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Experimental Analysis of Effects of Time of Metallization, Ph and Stirring of the Nickel Coated Multiwalled Carbon Nanotubes Prepared Using Electro Less Method.

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Abstract: Multiwalled Carbon nanotubes (MWCNTs) is a novel material which posses an excellent mechanical, electrical and thermal properties. MWCNTs are the suitable material for fabricating functionally graded materials. The present work discusses the Electro less Nickel (Ni) coating of the MWCNTs and the effects of metallization time and the pH over the coating are reported. The Ni coating upto 21% was achieved with the electroless method. The presence of pdcl$_2$ influences greatly on the coating by increasing the more activation sites over the surface of MWCNTs. The resultant MWCNTs was further characterized by using Scanning electron microscope and XRD techniques. The effect of various parameters over the percentage of Ni coating and its surface morphology has been studied.

Keywords: Multiwalled Carbon nanotubes (MWCNTs), Electro less Method, Nickel Coating, Functionally graded material.

I. INTRODUCTION

Ever since from the discovery of Carbon Nanotubes during investigation of fullerene production, from then onwards Carbon Nanotubes has attracted the researchers to study the unique properties of carbon nanotubes and has been focused on many research applications.carbon nanotubes are chemically inert this property makes it suitable for huge applications it passes high tensile strength and elastic modulus which attracts researchers to use it as reinforcements its electrical and thermal conductivity is also high thus, research into practical applications of MWCNTs, such as resin – MWCNT, ceramic – MWCNT, and metal – MWCNT composites, has been actively pursued. In the case of metal - CNT composites, the focus has been on mechanical properties of composites [2]. When the MWCNT are coated before adding as a reinforcement to the matrix , the composite prepared have enhanced the properties considerably hence the quest for the improved metal oxide coating has resulted in the development of relatively new technique known as electroless coating and one of them is nickel coating by incorporating CNTs in Nickel sulphate [3]. MWCNTs are a novel material and possess sophisticated properties. The nature of interface plays a prominent role in the overall grades of a composite material, improper wetting and chemical reaction occurring between the reinforcement surface and matrix at the interface can deteriorate the mechanical properties of the resulting composites [4]. Formation of reaction product layer on MWCNT surface enhanced the wettability of molten Al – Si alloy on MWCNT during thermal spraying [5]. The composites with the nanotube reinforcements can be synthesized in various routes but the interface bonding during synthesis of composites or during service conditions will be affected if there is a chemical reaction at the interface [6]. The Ni coating of the reinforcements is the way to tackle above problems; various researchers have adopted this coating technique to enhance the wettability [7] – [8]. The strong interface enhances the transfer and distribution of the load from the matrix because of the increased elastic modulus [9]. By coating the synthesized Carbon Nanotube with a conducting metal we can ensure that all the Carbon Nanotubes have similar electrical properties though their structural and physical properties may vary, the metallization of carbon nanotube is feasible by a simple evaporation process leading to metallic nanotube with reliable electrical and magnetic properties. It is also possible that metallic coatings which form intermetallic with the matrix alloy due to the presence of alloying elements the intermetallics along with the alloying elements can happen [10] – [12]. The ratio of ceramic to metal content can be altered to suit different applications and also the issue of varying the porosity level. Electroless nickel plating is used extensively within industry and in fact is the most prevalent electroless coating used for engineering purposes. An advantage of this process is that both metallic and non-metallic substrates can be coated provided they have been suitably pre-treated [14]. The coating bath solution can be used to control the pretreatment of the MWCNTs the high pH increases the active sites for good adhesion [15].

According to H. Landis et al., [16] good wetting between the solid and liquid is essential for the formation of strong bonds between them during casting. This paper will briefly discuss the phenomenon of Ni coating on MWCNTs. Wettability is the ability of the liquid to spread on a solid surface and represents the close contact between them. Techniques such as the addition of an alloying element, coating and/or treating the particles to increase the wettability in MMCs.

The aim of this work is to use electroless Nickel coating technique in metallization of MWCNTs and coating was carried out in three steps starting from pretreatment procedure which comprised of cleaning MWCNTs with the concentrated sulphuric acid known as sensitization then activation in the presence of palladium chloride and finally metallization with nickel sulphate solution. The different deposition time resulted in different percentage of coating the effect of varying parameters was studied then the morphology of the coating was characterized using SEM, XRD and EDAX techniques.

