

Effect of Electro-Co-Deposition Parameters on Corrosion Behavior of Ni-WC Nano Composite Coatings

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Abstract - Influence of electrocodeposition process parameters such as current density, solution temperature and reinforcement loading on the corrosion behavior of Ni-WC coatings under 3.5% of NaCl solution were studied. Coatings are porous free, continuous and uniform thickness on the MS substrate in all conditions were considered. The coatings were characterised using salt spray test under the room temperature condition. The morphology of before and after corrosion studies was investigated using XRD, EDS and scanning electron microscope. The corrosion results revealed that the higher influence of WC could be seen and other parameters such as current and solution temperature showed its influence on the corrosion properties.

Keywords — *Electro-co-deposition; Ni-WC composite coating; SEM; Corrosion resistance.*

I. INTRODUCTION

The composite electro-co-deposition could be a technique to combine nano particles into metal matrix by electro-co-deposition. The coatings eventually have the benefits of each the metal matrix and therefore the reinforces. Nickel electro-co-deposition is outstanding among the coating techniques and is extensively utilized in many industries. This being the reason, in recent years, electro-co-deposition with nickel composite coating has been one of the research interests. Many researchers have reported on electro-co-deposition of SiO₂, Al₂O₃, ZrO₂, TiO₂, SiC [1, 2, 3-5, 8]. Researches showed that the composites carry higher hardness value, superior wear resistance and corrosion resistance than pure nickel coating. Carbon nanotubes (CNTs) ideal reinforces for composites as a result of this high young's modulus and better tensile strength [4]. The addition of CNTs increases the hardness value, wear resistance and corrosion resistance showed in similar article [6,7]. In present research, the effects

of the coating parameters throughout electro-co-deposition on properties of Ni-WC nano composite coatings were studied.

II. EXPERIMENTAL METHOD

Ni and composite coating were electro-co-deposited on MS substrate from nickel sulfamate type bath containing 250 gm/l Nickel Sulphate, 35 gm/l Nickel Chloride, 40 gm/l Boric acid, pH 4 and reinforcement nano particle WC of size 200-300 nm. The electro-co-deposition solutions were used by using analytical graded chemicals and distilled water. For one hour bath ultrasonication is used for the solution, such that all the reinforcement WC particles adequately moistened and uniformly remains same in the solution. Coated specimens were prepared as per ISO 6892-1998 for mechanical, microstructural and corrosion properties.

The mild steel specimen were polished mechanically and cleaned with detergent solution and acetone and then rinsed with a 5% sulfuric acid at room temperature for three minutes and rinsed with deionized water for 5 minutes. Bath solution were subjected to magnetic stirring at 250 rpm and bath current density (2, 3 & 4 A/dm²), bath temperature (45 °C, 55 °C & 65 °C) were set to desired value. Electroplating was done for 1 hour. In order to remove the absorbed composite from the surface of the coating specimen and washed with deionized water.

The distributions of particle size of particles were characterized by using JEOL JSM-5600LV scanning electron microscope (SEM) equipped with an EDAX (Energy Dispersive X-ray spectroscopy) detector. Crystallinity of the Ni-WC were characterized by using X-ray diffraction (XRD) Maxima-7000, Shimadzu. Elemental composition of reinforcement particles were characterized by using energy

dispersive X-ray spectroscopy (EDAX) and the amount of reinforcement particles and Ni-deposited were determined based on the mean value; five measurements for each deposition were taken. By using Elektro Physik thickness gauge, coating thickness of the substrate Ni-WC was measured. Micro hardness measurements were carried out by using Vickers microhardness (HV in kgf/mm²). Pure nickel and Ni-WC based nano composite coating deposition were performed by using Reichert microhardness tester. Commonly applied accelerated corrosion test were performed by using ASTM standard B-117. Fine-fog salt solution was used for coated specimens were exposed to (3.5% NaCl) 35°C temperature and percentage of the specimens of coating surface covered in red rust was measured.

III. RESULT AND DISCUSSION

Fig 1(a) shows SEM micrographs presenting the typical morphology of Ni crystallites of pure nickel deposits showing clear and homogeneous, compact and without visible dendrites. Compared to Ni alloying coating, the Ni/WC nano composite coating shows granular-like structure, which shows the co-deposited WC nano-particulates were uniformly distributed in Ni composite coating as shown in *Fig. 1 (b-d)*. It is assumed that the co-deposited WC particulate has a uniform distribution and agglomeration to some amount it may contribute to increase in hardness value of Ni-WC composite coatings.

