

Preparation, wear resisting property and application of MWCNTs/Ni composite coating for Sewing Needles

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Abstract: In this research work, multiwall carbon nanotube (MWCNT) based nickel coating is prepared and deposited on unfinished metal made sewing needle by employing electrochemical process. The coating thickness was attained to 3 microns per 90 minutes. The microstructure, micro-hardness, friction and wear properties of the Nickel-MWCNTs coatings were investigated and discussed. The uniformity and presence of primary components in the coating layer were observed and confirmed through scanning electron microscope. The friction and wear behavior of the coated and uncoated samples were tested on a pin-on disc tester. The specific wear rate of the coated sewing needle is improved significantly than the uncoated sewing needle; the co-efficient of friction of the coated needle is reduced to 2.1. The surface roughness of the coatings was measured by employing portable surface roughness tester. The surface roughness Ra of the coated sample is improved to 0.095. The Vickers hardness of the coated needle is improved to 63 compared with the uncoated needle.

I. INTRODUCTION

The sewing needle is the precise and important part which is made up of steel with enhanced surface finishes. A good sewing needle is defined with the essential properties such as smoother eye, stronger tip, low penetration force and stability in sewing. Most of the sewing threads are made up of blended fibres for improving strength and heat resistant. During the sewing process, the metallic needle has to penetrate the sewing fabric along with the threaded sewing thread. The continuous sewing operation causes the generation of heat in the needle surface due to the fabric penetration. The generated heat is transferred immediately to the sewing thread and the fabric that is in sewing in operation as well. This may cause fusing of sewing thread and sewn fabric. In order to reduce/dissipate the heat generation and reducing the friction of needle, an attempt has been made using metal matrix composite is prepared.

Where nickel used as matrix material and carbon nanotubes as reinforcement material.

A. Chatterjee and B. L. Deopura were reported that CNTs are tiny tube like structure (grapheme sheets rolled up in cylinder with diameter as small as 1 nm). It has high strength and lubrication properties compared with graphite [1].

Ramesh Chandra Agarwala, Vijaya Agarwala, V. Shadhana were concluded that the electroless plating process is suitable and reliable method for developing Ni-P based composite coating. During the deposition process, several Ni-P globules nucleate at isolated sites and grow laterally and vertical to cover the entire surface of the substrate. Then auto catalytically cover the substrate area by repeated nucleation and for lateral growth [3-6].

J. Tan, T. Yu, B. Xu and Q. Yao were investigated the microstructure and wear resistance of nickel-carbon nanotube (MWCNT) composite coating deposited through brush plating method. The Ni/MWCNT coating improves less porosity, higher hardness and higher wear resistance than that other Ni coating. MWCNT greatly improve the coating performance [6].

Yang hua was experimented and reported that the Nickel matrix Carbon nanotubes composite coating can be fabricated in the electrochemical route and the effect of MWCNTs content in the coating improves tribological performance [7].

J. Sudagar, J. S. Lian, Q. Jiang, Z. H. Jiang, G. Y. Li, R. Elansezhian reported that the performance of surfactant on electroless nickel plating over magnesium alloy material has significant influence on the coating uniformity, surface roughness and surface morphology [8].

X.H. Chen, C.S. Chen, H.N. Xiao, H.B. Liu, L.P. Zhou, S.L. Li and G. Shang were investigated that the Ni-C coating possess not only for higher wear resistance but also a lower friction co-efficient resulting in improved mechanical properties[9]. C.R. Carpenter, P.H. Shipway and Y. Zhu et al. were concluded that electrodepositing of nickel-carbon nanotube nanocomposite coatings for enhanced wear resistance which says that the pure nickel coatings do not have good adherence whereas the Ni-CNT coatings have good adherence [10-12].

Vladimir Marascu-Klien have reported that carbon is readily obtained from the pyrolysis of hydrocarbons such as resins and pitches, and can be deposited from the vapor phase by cracking hydrocarbon rich gases according to the application of carbon materials in advanced methodologies [13].

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I.E. Ayoub was reported that the deposition rate of the nano particles in electroless plating method can be varied by adjusting the voltage and current [14].