II. EXPERIMENTAL

A. Materials

MWCNTs used in this study were supplied by United Nanotech Innovations Pvt. Ltd., Bangalore the carbon nanotubes in the powder form. These are synthesized through catalyst assisted chemical vapour deposition method they have average diameter of 20-30 nm and length measured is about 16-20 µm, , yield stress of around 4.3 GPa and elastic modulus of 70 GPa

B. Electroless Nickel coating

The MWCNTs was pre-cleaned in concentrated sulphuric acid for about 2 hrs and it was thoroughly rinsed with deionised water. The pre-cleaning is necessary to remove the residual catalyst metal particles from MWCNTs which would have been present during the synthesis of nanotubes through chemical vapour deposition so that any side effects due to its presence can be eliminated. The advantage of the pre-clean is it also enhances the surface of the MWCNTs to adhere the Ni metal film over it.

The pre-cleaned MWCNTs were sensitized in aqueous solution containing 0.0081M $SnCl_2$ (Stannous Chloride) and 0.0026M HCl (Hydrochloric acid) for about 10 min. Later $Pdcl_2$ (palladium Chloride) of about 0.025 grams is added to the solution for activation and stirred for about 15 minutes at 40^{o}C. followed by rinsing with deionised water for about 15 min. the sensitization improves the adsorption capacity of the surface of MWCNTs. the presence of $PdCl_2$ helps in nucleating the centers for active sites over the surface of nanotubes. Finally the pre treated MWCNTs is immersed in the bath, as a the electroless process involves the reduction of nickel sulphate salt solutions with sodium hypophosphite. The reaction utilizes about one third of the hypophosphite reducing power because of a concurrent reaction between the hypophosphite and water to produce hydrogen and phosphite. The general process condition involves a bath composition wherein is included nickel sulphate, ammonium chloride, sodium citrate, ammonium citrate, and sodium hypophosphite. The concentration of nickel may be 30 g per liter, the concentration of ammonium chloride may be 50 g per liter, the concentration of sodium citrate 100 grams per liter, and the concentration of sodium hypophosphite 10 g per liter.

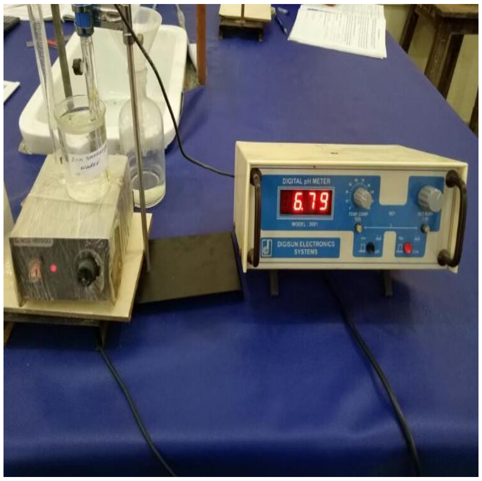

Fig. 1: The pH of the Concentrated sulphuric acid

Fig 2 : Nickel Sulphate Solution addition

Fig 3: MWCNTs immersed in nickel sulphate solution Fig. 4: Nickel coated MWCNTs is filtered using

The pH of the solution strongly controls the deposition rate of the nickel. The most satisfactory pH range for optimum nickel deposition is between 8.5 and 14.0 with an optimum range of between 9.0 and 9.2. However, the pH may be as high as 10. Since the pH control is critical, the bath must be monitored and corrected as required to maintain the proper pH. This may be accomplished by the addition of ammonium hydroxide in a continuously recirculating system.
The temperature of the bath appears to have no significant effect upon the alloy common position, but seriously affects the deposition rate. Deposition rates at solution temperatures below 92°C are negligible. At temperatures in excess of 100°C, the solution may become unstable so as to spontaneously reduce the metals from the solution, for this reason, the preferred temperature of the bath is in the range of between 93 and 95°C. Because of the relatively high temperature involved and the narrow range of pH which is allowable, it may be necessary to add large quantities of ammonium Sulphate during the coating process. The nickel sulphate solution is added to the resulting solution and allowed for about 5 mins. The deposited MWCNTs were then filtered and rinsed with distilled water then dried in hot oven at different time and temperatures.