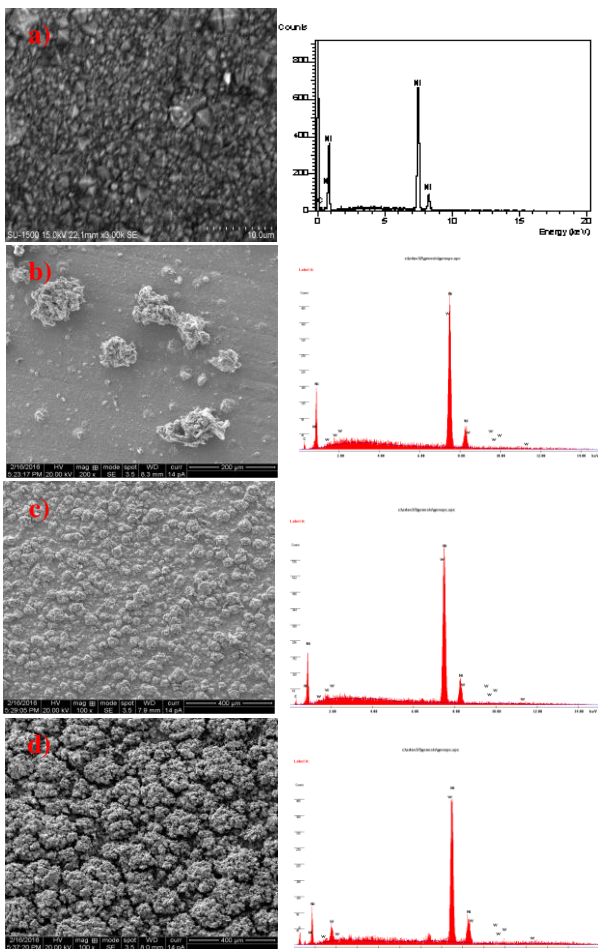


Fig 1: Coating morphology of a) Pure Ni, b) Ni-WC 2gm/l, c) Ni-WC 4gm/l, d) Ni-WC 6gm/l nano composite coatings & respective typical graphs of EDXA signature.

EDAX, results showed that the loading effect on the wt. % of reinforcement in the composite coating as shown in *Fig.1*. Current density and bath temperature were increased with increase in WC wt. % in the reinforcement coating.

Fig. 2 shows the XRD peaks of electro-co-deposited Ni and Ni-WC nano composite coatings. XRD patterns confirm the presence of WC in the Ni-Composite crystalline matrix during electro-co-deposition. The pure Ni peak was obtained at $2\theta = 45^\circ, 52^\circ$ and 76° . The average crystal size of the coating was 43, 10.3, 9.1 and 8.2 μm for Ni, Ni-WC composite coatings. At higher rate of formation of nuclei fine-grained depositions were obtained.

