	Unit	DWCNTs TND	Method of Measurement
OD	nm	2-4	HRTEM,Raman
Purity	wt%	>60	TGA & TEM
Length	microns	~50	TEM
SSA	m2/g	>350	BET
ASH	wt%	<1.5	TGA
EC	s/cm	>100	
Tap Density	g/cm3	0.14	

Table 1.4 specifications of MWCNTs.

Materials	Multi walled Carbon Nanotubes
Stock NO	NS6130-06-640
Category	M-SL-1
Diameter	20-30 nm
Length	3-8 µm
Purity	>95%(MWCNT)
Amorphous carbon	<3%
Residue (Calcination in air)	<2%
Average interlayer distance	0.34nm
Special surface area	90-350* m2/g
Bulk density	0.05-0.17 g/cm3
Real density	1-2 g/cm3
Charging	2180 (Capacity: mA h/g)
Discharging	534 (Capacity: mA h/g)
Volume Resistivity	0.1-015 ohm.cm (measured at pressure in powder)

2.2 Experimental Method

2.2.1 Samples Preparation and Cladding Design

The preparation steps that were followed for (Al 6061) metal plate of 3 mm thickness are listed as follows :

1- Cutting the Al 6061 plate into small sheets of (10 ×10) mm.
2- Cleaning these sheets using pure alcohol for removing dirts and oil.
3- Grinding the sheets along the same direction using different grades of abrasive silicon carbide papers of grad (600,800 and 1000) grain/cm2 for removing the oxide films and reducing the roughness in order to achieve the best deposit film.
4- Washing the sheets by pure alcohol and then drying to prevent surface oxidation for longer time as possible before cladding.

Preplaced powder method was used in this present work. slurry of CNTs was made by mixing the CNTs with alcohol. Alcohol was used because it is a volatile substance. The slurry was placed over the (10mm * 10mm) Al 6061 sample's surface to cover the whole surface as shown in Figure (1.1). Then the samples were left for ten minutes to allow the alcohol to evaporate. This process was repeated for all four samples.

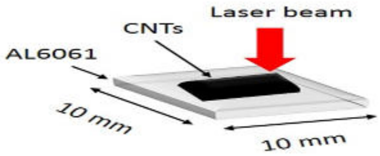

Figure 1.1 Schematic diagram illustrating the coating process.

2.2.2 Working Setup

The working setup that was employed for executing this work consists of Automated Nd:YAG 1.064μm wavelength pulsed laser model PB80 manufactured by Han's Laser Technology Co., Ltd. was employed that provides the laser beam for cladding process and the working station system that provides the motion for both laser head and sample as shown in Figure (1.2).

Figure 1.2 Working setup. (a) the laser (b) the manipulator (c) automated working table (d) the delivery system, (e) the attachments.

Table 1.5 shows the laser parameters for Cladding process.

Table 1.5 laser working parameters.

Spot size (mm)	Area (mm^2)	Δt (ms)	P_P (Kw)	Pav (W)	W. Freq (s^{-1}).	E (J)	Scanning Speed (mm/sec)	Power density (W/mm^2)
3	7.068	16	3.1	67.43	1.5	44.95	.5 mm/sec	438.596

3 Results and discussions

3.1 MicroHardness

The effect of both types of CNTs (MWCNTs and DWCNTs) on microhardness of Al 6061 can be seen clearly from Figure 1.3. Vickers microhardness of Al 6061 is about 40VH. By cladding with MWCNTs the microhardness achieved 71VH. The value of microhardness becomes more than three times greater than that for Al 6061 after cladding with DWCNTs. As the abrasive wear resistance is directly proportional to the increase in hardness this means that the surface wear resist of Al 6061 is increased by means of cladding with CNTs and precisely by DWCNTs. As a result one of the most important surface mechanical properties of Al 6061 is enhanced by this process.

