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***Research paper***

# Investigation of the Electrodeposited CoNiFe Alloy Coating on Microstructure and Mechanical Properties

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**Abstract**

In recent years, intensive study has been conducted on ternary Co-Ni-Fe alloys because of their outstanding physical and chemical properties which are nearly close to hard chromium deposit. The CoNiFe coating is the most commonly produced by an electrodeposition method for its flexibility and easy manipulation of coating properties. Coating process acts as preventive action to corrosion problems which are one of the significant problems that can be related to the metal works. In the transportation sector such as rail transport, for instance, there is one vital part that is exposed to corrosion, and it causes a lot of negative results. Corrosion defect cost lots of money to replace the defected ones; however a functional coating which involves nanoparticles can be used to counter the problem. The objective of this research is to recycle the used or corroded fastener by implementing a coating process could be a huge step in reducing the total cost and therefore, help to maintain the environment's sustainability. Throughout this research, Nanocrystalline CoNiFe alloys were electrodeposited in sulphate bath with several constant parameters. The electrodeposition method was done in 30 minutes with a current of 1.7A and was proven to slightly improve the surface morphology. CoNiFe elements were successfully identified with lumpy globular pattern grains and acceptable thickness. Through the hardness investigation, the coating produced the microhardness of 317.94 Hv which almost doubled up the non-coated hardness. Besides that, CoNiFe coating also exhibited a lower rate of wear, a coefficient of friction and frictional force compared to non-coated fastener.

***Keywords***: *Coating; CoNiFe; Corrosion; Electrodeposition; Nanoparticles.*

## 1. Introduction

A coating is a layer or cover applied to the surface of an object, usually referred to as the substrate. The application of coating may be used for decorative, functional, or both. In the industrial environment, the surface coatings are used to increase the lifetime of the components exposed to corrosion or any destructive nature. Nanoparticle alloy coating that has been proven to be high mechanical and corrosion properties is needed for the design and operation of devices, machine and structural systems in the extreme environment [1]. These findings have been widely used to help to improve the quality of the product substantially and eventually increase their lifespan.

Nowadays, nanoparticles have become one of the advanced technologies which are useful to improve the material's performance. Their small particle sizes (<100 nm) characteristic and high volume fraction of particle boundaries have made them well known. These characteristics can be used to give rise to the unique physical and mechanical properties. Continuous developments in electrodeposition technology have led to the fabrication of nanostructured coatings and succeeded to make their steps into the industrial use and met the demands of more severe applications [2]. Previous studies have reported the improvement in strength, ductility, erosion, corrosion, and wear resistance of coatings.

Nanocrystalline Co-Ni-Fe consisting of binary and ternary Co, Ni, and Fe electroplated systems have widely been studied for decades. Electroplating of CoNiFe alloy follows the anomalous co-deposition mechanism which is defined as the less noble metal being deposited preferentially and its percentage in the deposit is higher than that in electrolytes [3]. This study is done to deposit an alternative material focusing on nanocrystalline CoNiFe which is identified as a potential candidate for replacing all hazardous corrosion resistant coating [4].

One of the major problems relating to the surface and coating technology is the corrosion caused by wear problem. The products or parts widely used in the industrial area are often exposed to an extreme environment. The railway fasteners used in the transportation sector, for instance, are one type of parts which is likely to experience corrosion problem. A suitable coating on this particular parts may lengthen its lifespan or even improve its properties. A good understanding of the relation between the corrosion properties of the nanoparticle materials and their microstructure is essential. Throughout this research, it uses railway fastener electrodeposited with the nanocrystalline CoNiFe coating with constant parameters such as temperature, deposition time and pH value. By the end of this research, the characterization, hardness and wear properties of CoNiFe applied to the recycled railway fastener are determined.

# 2. Methodology

## 2.1. Material selection and preparation

The used railway fasteners were chosen as the working specimens to be implemented with alloy coating to study the characters and its properties. The hexagonal nut was chosen as a substrate to coat with CoNiFe alloy through the electrodeposition method. For the preparation of mild steel nut, several cleaning processes were needed before it could be used for the electrodeposition process. The specimens have been used for approximately 10 years, which were entirely covered with rust condition. Due to the rusty surface condition, the first step to be done was cleaning the rust. The specimen was cleaned with water before being soaked into the vinegar with the standard procedure of 24 hours. The specimen was then removed from the vinegar and cleaned to observe the cleaned surface condition. If there were still some rusty spots on the specimen, another 12 hours of soaking into the vinegar would be needed. If the specimen was not cleaned thoroughly, the procedure was repeated. The next step was polishing the surface with copper brush to remove the remaining rusty spots.