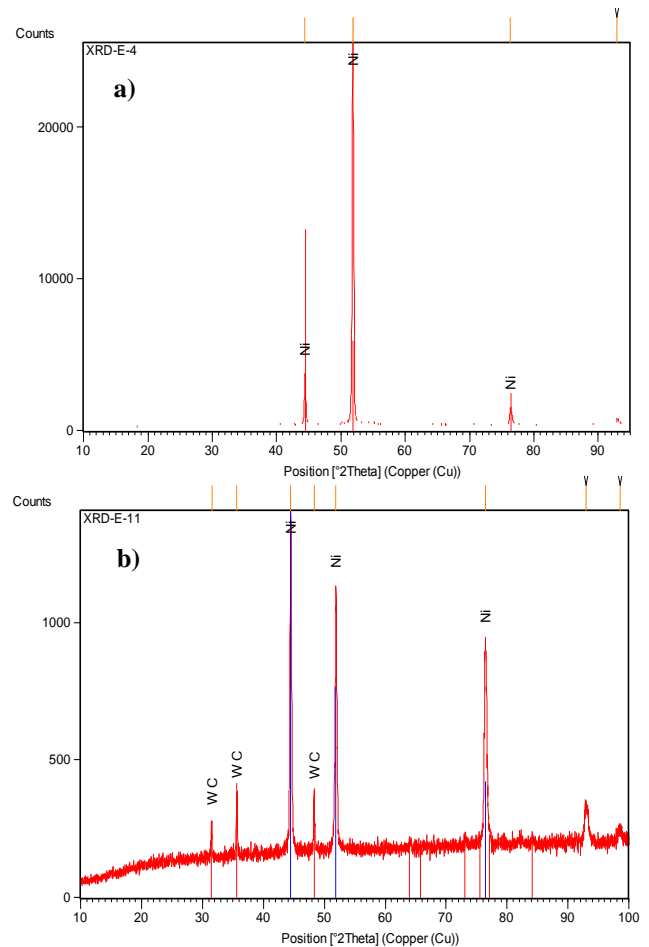


Fig. 2: A typical graph of XRD signature of a) Pure Ni, b) Ni-WC nano composite coatings on MS substrate.

A typical cross sectional view of a Ni-WC nano composite coating samples is presented in *Fig. 3* (200 and 100 μm magnification). The coating represents homogeneous distribution of the Ni-WC composite coating that leads to more stronger mechanical property.

Fig 4 (a-c) shows the hardness Ni-WC nano composite coatings as a function of the bath temperature. The hardness of the as-deposited Ni-WC coating was approximately 1244 kg/ mm², which is approximately 50% greater than pure Ni coating. Furthermore, Increase in current density, bath temperature and WC-loading hardness value of Ni-WC coating decreases. Current density increased whereas the

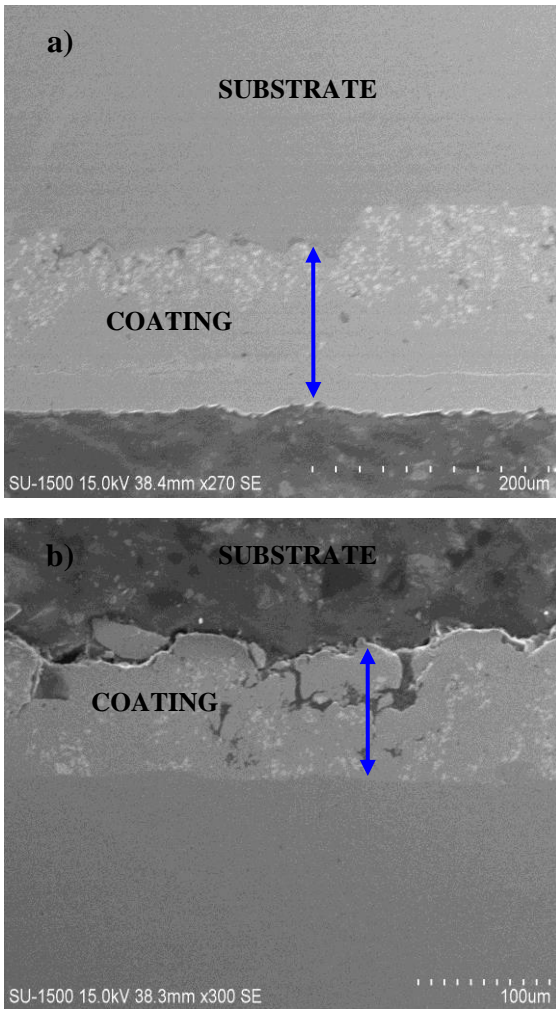


Fig. 3: Cross-section of SEM micrographs of a Ni-WC coated on MS substrates, after cutting and polishing (a) 200µm & (b) 100µm

microhardness of the coating showed mixed trends. Initially it increases then decreases with bath temperature and loading irrespective of the current density. In the first phase, the hardness raise with increasing temperature due the higher concentration of WC. Although higher concentration of Ni-WC is higher more porosity can be seen in the coating hence the microhardness decreases after 55°C temperature. Hydrogen bubbles pushes the electrolyte away from the deposition zone, possibly stopping the electroplating and hindering the electrolytic processes and prone to more porosity in the coating surface.

Fig. 5 & 6 (a-c) showed corrosion test by the lost weight method (NaCl 3.5%) and ambient temperature shows initially corrosion rate decreases with increase in WC [9]. Because of this it leads to higher surface corrosion.