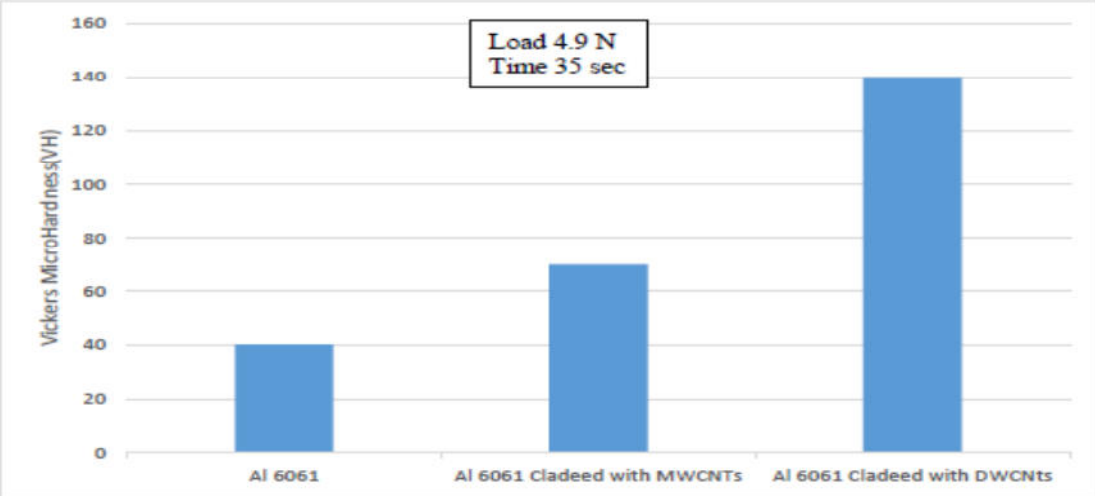

Figure 1.3 Vickers MicroHardness.

3.2 Energy Dispersive Spectroscopy (EDS)

The first EDS test is for cladded sample with MWCNTs as shown in Figure (1.4). Three peaks can be seen in this Figure. The higher one is for carbon element at about 12800 counts of X-ray emission and the other two peaks are for aluminum and oxygen elements. The aluminum and oxygen element's peaks are much lower than that for carbon which is about 800 numbers of count for aluminum and 400 counts for oxygen. Thus, the most distributed element on the surface absorbs most of the electron beam and emits X-ray that can be detected as a higher peak and that element is carbon as can be seen clearly in the Figure. Also low peak of aluminum means that the MWCNTs cover the whole surface of the aluminum substrate. Moreover, the low peak of oxygen means that there is low oxidation during laser cladding process.

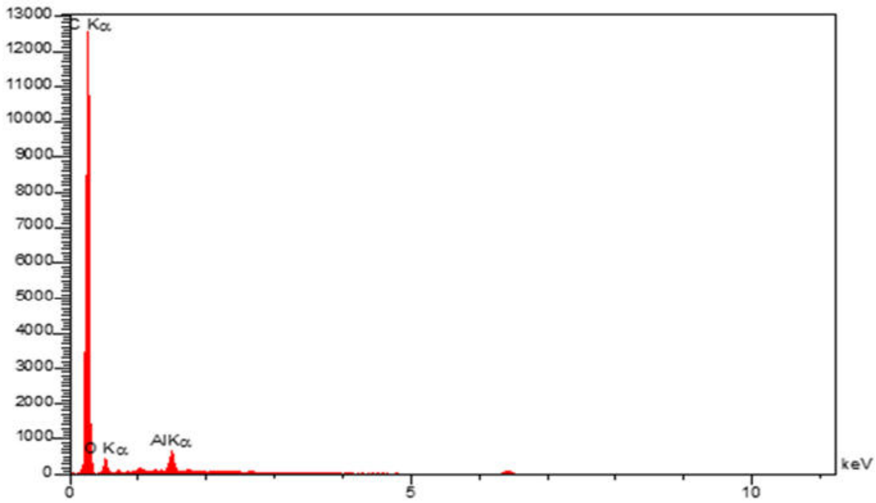

Figure 1.4 EDS for Al 6061 cladded with MWCNTs.