Table 1. Electroless Bath Composition

Carbon Nano-tube	40gm
Palladium chloride	0.025gm
Sodium hypophosphite	1g/100mL
Ammonium citrate	6.5g/100mL
Ammonium chloride	5g/100mL
Nickel Sulphate	3g/100mL

Table 2. Operating conditions for Electroless Nickel coating

Temperature	70-90° C.
pH	6-12
Time	10-50 minutes
Deposition rate	6 µm/hour

The coatings on MWCNTs were studied varying time of metallization and pH values using XRD to understand coating morphology then SEM and EDAX analysis was also carried out to characterize the resulted nanotubes.

III. RESULTS AND DISCUSSION

With the increase in the sensitization time, the surface was susceptible more to receive a metal coating. Later it was sensitized for about 10 minutes , there was no remarkable increase in the percentage of Ni deposited with the increased time of sensitization , slight increase in deposition was found. As for the time of activation is concerned Fig. 5 shows the 15 minutes of activation allows the highest percentage i.e. 21% of Ni coating with the further increase in the activation time beyond 15 mins, considerable percentage deposition was not found. The pH of the metaloxide solution was kept higher and for higher pH value the deposition rate foundto be increased, Fig. 10 shows the SEM image for pH 9 where the recorded time to get a considerable Ni coating of MWCNTs is 6 min meanwhile for higher pH 12 the minimum time required to get a equivalent Ni coating of MWCNTs was about 3 min almost 50% of the time required to coat is reduced with the 75% increase in the pH value as shown in the fig 6.