## 2.2. Electrodeposition of CoNiFe alloy

The hexagonal nut made up from mild steel was used as the cathode substrate during the electrodeposition process. During the electrodeposition process, several parameters were identified which included temperature, current density, time deposition and electrolyte composition. Along the process, some parameters were set to be continually referred to the previous study. The sulphate bath composition was heated and kept at a constant temperature which was at 50-60°C. The current and pH of the bath composition were constant at 1.73±0.01 A and 3, respectively. Time taken for the CoNiFe deposition was set to be 30 minutes.

**Table 1:** Composition of sulphate solution using 2 litres distilled water

| Volume | 2L |
|---|---|
| Element | |
| Cobalt sulphate | 28.12 g |
| Nickel sulphate | 70.08 g |
| Iron (II) sulphate | 11.12 g |
| Boric acid | 32.96 g |
| Ascorbic acid | 23.48 g |
| Saccharine | 2.72 g |

The electrolyte bath composed of $CoSO_4$, $NiSO_4$, $FeSO_4$, boric acid, saccharine, and ascorbic acid. Then the preparation of the sulphate could be preceded by using the exact measurement based on the previous study. The chemical was weighed using a digital weighing scale to obtain an accurate amount of compound concerning the desired solution volume as shown in Table 1. Next, the powder chemical compounds were mixed with the respective volume of distilled water which was heated up until 50°C. The electrolyte mixture was stirred using a magnetic stirrer so that all mixtures would dilute entirely in the solution. The temperature increment was monitored to avoid an overheated solution.

## 2.3. Characteriation, hardness testing and wear testing of CoNiFe alloy coating

CoNiFe alloy coating characteristics such as surface morphology, element composition, and thickness of coating were analyzed using the Scanning Electron Microscopy (SEM) HITACHI. This equipment was operated using a high energy beam of electrons to scan the surface of the specimens, a CoNiFe coated Nut and a Non-coated Mild Steel Nut. The observation on the surface area helped to study the morphology of the nanoparticle produced and the uniformity of its composition. Besides that, it was also capable of determining the thickness of the electrodeposited coating on the Mild Steel Nut.

Microhardness testing was done by using Vickers microhardness tester machine, MITUTOYO MVK-H1 to identify the hardness of CoNiFe alloy coating on the mild steel nut compared with the non-coating mild steel nut. Load of 1kg was used for the indentation on the specimen surface for about 15 seconds. Five (5) measurements were carried out at different positions on the specimen and the average microhardness was calculated.

In this research, the wear test was conducted using a Pin-on-Disc Wear Testing Machine (Model: Magnum TE-165-SPOD) with ASTM G99 standard. The samples used for this research were divided into two (2): a CoNiFe coated mild steel and non-coated mild steel. This research was conducted to determine the wear and coefficient of friction of the materials respectively. The testing involved two (2) different parts that were sliding over each other, a pin and a disc. The CoNiFe coated nut and the non-coated nut were decided to be fabricated into a pin with a bar-like shape and slid against a disc (EN31- high carbon alloy steel). Both specimens were tested by using the same parameters as shown in Table 2.

**Table 2:** Constant parameters used for Pin-on-Disc Testing

| Parameter | Values/Description |
|---|---|
| Sample size | 8 x 8 x 25 mm |
| Normal load | 10 N |
| Total distance | 1012 m |
| Time variable | 70 Minutes |
| Test speed | 100 Rpm |
| Wear disc track radius | 23mm |

# 3. Result and Discussion

## 3.1. Characterization analysis

### 3.1.1. Elemental composition

Energy-dispersive X-ray Spectroscopy (EDX) analysis was conducted to determine the elemental composition of CoNiFe nanoparticles compared with non-coated mild steel nut. Fig. 1 shows the average EDX result for the selected area for Mild Steel specimen. EDX results showed that the mild steel surface composition consisted higher percentage of iron (Fe) and Aluminium (Al). On the other hand, Fig. 2 shows CoNiFe coated area with its average of EDX result. EDX results proved that the coating process was successful. It seems that cobalt (Co), Nickel (Ni) and iron (Fe) elements were successfully detected on the selected surface area. This result complied with the previous study done by Resali et al. [1].