The second EDS test is for cladded sample with DWCNTs. As shown in Figure (1.5). Also three peaks are recorded in this Figure, for carbon, oxygen and aluminum. The highest peak is for carbon element which is 10000 counts, the lower peak is for aluminum which is 800 counts, and the lowest one is for oxygen which is 500 counts. From these numbers it can be concluded that the DWCNTs clad layer covers the whole surface of the aluminum substrate. Moreover, it can be noticed here also that the low peak of oxygen means that there is low oxidation during this laser cladding process.

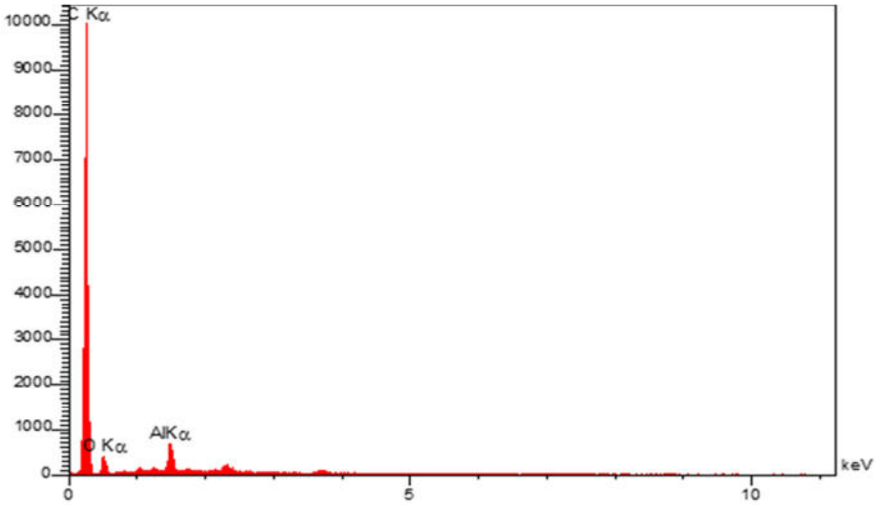

Figure 1.5 EDS for Al 6061 cladded with DWCNTs.

3.3 Corrosion

Three groups of samples undergo the corrosion test under the effect of two deferent environments 3% Nacl solution and 3-mol HCl acid. Each group consists of two samples. The first group of three uncladded Al samples one of them undergoes the salty solution test and the other undergoes the acidic solution test. The second group of two cladded samples with MWCNTs follows the same sequence for the first group. The procedure executed for the third group of two cladded samples with DWCNTs.

Figure (1.6) shows different images using an optical microscope for Al 6061 after and before the corrosion test. Figure (1.6 a) illustrates an image for the substrate before immersing in salty and acidic solutions. It shows that the substrate's microstructure is clear from any dissolution and pits.

After immersing the Al 6061 in the salty solution for ten days and inspecting the microstructure via the optical microscopic Figure (1.6 b) a distribution of pits due to corrosion appears clearly on the surface of Al 6061. Doing the previous procedure after immersing the Al 6061 in the acidic solution for ten days the optical microscopic microstructure's image shows severe pits and defects on the surface as can be seen obviously in Figure (1.6 c).

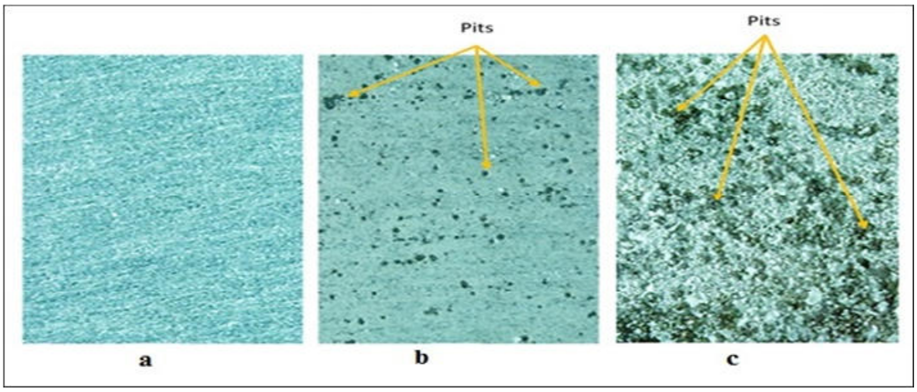

Figure 1.6 Al 6061(a) Before corrosion test, (b) after immersion in salty solution for ten days and (c) after immersion in acidic solution for ten days.