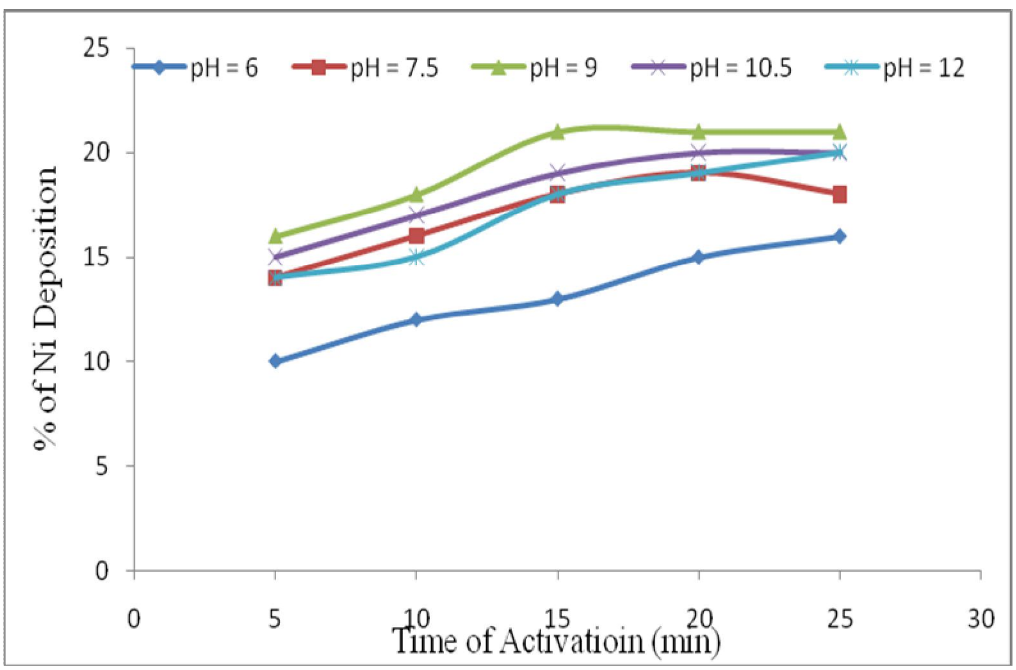

Fig. 5: Percentage of Ni Deposition vs Activation time (min

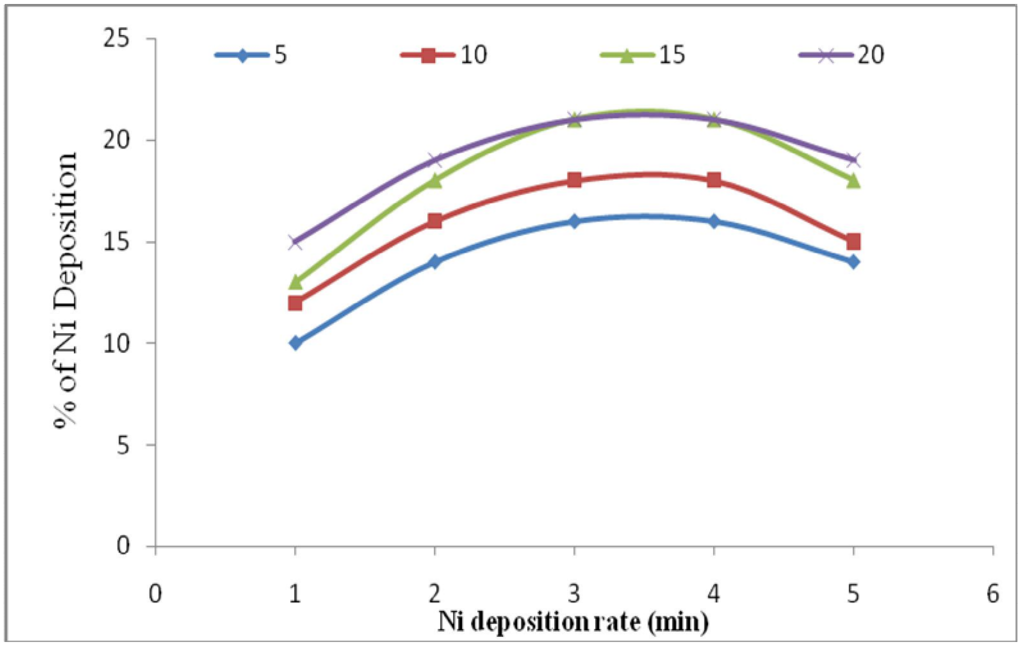

Fig. 6: Percentage of Ni Deposition vs Activation rate (min)

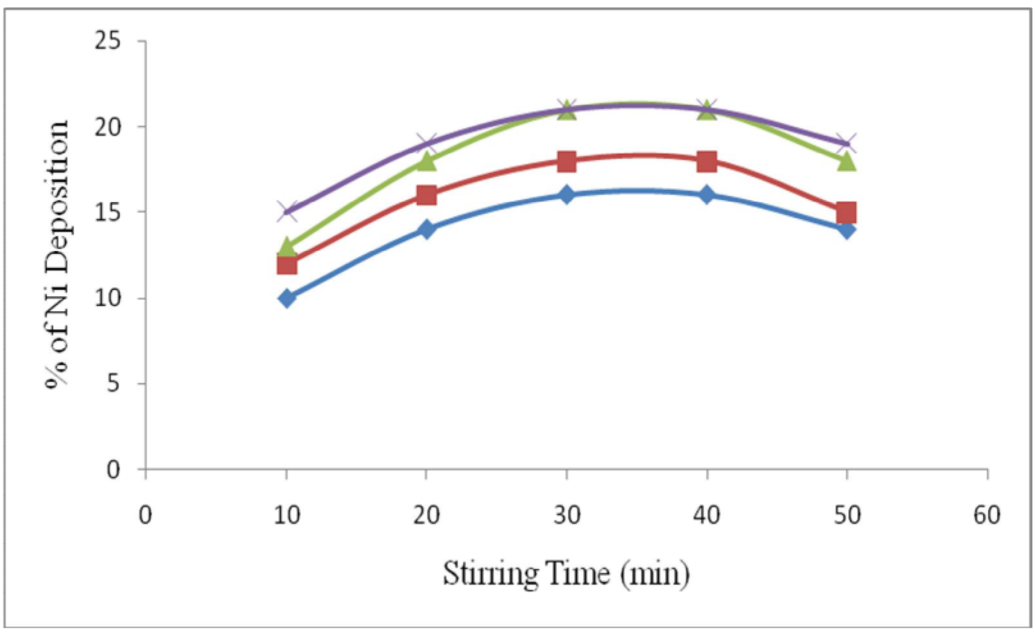

Fig. 7: Effect of Stirring time vs percentage of Ni deposition rate

Fig. 7 shows with the increase in the time of continuous stirring of MWCNTs contained electroless bath, the percent of Ni deposition onto carbon nanotubes also gone up with the time of stirring about 40 min. the presence of palladium activating agent on the nanotube surface serves like catalytic reagent with increased activation sites . Once the coating is initiated further it continues on its own. The immersion of the activated MWCNTs in the nickel sulphate bath and it should be stirred with non stop to get a uniform metal coating on the surface of MWCNTs.