Nevertheless, referring to Fig. 2, there was a presence of aluminium (Al) in the coating area which can be interpreted as a void or uncovered surface from the coating. The inconsistency of the coating could happen because of the surface finish of the nut itself which was not flatted and polished. On the contrary, compared to the non-coating specimen in Fig.1, it identified the presence of oxygen (O) on the non-coated specimen only, even though both were fabricated, processed and kept at the same environment. The presence of oxygen in the specimen indicated that the samples were exposed to an oxidation process which had led to rust [5]. Therefore, it was denoted that CoNiFe coating specimen yielded a better corrosion resistance.

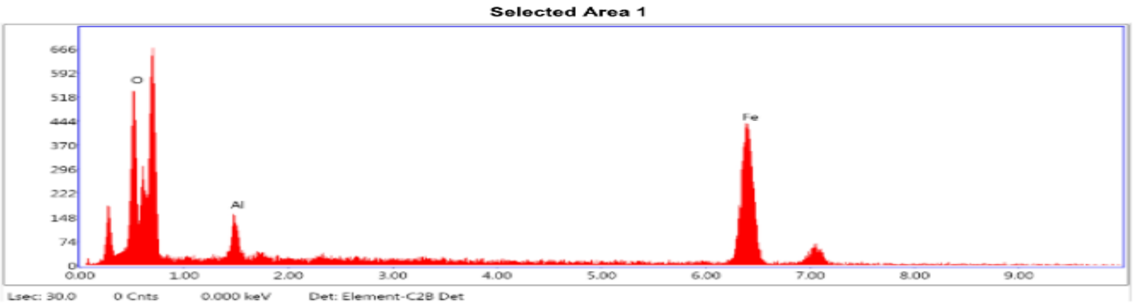


**Fig. 1:** Graph of the EDX pattern for Mild Steel area

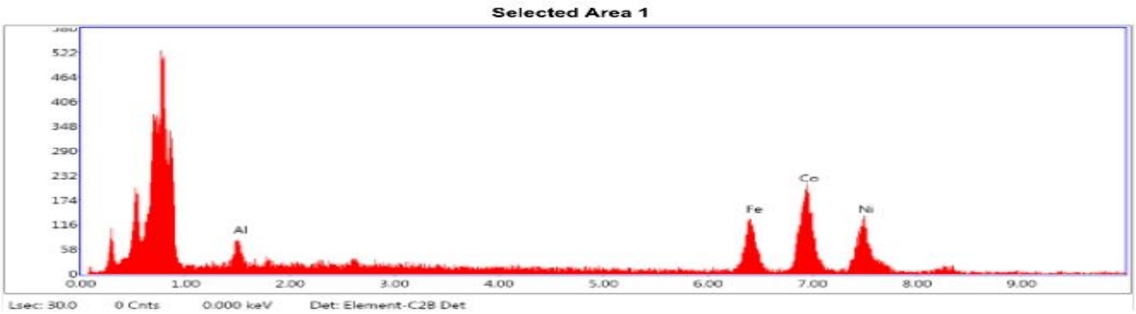


**Fig. 2:** Graph of the EDX pattern for CoNiFe coated area

### 3.1.2. Morphology analysis

The morphology result of nanocrystalline CoNiFe alloy coating was obtained from the SEM micrograph. Fig. 3 shows the SEM image of CoNiFe Alloy Coating using magnification resolution of 100 X with 15kV. The identified surface was not sufficiently smooth, unlike the previous study which had been done by Resali et al. whereby the surface was smooth and full of compact irregular structures. The grains displayed a globular-like pattern with different dimensions, whereby the particles grew preferentially out of the surface. In this case, the structures corresponded to the progressive nucleation and growth mechanisms which were probably due to the un-flat surface of the specimen [6]. As the magnification increased in Fig. 4, the grain nodules with ununiformed size and grain boundary were observed. Inhomogeneous grain size could be caused by higher current density applied throughout the electrodeposition method [7]. Besides that, higher volume of grain boundaries could happen because of the disorientation arrangement and alignment of the nanoparticles [1].