Figure (1.7) shows three optical microscopic images for the sample's surfaces of the second group before and after immersing in the salty and acidic solutions. Figure (1.7 a) shows the microscopic structure sample's surface cladded with MWCNTs before immersing in any solution.

On one hand the microscopic image for the cladded sample with MWCNTs which is immersed for ten days in salty solution shows obviously the deposited NaCl on sample's surface and only few pits can be seen as shown in Figure (1.7 b). The reason of this result is due to the cohesive and undissolved layer of MWCNTs in such salty environment which immunizes the original surface of Al from being eroding. Hence, this clad layer retarded the corrosion rate.

On the other hand Figure (1.7 c) shows the microscopic image for the cladded sample with MWCNTs that is immersed for ten days in acidic solution. It can be seen clearly that the clad layer remains cohesive with no pits and just narrow zones of the uncladded Al surface are dissolved by the acid. Again the reason of this result is due to the cohesive and undissolved layer of MWCNTs in such acidic environment which immunizes the original surface of Al from being eroding. Hence, this clad layer retarded the corrosion rate in this environment also.

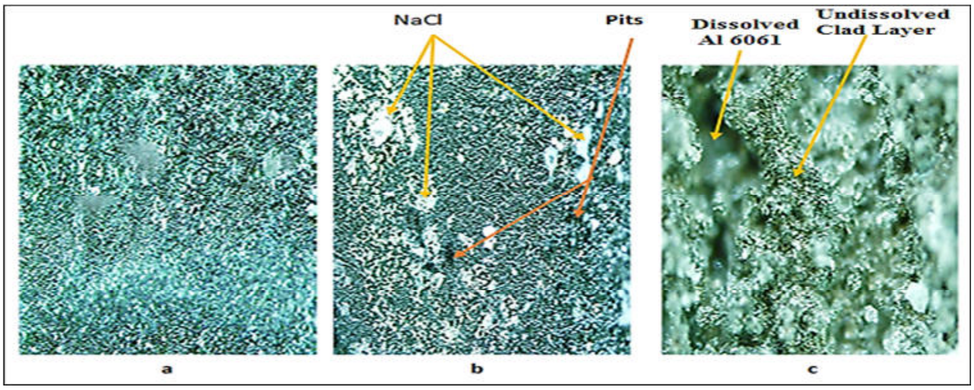

Figure 1.7 Al 6061 cladded with MWCNTs (a) Before corrosion test, (b) after immersion in salty solution for ten days and (c) after immersion in acidic solution for ten days.

For completing the comparison and having full indication about the corrosion behave for both cladded and uncladded samples Figure (1.8) shows three optical microscopic images for the sample's surfaces of the third group before and after immersing in the salty and acidic solutions. Figure (1.8 a) shows the microscopic structure sample's surface cladded with MWCNTs before immersing in any of the two solutions.

After immersing the cladded sample in the salty solution for ten days Figure (1.8 b) illustrates the microstructure image for the surface showing the deposited NaCl on sample's surface above the clad layer with no trace for pits that means the DWCNTs forms undissolved protection layer in a salty solution even better than that for the MWCNTs one. Hence again, this clad layer retarded the corrosion rate in this environment. Figure (1.8 c) shows the microscopic image for the cladded sample with DWCNTs that is immersed for ten days in acidic solution. It can be seen obviously that the clad layer remains cohesive with no pits and just narrow zones of the uncladded Al surface are dissolved by the acid and even less than that with the MWCNTs. This result is due to the cohesive and undissolved layer of DWCNTs in such acidic environment which protects the original surface of Al from being eroding. Hence, this clad layer creates high protection and retarded the corrosion rate in the acidic environment also.