The morphology of uncoated and coated MWCNTs was analysed with a scanning electron microscope (SEM) JEOL JSM 6360—A model with a magnification capacity of 40k× and accelerating voltage of 10 kv with working distance (WD) 6.9 mm and a spot size 2.2 . Microscopes were equipped with analytical facilities (dispersive X-ray Diffraction-XRD).

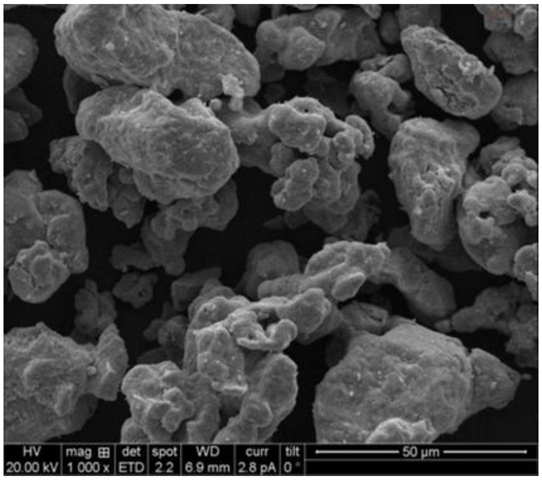

Fig 8: SEM image of uncoated MWCNTs

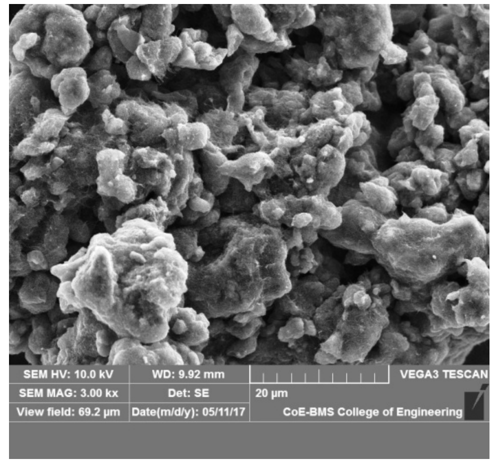

Fig 9 : SEM image of Ni coated MWCNTs

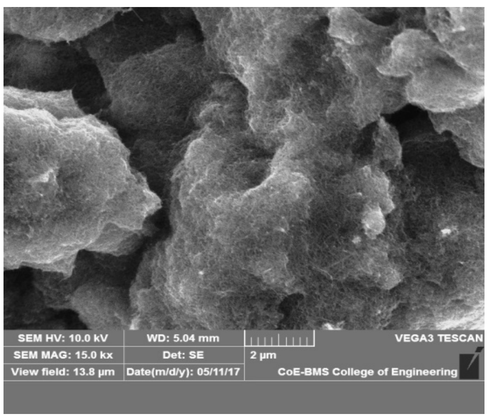

Fig 10 : SEM image of Ni coated MWCNTs pH 9 deposition time 6 mi

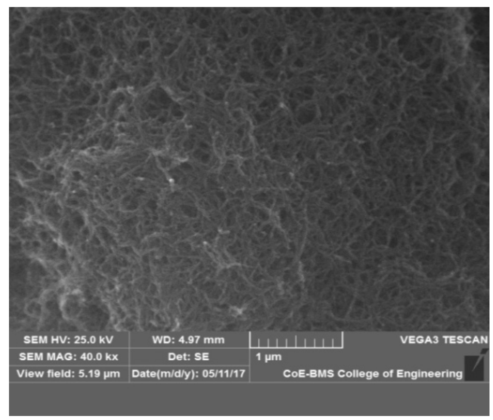

Fig 11: Nickel coated MWCNTs is filtered

using WATT-MAN filter paper

The aim is intended to measure the percentage of metal coating on the surface of MWCNTs at a nano level. The experimental results were characterized by micro-structural observations using scanning electron microscopy (SEM) and X-ray Diffraction (XRD) and Energy Dispersive X-Ray Analysis (EDAX) of Ni coated MWCNTs resulted from the Electroless deposition method.