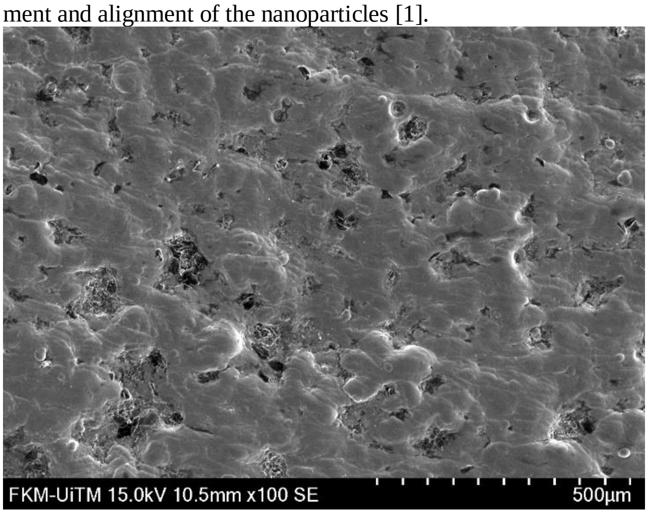


**Fig. 3:** SEM image of CoNiFe Alloy Coating using magnification resolution of 100 X

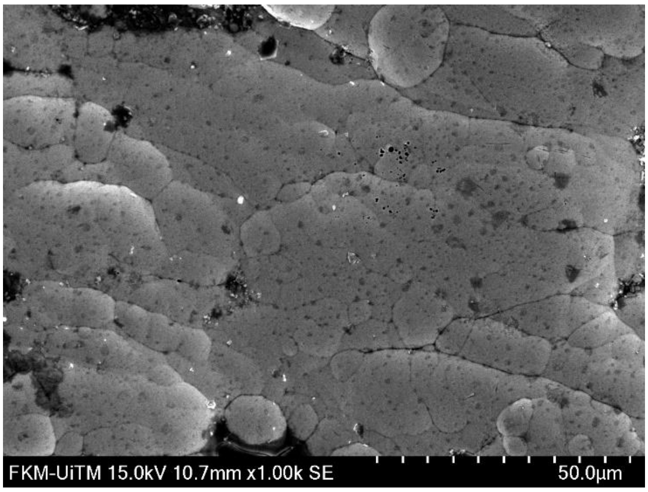


**Fig. 4:** SEM image of CoNiFe Alloy Coating using magnification resolution of 1000 X

### 3.1.3. Thickness analysis

The thickness of the nanocrystalline CoNiFe alloy coating was obtained from the Scanning Electron Microscope (SEM) with the magnification of 450 X. Based on Fig. 5, it seems the fluctuation reading was recorded lowest at 7.64 µm while the highest was at 22.36 µm. Previous study of Koay et al. obtained thickness of about 103 µm with 30 minutes of deposition time [8]. This comparable difference could be caused by different geometry and size of the substrate. This difference was probably due to the error during the electrodeposition process conducted, such as the way the specimen was held during the electrodeposition process. Based on the observation, the coating was thicker at the bottom area and the thickness reduced as it moved upwards.