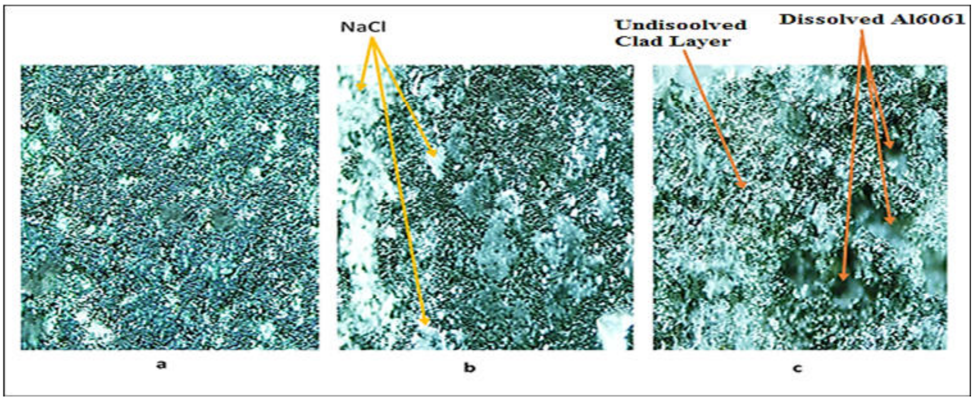

Figure 1.8 Al 6061 cladded with DWCNTs (a) Before corrosion test, (b) after immersion in salty solution for ten days and (c) after immersion in acidic solution for ten days.

3.4 Scanning Electron Microscope (SEM)

3.4.1 Cross Section (SEM)

The microstructure topography for the cross section of each cladded sample (with MWCNTs and DWCNTs) is to record how deep is the propagation of the CNTs inside the original base metal surface which indicates the efficiency of the laser cladding process that was followed for the benefit of this work study. Figure (1.9) illustrates the cross section microstructure topography for the sample with MWCNTs. With magnification of 3.50 kX it can be seen clearly the thickness of the clad layer at three deferent locations and the average clad layer thickness is determined to be 22.88 μm.

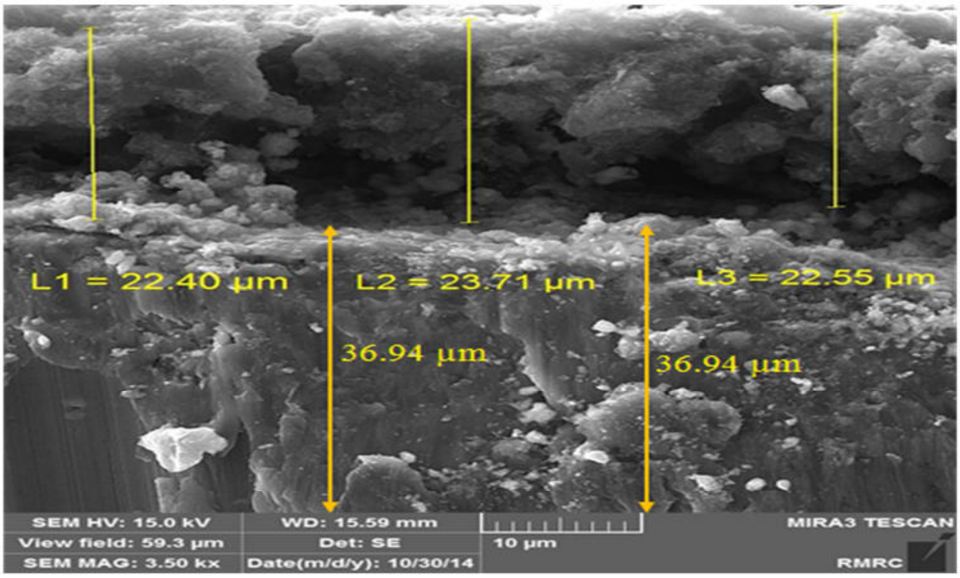

Figure 1.9 Cross section SEM for sample cladded with MWCNTs.

The second cross section microstructure topography is for cladded sample with DWCNTs. Figure (1.10) shows the image with magnification of 1.50 kX for the thickness of the clad layer in different locations and the average value is determined to be 38.68 µm.

