

## Corrosion study of heat treated nanocrystalline CoNiFe coating exposed in sodium solution

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**ABSTRACT** – Repair of corrosion damaged equipment is the largest maintenance requirement for the industry. Recently, with the development of alternative material such as electrodeposited CoNiFe, these problems have largely been overcome. In this study, nanocrystalline CoNiFe with particle size in the range of 26.3-49.3nm are being developed to enhance the corrosion properties. It is believed that the heat treatment applied on the electrodeposited CoNiFe produces better coating with less porosity. Hence, this paper focused on the comparison between the electrodeposited CoNiFe with and without heat treatment. The findings showed that the electrodeposited CoNiFe exhibit the enhancement in corrosion resistance after heat treatment. However, the heat treated CoNiFe using mixing gases (Argon and Hydrogen) revealed higher oxygen content at 5.34 wt% and thus contributed to the highest rate to corrode.

### 1. INTRODUCTION

Heat treatment of structural material is one of the ways of improving its resistance to corrosion. [1]. During the heat treatment process, the material usually undergoes phase microstructural and crystallographic changes and this cause an effect on the corrosion properties of the material [2]. In this paper, the nanocrystalline CoNiFe with particles size of less than 100 nm was synthesised by electrodeposition method. The as-synthesised CoNiFe was heat treated to produce a great variety of microstructures. The corrosion properties of the electrodeposited CoNiFe were studied to determine the effect of sodium solution on the heat treated nanocrystalline samples. The present work has been focused to compare the corrosion behavior of nanocrystalline CoNiFe in different conditions; as-synthesised CoNiFe, heat treated CoNiFe using argon gas and mixing gases.

### 2. METHODOLOGY

The preparation of CoNiFe coating was achieved by mixing the electrolyte solution that contained CoSO<sub>4</sub>, NiSO<sub>4</sub>, FeSO<sub>4</sub>, H<sub>3</sub>BO<sub>3</sub>, ascorbic acid and saccharin. The deposition was performed at constant current density of 0.143 A/cm<sup>2</sup>. The experiment was carried out in pH medium of 1.

A Vacuum Tube Furnace; model HTF-15/300-50 was used for heating purpose. In this study, there were three different conditions to be compared. First sample

was the as-synthesised CoNiFe coating. There were two types of gases used in the heating process; which were 100% argon and mixing gases containing 95% argon + 5% hydrogen gas.

A corrosion test in the artificial atmosphere which termed as salt spray test was carried out in a salt spray chamber following ASTM B117-85. Sodium chloride solution was used in this test to immerse the whole coating products for 24 hours. The crystalline structure of the sample was studied by X-ray diffractometer (XRD, Ultima IV, FD3668N). Surface micrographs of the corroded samples were taken using a camera. Average particles size was measured using the Transmission Electron Microscopy (TEM) equipment. An alloy composition analysis was performed using OXFORD INSTRUMENTS EDX test with INCA software. Further corrosion testing was performed on nanocrystalline CoNiFe using potentiodynamic polarization test.

### 3. RESULTS AND DISCUSSION

#### 3.1 Structural Analysis

The phases detected in the entire XRD patterns were FeNi and CoNi [3,4] before and after heat treatment. It can be concluded that no phase was changed after the heat treatment process.

The XRD and EDX analysis of the samples exposed to 24 hours salt spray test have shown similar results where there is no corrosion products have been found on the coating surface. Hence, there is no chemical reaction happen during the immersion of electrodeposited CoNiFe in sodium chloride solution. Besides, the oxygen content in the as-synthesised sample revealed as 3.13wt%. On the other hand, the sample heated in an inert argon atmosphere (2.01wt%) produced less oxygen content if compared to heating in the mixing gases atmosphere (5.34wt%).

From the XRD results, the crystallite size of the as-synthesised sample, heat treated sample using argon gas and mixing gases are 12, 23 and 28nm, respectively. It shows that the crystallite size is increased after the heat treatment process. This finding was further confirmed by average particle size (in the 26.3-49.3nm range) obtained from the TEM results. Figure 1 shows the TEM image for as-synthesised electrodeposited CoNiFe.

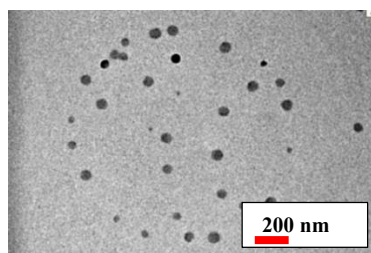


Figure 1 Transmission electron microscopy (TEM) image of as-synthesised electrodeposited CoNiFe.

### 3.2 Surface Micrograph Characterization

The purpose of this testing is to observe the corroded surface after exposure to the Sodium Chloride solution for 24 hours. Surprisingly, there are similar micrographs without any changes surface of nanocrystalline CoNiFe even after exposed in 24 hours of Sodium chloride solution. There is no corrosion products was visible for all samples. It can be summarised that the nanocrystalline CoNiFe coatings have performed well in three different conditions and protected the substrate material.

### 3.3 Surface Roughness

It is believed that the oxygen content in the coating surface helped in the voids formation [5]. Fig. 2 presents the graph of surface roughness of nanocrystalline CoNiFe in different heating conditions. The samples heated using mixing gases produced the highest oxygen content during the heat treatment process. The voids on the surface morphology formed due to the oxygen presence and thus produced the highest roughness.

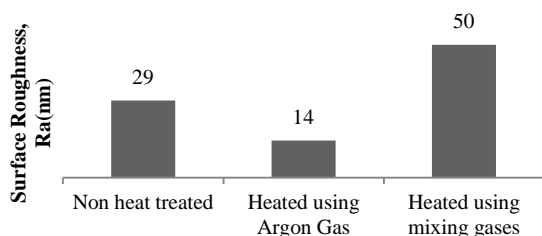


Figure 2 Surface roughness values for the nanocrystalline CoNiFe at different heating conditions.

### 3.4 Potentiodynamic Polarization

The corrosion rate for nanocrystalline CoNiFe heated using argon gas (0.0015mmpy) revealed the lower value compared to other conditions. This may be due to the less weight percent of oxygen element was found in this sample which helps in lowering the corrosion rate. In water containing high salt concentrations, corrosion is proportional to the amount of oxygen dissolved in the water [6]. The corrosion rate is increased as the solubility of oxygen increases. Hence, the oxygen content in the sample influences the corrosion rate. Anyway, the corrosion rate for all samples still perform better corrosion resistance compared to steel, which has corrosion rate of about 0.3 mmpy in pH range of 4 to 10 [7].

Besides, the crystallite size is believed to affect the

corrosion properties of coatings. The decrement of corrosion rate is affected by the crystallite size reduction [8]. The electrodeposited CoNiFe heated by using argon gas revealed the lower corrosion rate compared to mixing gases due to the smaller particle size.

## 4. CONCLUSION

In this work, electrodeposited CoNiFe exhibited the good corrosion resistance after the heat treatment process. It was found that the corrosion rate was slower when the particle sizes were smaller. Besides, lower oxygen content found in the nanocrystalline CoNiFe contributed to the lower surface roughness due to less void formation. The low oxygen content also resulted in a lower corrosion rate as the tendency of oxidation has been minimized. Therefore, it can be concluded that the heat treated nanocrystalline CoNiFe coating under argon gas atmosphere provided a protection to the surface of electrodeposited CoNiFe from being attacked by the sodium solution and thus was the least tendency to be corroded.

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