XRD pattern of MWCNTs is shown in the fig. 12 which confirms Ni coating on MWCNTs the agglomerations among the nanotubes were observed due to the severe chemical reaction of Ni atoms. The intial MWCNTs exhibit an amorphous surface which becomes dim after it was sensitized and activated , the SEM results in fig 11. Shows that the Ni coated MWCNTs appear like fiber with uniform dispersion.

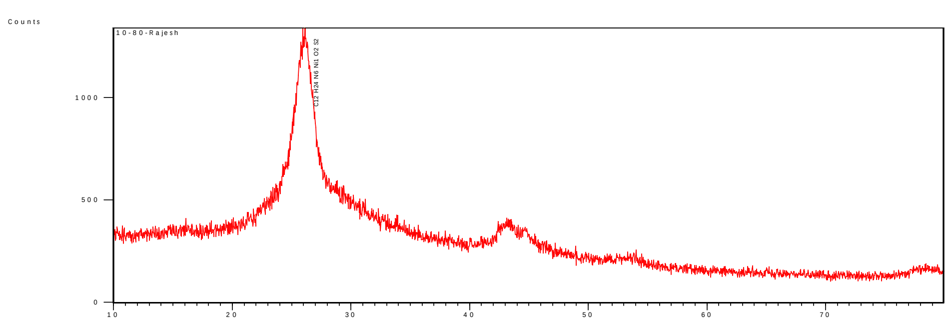

Fig 12: XRD pattern of Nickel coated

The XRD results indicate a successful sensitization and activation process and it reveals that diffraction peaks occur at $2\theta = 79.18^o$, 43.8^o and 50.3^o are attributed to the fcc Ni (1 1 1), (1 0 0) and (2 0 0) present in the samples. The intensity of Ni phase increases with the increase in temperature. Several weak peaks were also observed the XRD results clearly indicates that the Ni metal coating was introduced over the surface of the MWCNTs by electroless nickel deposition. The Ni coated MWCNT have a polycrystalline structure which is evident from the diffraction spots. Fig. 13 shows different surfaces of MWCNTs that was selected and analysed for the metal coating characteristics for pH 12 majority of the area was found to be uniformly coated, for the increase in time of stirring also improved the surface area covered with the nickel.

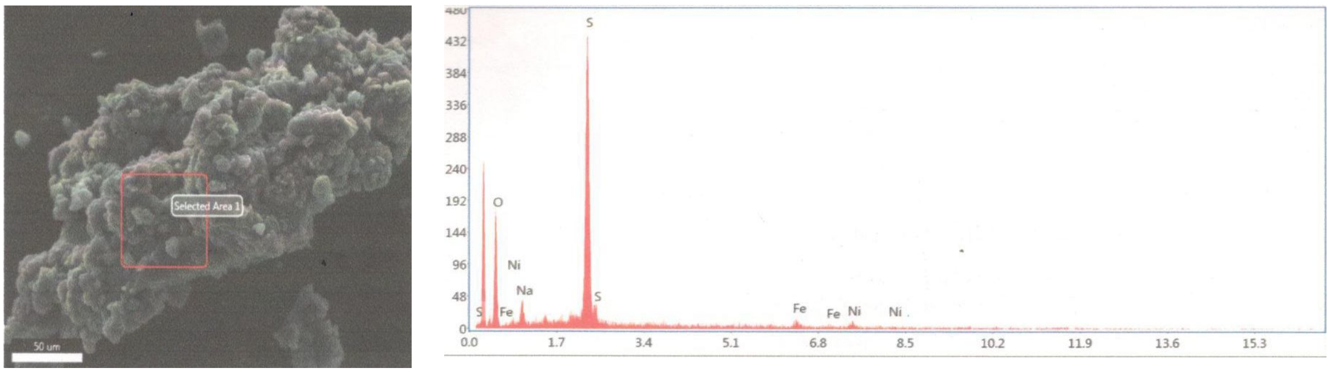

Fig 13: EDAX pattern of Nickel coated surface 1 surface192457987u9i008098674 1! MWCNTs

IV. CONCLUSIONS

The MWCNTs was coated with Ni using electroless method which included initial pre – cleaning followed by sensitization, activation and finally metallization using Nickel Sulphate. The SEM and XRD results revealed that MWCNTs was coated with Ni with an average thickness of 50nm. Due to the Ni coating of the MWCNTs will exhibit fiber like characteristics which is desirable for the preparation of the composites. The present study proves that the electroless coating was the best method to deposit Ni over the surface of the MWCNTs. The Metallization rate can be improved with the increased parameters such as pH, activation time, Stirring time and concentration of $PdCl_2$. The EDAX test confirms the uniform Ni coating on the carbon nanotubes surface.