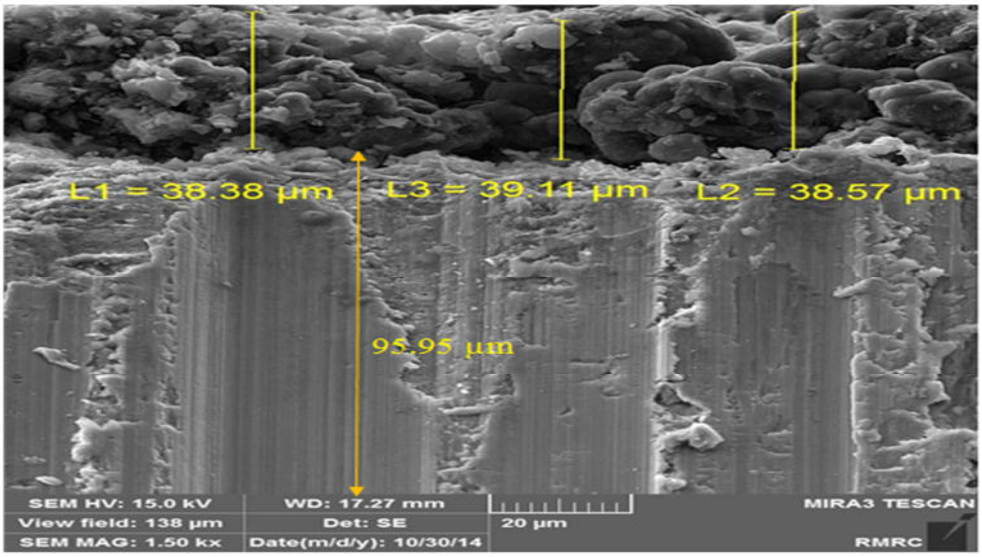

Figure 1.10 Cross section SEM for sample cladded with DWCNTs.

3.4.2 SEM for Surface

Three samples (cladded with MWCNTs, cladded with DWCNTs, uncladded) were scanned by SEM. Figure (1.11) illustrates three images with magnifications of 1.00 kX, 50.0 kX and 100.0 kX for the uncladded sample showing the surface microstructure topography of Al 6061.

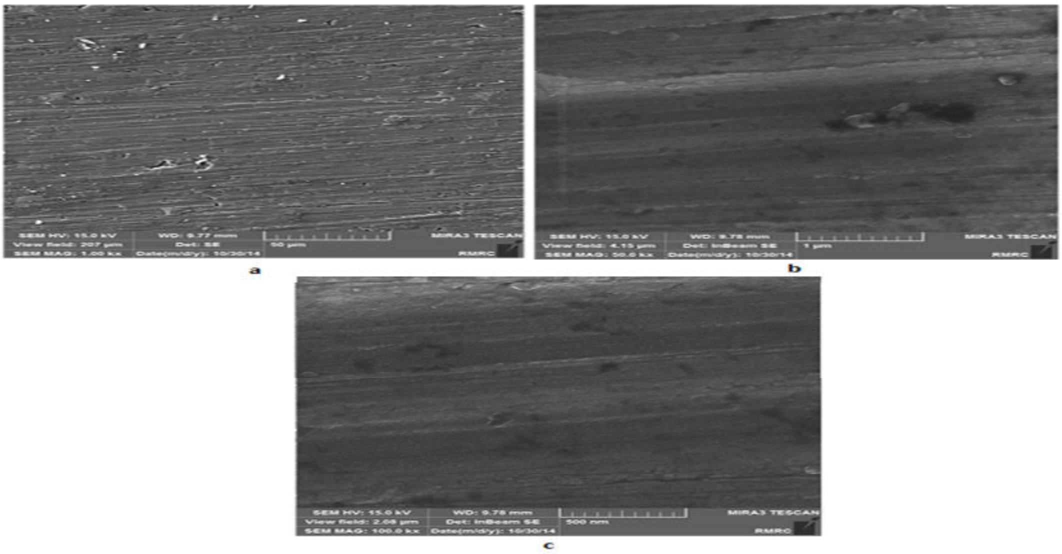

Figure 1.11 SEM images for uncladded sample (a) 1.00 kX magnification, (b) 50.0 kX magnification and (c) 100.0 kX magnification.