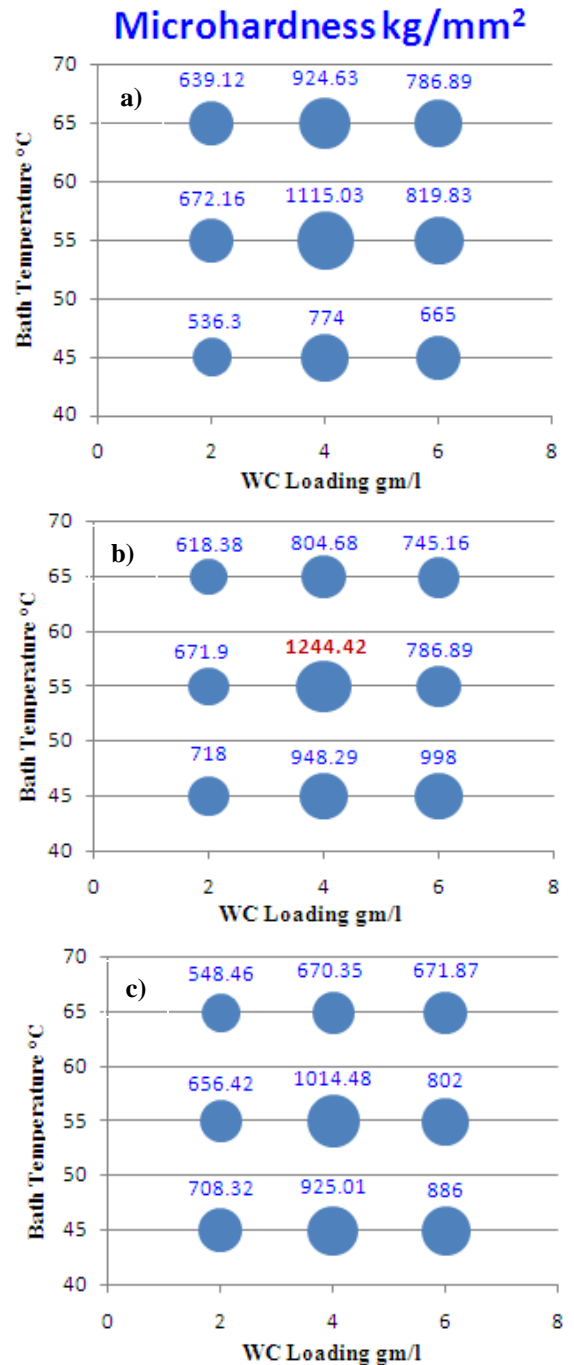


Fig 4: Effect of Bath temperature and WC loading on Microhardness for Ni-WC coatings of WC loading a) 2 gm/l, b) 4 gm/l & c) 6 gm/l

From the experiments it shows that the composites loading are having less corrosion resistant than pure nickel and lower loading composites. After deposition of 4 gm/l the WC content increases and its corrosion resistance decreases [10].

Subsequent to the quantity of particles in the bulk of the coatings, a constant appropriation of the erosion current