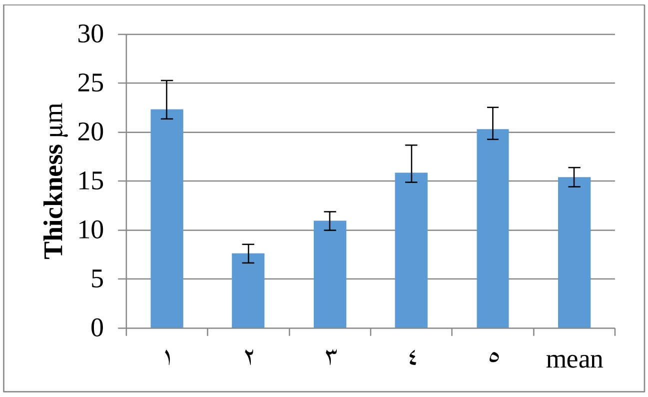


**Fig. 5:** Graph of coating thickness against the location

### 3.2. Hardness analysis

The hardness properties of two samples (CoNiFe Coated nut and Mild Steel Nut) were recorded using the Vickers Microhardness test. Fig. 6 shows a simplified data, and it can be clarified that the hardness of the CoNiFe nanocrystalline coating (317.94 Hv) was higher compared to non-coating specimen (180.60 Hv). The result complied with the research paper of Resali et al. as the hardness for CoNiFe coating was found to be around 333Hv [1]. It is observed in Fig. 4 that there were several grain boundaries in the microstructure. The presence of these grain boundaries resulted in the reduction of particle size which could increase the hardness properties. Furthermore, the existence of these grain boundaries acted as a hindrance to stop the dislocation motion or change the dislocation direction [9]. Consequently, the material became harder and the plastic deformation in the material was prevented due to the restricted motion of dislocation in the nanoparticles [10].

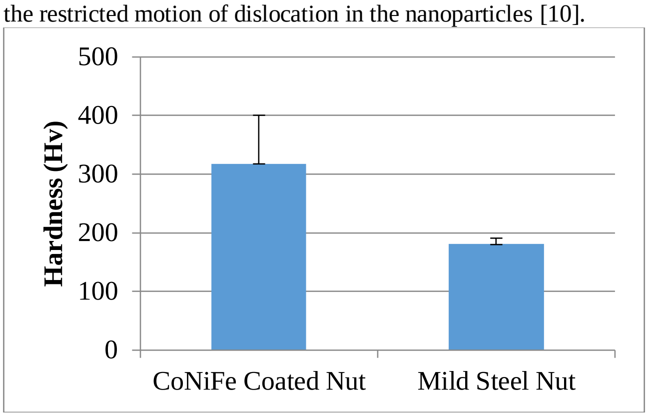


**Fig. 6:** Comparison of average micro hardness between two (2) different specimens

### 3.3. Wear analysis

The Pin-on-Disc wear test was done to two (2) different samples; a CoNiFe coated and Mild Steel sample. The CoNiFe coated sample presents wear rates of 0.0003913mm$^3$/Nm resulting reduction in wear rate of about 13% compared to mild steel sample (0.0004481mm$^3$/Nm). Fig.7 proves that the CoNiFe Coated sample experiences a lower rate of wear compared to the mild steel sample. Correlations between the morphology characteristic and the higher hardness of the coating have resulted in the lower wear rates [11]. CoNiFe coating had conferred to the mild steel nut as a protective layer with a high volume of grain boundaries and high hardness of about 317.94 Hv in which as a result, becomes an excellent wear resistance.

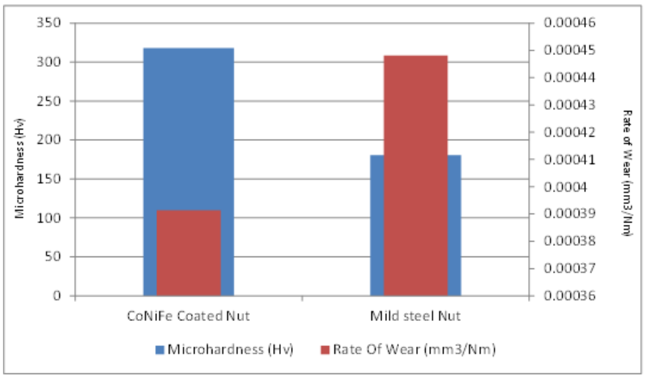


**Fig. 7:** Microhardness and rate of wear for CoNiFe and mild steel nut

## 4. Conclusion

Nanocrystalline CoNiFe alloy coating was a success by using the electrodeposition method. Uneven coating and presence of void can be reduced if the surface of the specimen was polished before going through the electrodeposition and probably, decreasing the total deposition time which may reduce the errors as suggested from the previous study [5]. Nanoparticles coating does increase the total hardness of the specimen. Based on the testing, the coated specimen manages to have a microhardness of 317.94 Hv which has almost doubled up the value of the specimen without the coating. CoNiFe alloy coating exhibits a lower rate of wear, the coefficient of friction and friction force. Implementation of CoNiFe coating manages to yield a better corrosion resistance by relating elemental composition, microhardness and wear resistance. As the conclusion, the objectives of this research are successfully achieved as the railway fastener is well coated by CoNiFe alloy and the characterization, hardness and wear properties of the coating are successfully determined.

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