Figure (1.12) illustrates SEM images for the MWCNTs layer showing four images with different magnifications. Figure (1.12 a) shows the cluster of MWCNTs with 1.00 kX magnification. Figure (1.12 b) with 50.0 kX magnification illustrates how the MWCNTs form continuous fibers of CNTs. The separate MWCNTs of about (20 - 30) nm in diameter can be shown in Figures (1.12 c) and d with 100.0 kX and 200.0 kX respectively.

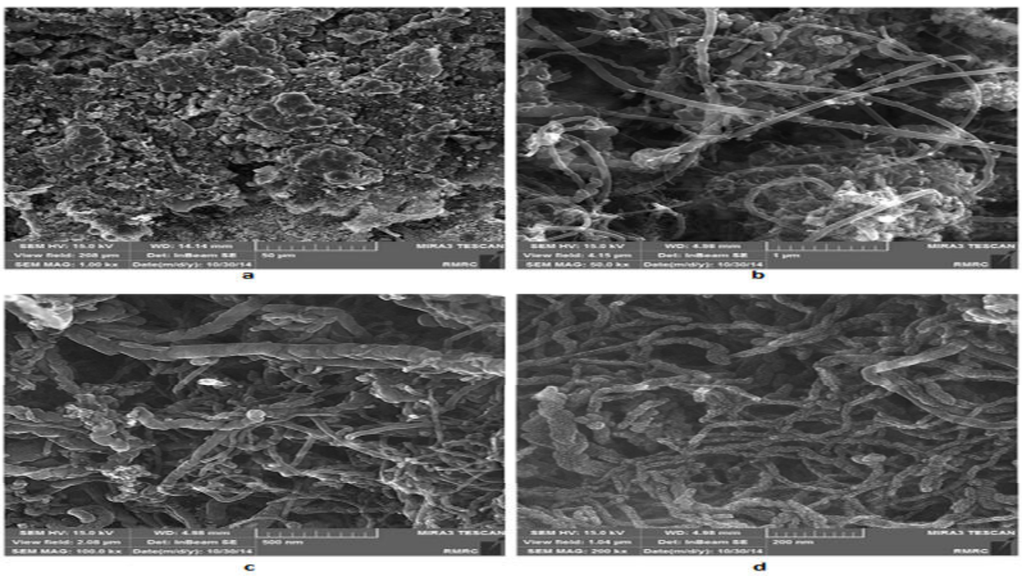

Figure 1.12; (a) SEM picture for sample cladded with MWCNTs (a) 1.00 kX magnification, (b) 50.0 kX magnification, (c) 100.0 kX magnification and (d) 200 kX magnification.

Microstructure topography for the surface of the cladded sample with DWCNTs is shown in Figure 3.10 with different magnifications. Figure (1.13 a) illustrates the DWCNTs as cluster with magnification of 1.00 kX. Figures (1.13 b) show film of DWCNts with magnifications of 50.0 kX. Figures (1.13 c and d) present the DWCNTs as separate fibers with magnifications of 100.0 kX and 200.0 kX respectively.