Susumu Aria et al were discussed about applications of MWCNT composite, including resin-MWCNT, ceramic-MWCNT and metal-MWCNT composite has been fabricated by plating techniques, tribological behavior of MWCNT composite coating fabricated by electroless plating, the hardness of Ni-MWCNT composite films was found to increase but the low friction co-efficient [15].

II. MATERIALS AND METHODS

The sewing needle is made up of different ranges of steel grades. In this research work, the uncoated needle made up of common steel grade of 304 is used for coating and analysis.

2.1 Methodology

The preparation of nickel-MWCNTs coating is done by employing electroless plating method. In order to get the coating uniformity and composite preparation, this method is popular to develop metal matrix composite.

2.1.1 Pretreatment

The uncoated sewing needle was carried out to various pretreatment processes (refer figure.1) for removing the surface impurities and for sound deposition. The sequence of pretreatments are as follows, soap cleaning for one minutes; acetone rinsing for 5 minutes; rinsing with demineralized water for 1 minute ; ethanol treatment for 5 minutes; demineralised water rinsing for 1 minute; HCL treatment for 30 seconds; and finally the samples were rinsed with fresh distilled water.

2.2 Electroless Plating

Electroless nickel (EL) plating was preferred for coating the uncoated needles. This is due to the versatility of bath chemicals that is used for regular nickel plating and its functions with many metals and materials are effective. The constituents of an EL solution are source of nickel ions, reducing agent, complexing agents, stabilizers/inhibitors and energy. The electroless nickel bath is prepared with the standard chemicals as shown in the below table.1

Table.1 EL Bath chemicals

Bath Chemicals (Analytical Grade)	Composition (grams per liter)
Nickel Sulphate	20 g.L ⁻¹
Sodium Hypophosphate	16 g.L ⁻¹
Sodium Acetate	10 g.L ⁻¹
Sodium Citrate	10 g.L ⁻¹
MWCNT	0.1 g.L ⁻¹
Sodium laurel sulphate (SLS)	0.1 g.L ⁻¹
pH	4.5-4.7
Temperature	85-90°C

In the plating bath, the nickel sulphate is used as source of nickel ions, sodium hypophosphite is used as reducing agent for better reduction of nickel ions and controlled bath for condition, the use of sodium acetate and sodium citrate as a complexing agent that can exert a buffering action that prevents the pH of the solution from decreasing too fast,

prevent the precipitation of nickel salts and reduce the concentration of free nickel ions.

The electroless nickel bath solution is prepared for 150 ml with the standard chemicals, and the MWCNT is dispersed in a 50 ml of distilled water with SLS by sonication for 30 minute. Then the dispersed MWCNT is loaded with the EL bath and agitated. The bath temperature is raised to 88°C and the pH of the bath is maintained in the range of 4.5-4.7. The pretreated substrate is vertical hang in to the bath for better deposition. The pH level of the EL bath is maintained by frequent addition of ammonia solution. The deposition is carried out for 1 hour 30 minutes. After that the substrate is taken out and washed with distilled water, finally air dried.

2.2.6 Heat Treatment

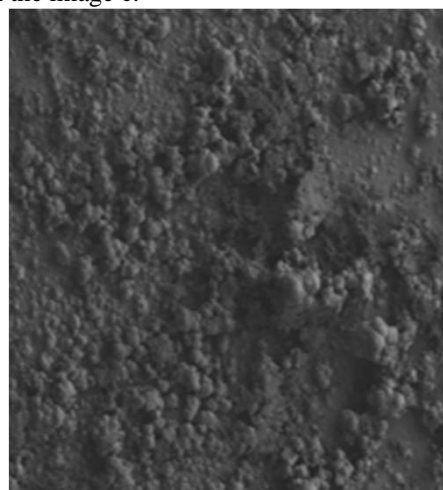
After the electroless deposition, the coated needles are annealed for one hour at 673 Kelvin in the muffle furnace. During the heat treatment process, intermetallic bond is developed between the coating layer and substrate area. Due to the diffusion of carbon nanotubes through the interstices of nickel layer, the composite density was improved.