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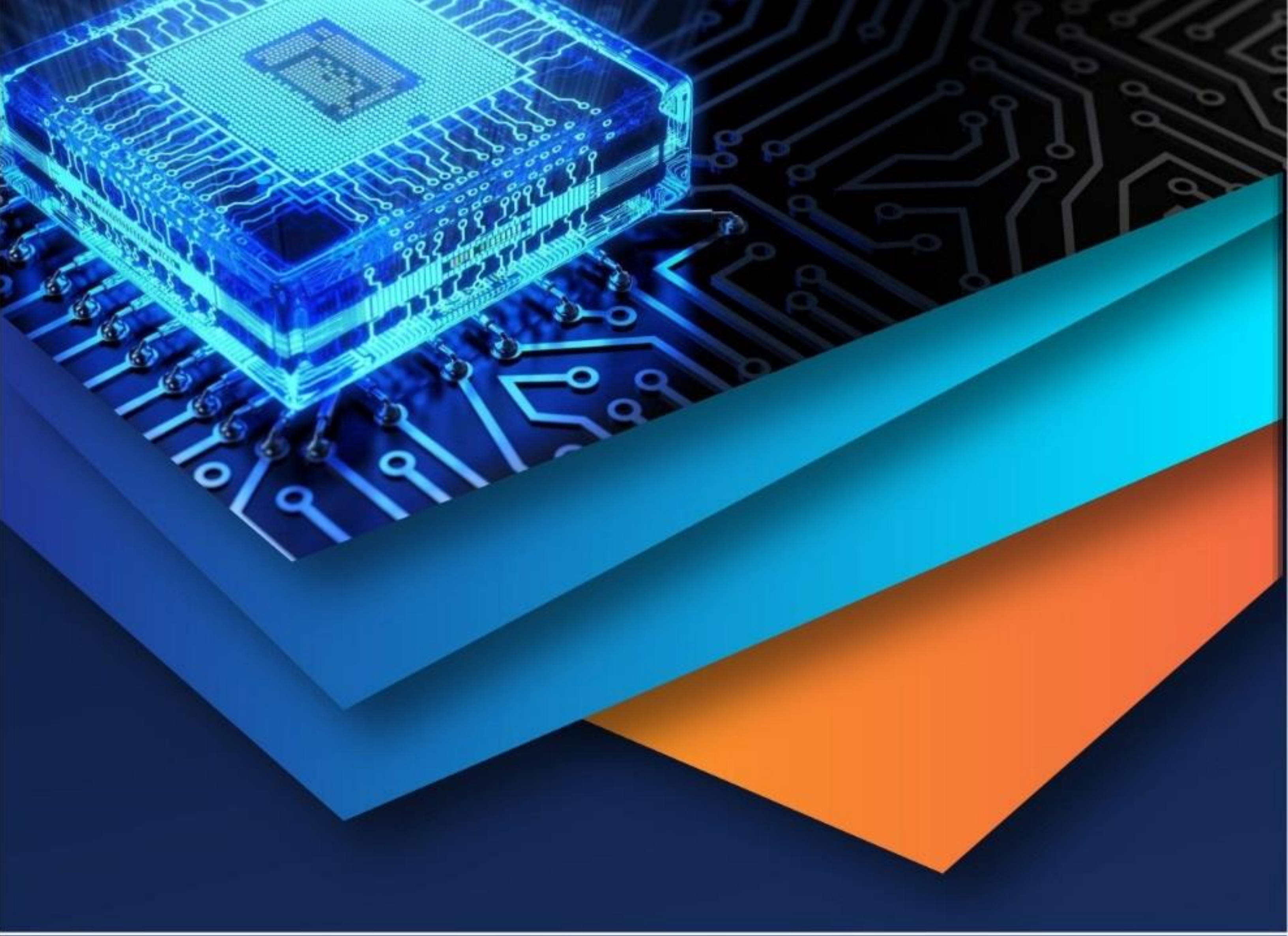

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