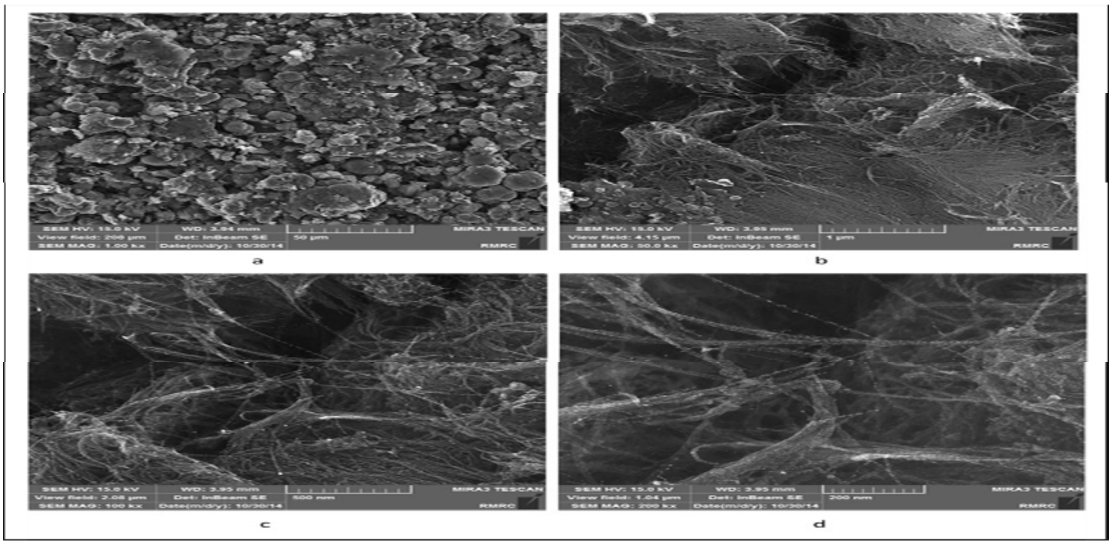

Figure 1.13; (a) SEM image for sample cladded with DWCNTs (a) 1.00 kX magnification, (b) 50.0 kX magnification, (c) 100 kX magnification and (d) 200 kX magnification.

4. Conclusions

An overall look on the experimental and computational results that were recorded leads to conclude the following:

1- MicroHardness test show that the cladded Al 6061 with both types of CNTs enhancing this property the abrasive wear risistance also enhanced as it is directly proportion with hardness. DWCNts enhanced the microhardness three times from the value of uncladded Al 6061 and up to two times from the value of cladded with MWCNTs this means that cladded Al 6061 with DWCNTs has the best result for enhancing the hardness and abrasive wear resistance than from cladded with MWCNTs.

2- EDS test show that the peak of MWCNTs higher than from peak of DWCNTs i.e. the counts of emitted X-ray from MWCNTs greater than counts of DWCNTs that means MWCNTs have better distribution on substrate surface than DWCNTs.

3- The optical microscopic images of sample cladded with both type of CNTs after Corrosion test in salty and acidic environments showing that cladded Al 6061 with both types of CNTs enhancing the corrosion resistance. The images also show that after immersion Al 6061 Cladded with DWCNTs in salty solution only precipitation of NaCl on clad layer can be seen from image under microscope while image for Al 6061 cladded with MWCNTs shows that there are precipitation of NaCl on clad layer and some pits on surface. Images also showing that after immersion Al 6061 cladded with DWCNTs and Al 6061 cladded with MWCNTs in HCl the dissolved Al 6061 in sample cladded with DWCNTs less than dissolved Al 6061 in sample cladded with MWCNTs.

The Corrosion test showing that the DWCNTs enhanced the corrosion resistance of Al 6061 in salty and acidic environments better than enhancing of MWCNTs.

4- SEM cross section images show that the thickness of DWCNTs clad layer thicker than MWCNTs clad layer that means DWCNTs penetrated inside the substrate higher than penetrating of MWCNTs.

5- SEM images showing the distributed DWCNTs and MWCNTs on Al 6061's surface as cluster, continuous fiber and individual CNTs.

6- As result of conclusion from all above. Cladded DWCNT on Al 6061's surface enhancing better than MWCNTs three of most important of surface properties which are hardness, abrasive wear resistance and corrosion resistance.

MWCNTs distributed on Al 6061 better than DWCNTs but the penetration of MWCNts inside substrate less than DWCNTs.

Suggested future works of the case study which is covered by this work can be more confirmed by many suggestions that are proposed for the future work as follows;

1- Studying the effect of the surface roughness on thickness of the cladding layer.
2- Studying the effect of the laser mode of operation by executing this process using an adequate CW laser.
3- Studying the effect of the initial temperature (pre heating) on the final quality of the process.
4- Measuring the optical and the thermal properties of the Al 6061 cladded with both types of CNTs (MWCNTs and DWCNTs) for enhancing the results.

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