III. RESULTS AND DISCUSSION

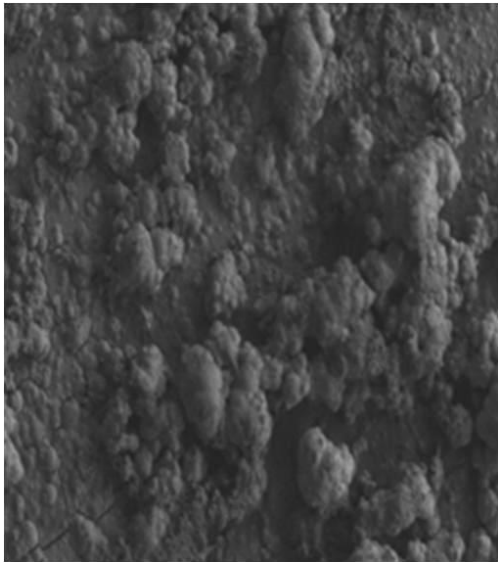
The microstructure of the coatings was observed through field emission scanning electron microscope (Fe-SEM) and metallurgical microscope. The mechanical properties such as friction, wear, weight gain, surface roughness, and microhardness were measured through various standard instruments.

3.5 Microstructure and elemental analysis

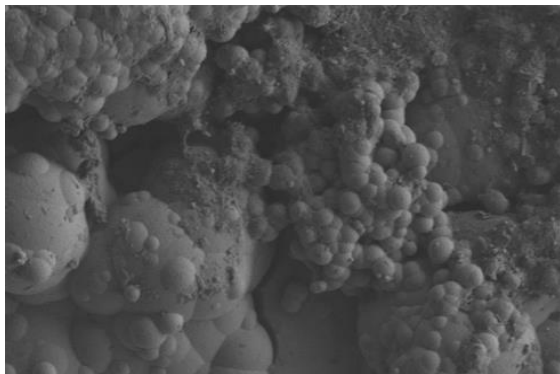
The microstructure and elemental composition of the coated and uncoated samples are analyzed through Fe-SEM. The surface morphology of the coating layer was observed at various magnification levels. The images exhibit the presence and distribution of carbon nanotubes to the coated area. The carbon nanotubes are observed at 2 micro meter magnification range and it looks like small group of worms. The images exhibit the presence of carbon nanotubes and its attachments towards the interstices of nickel globules as shown in the image c.



(a)SEM image of coated sample at 200µm



(b)SEM image of coated sample at 20µm



(c) SEM image of coated sample at 2µm
Figure 4. Fe-SEM images of coated sample

3.4 Elemental composition by EDS (energy dispersive spectroscopy)

The elemental composition of the coating layer was analysed by employing EDS. It was observed that the weight % of the nickel is about to 49% as major content. The carbon content shows about 7% and the phosphorous presence is about 6%. The carbon content is improved due to the addition of carbon nanotubes in the coating layer. In this result, all the primary elements are significantly presented in the coatings.

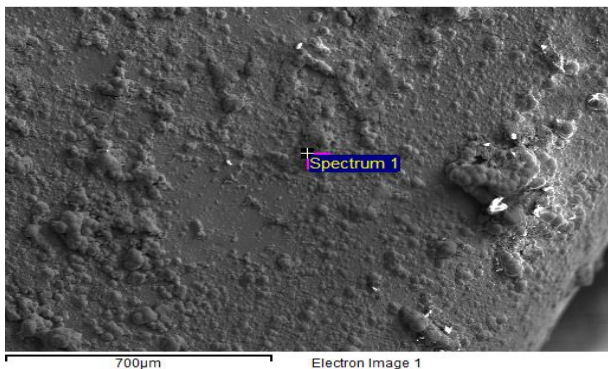


Figure 5. Image from EDS

Table 5. Elemental Composition

Elements	Weight%	Atomic%	Compd%	Formula
C	6.69	13.96	24.53	CO ₂
P	5.81	4.70	13.31	P ₂ O ₅
Cl	0.14	0.10	0.00	
Ni	48.74	20.79	62.02	NiO
O	38.62	60.45		
Totals	100.00			

3.1 Friction and Wear Testing

The wear and friction test is done using the PIN-ON-DISC apparatus as shown in figure.2. The friction and wear properties of Nickel-MWCNT coated substrate with uncoated one by pin-on-disc method.



Figure 2. Pin-On-Disc

The substrate is attached with an aluminum circular rod as a pin; EN 32 grade steel disc is used as abrading surface. The instrument was preset before testing. The specifications are pin track as 100 mm track diameter over the disc; the disc speed is 150 rpm; the load applied over the specimen is 0.5 kg; and the testing duration is 15 minutes. After the essential preset conditions, the wear test was conducted and the specific wear rate and frictional values are calculated as per standard method. The friction and wear of the coated sample is improved than the uncoated sample. This is due to the presence of carbon nanotubes and its lubricating nature.

Table 2. Frictional force and specific wear rate

Substrate	Co-efficient of friction (µ)	Specific wear rate
Uncoated sample	2.2	5.409x10 ⁻⁸
Coated Sample	2.1	5.408x10 ⁻⁸

3.2 Wear Scar Analysis

The wear scar is analyzed through the metallurgical microscope, the figure 3.a shows the intensity of the worn scar observed is very less compared to the uncoated sample. The figure.3.b shows the worn scar of uncoated samples. It appears as multiple channels and the surface atoms may be detached easily during the abrasion. This is due the strength and unique properties of carbon nanotubes.

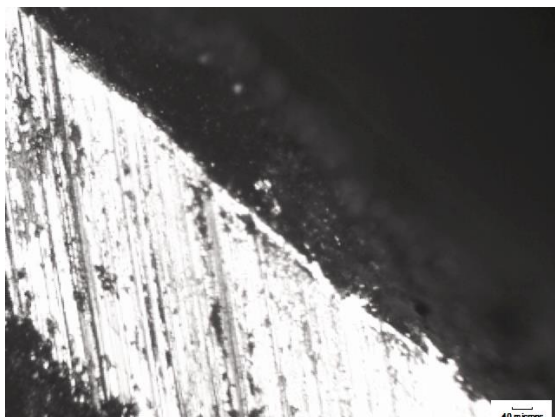


Figure 3.a. Wear scar of coated substrate



Figure 3.b. wear scar of uncoated substrate

3. 2 Weight gain and rate of deposition

The weight gain of the coated sample is calculated by simple weighing method to find the coating intensity. It was observed that the weight gain of the sample after coating is about 0.059 grams and the rate of deposition is about 3 micrometers.

Table 3. Weight gain

Sl. No	Uncoated sample weight	Coated sample weight	Weight Gain of coated sample
1.	1.058 grams	1.117 grams	0.059 grams

The rate of deposition of the coated substrate is calculated using standard formula with the following specifications, Density of nickel= 8.912 g.cm⁻³; Area of substrate= 22.2 sq.cms; Time= 1 hour 30 minutes.

3. 3 Surface roughness

The surface roughness is an important one for the abrasive wear as well as friction between the metal to fiber. It was observed that the average value of surface roughness *Ra* is about to 0.095 for coated sample, this is higher than the uncoated sample (*Ra* = 0.065). In general, the industrial recommended *Ra* value of textile machine component lies in the range of 0.068 to 0.08. The coated surface may have higher strength and lubrication nature.

3. 4 Microhardness

Microhardness of the coatings was measured through Vickers hardness tester. The tester is preset with the normal load of 50 grams for indentation. It was observed that the

micro hardness of the coated surface is higher than the uncoated samples. This is because of the improved interfacial bonding strength of carbon nanotube with nickel ions during heat treatment.

Table 4. Vickers hardness test

Hardness Values (HV 50)	
Coated sample (mean of 5 samples)	Uncoated sample (mean of 5 samples)
61	63

IV. CONCLUSION

The Nickel-MWCNT composite was successfully deposited over the unfinished sewing needle by electroless plating. The presence of primary components such as nickel, carbon nanotubes and phosphorous are confirmed through the elemental analysis quantitatively. The coating uniformity and the distribution of carbon nanotubes are obvious over the coating area. The mechanical properties such as friction, wear, surface roughness and microhardness of the coated sample is improved significantly. The results are as follows.

- The specific wear rate of the coated needle is improved to 5.408x10⁻⁸.
- The co-efficient of friction is enhanced to 2.1.
- The weight gain is obtained to 0.059 grams (after 1 hour 30 minutes).
- The deposition rate is attained to 3.010 microns / 1 hour 30 minutes.
- The surface roughness *Ra* values are improved to 0.095.
- The Vickers micro hardness is improved to 63.

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