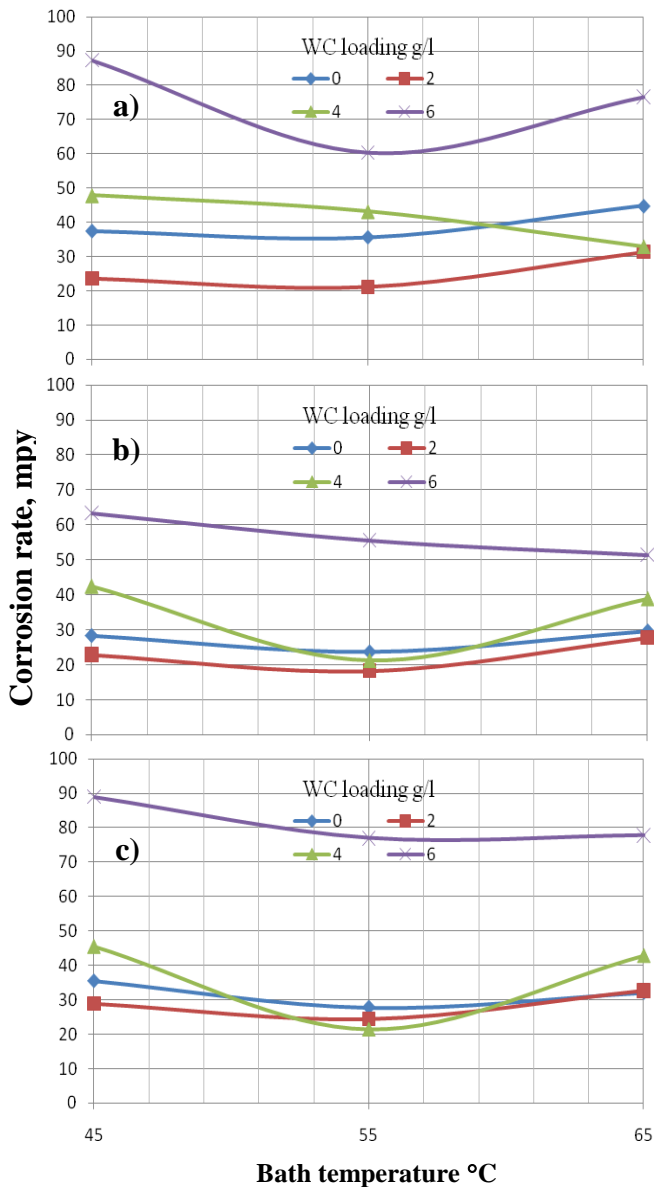


Fig 5: Corrosion rate as function of bath temperature for Ni-WC composite coating at WC Loading of a) 2 gm/l, b) 4 gm/l and c) 6 gm/l

happens on the anodic boundary. Incorporations might go about as ideal locales for fissure corrosion and speed. Particles might get to be ousted amid disintegration, presenting more range to corrosion and it confirmed by the corrosion potential showing a positive movement. For the vast majority of the cases the Nyquist plots are half circle showing the enactment control of the consumption response.

Addition of WC is useful to prevent the corrosion resistance of the composite coatings. Influence of WC on corrosion resistance of the composite coatings could hold two opposite aspects. Potential of carbon is a lot of conclusive than that of nickel in solutions potential difference between WC,

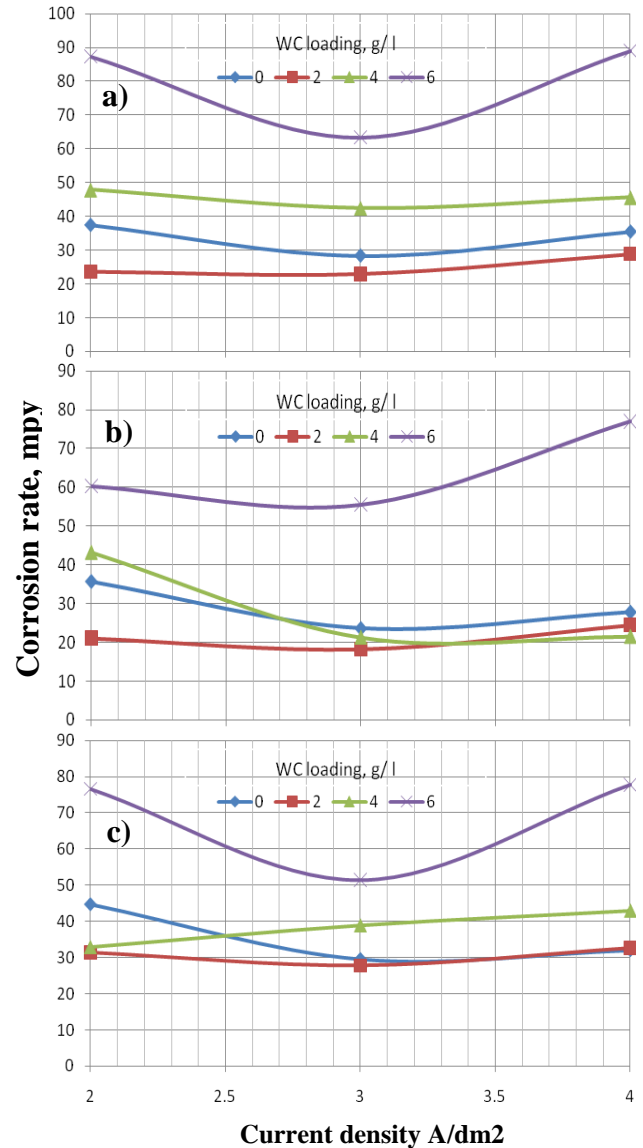


Fig 6: Corrosion rate as function of current density for Ni-WC composite coating at bath temperature of a) 45 °C, b) 55 °C and c) 65 °C

neighboring Ni could stimulate corrosion of Ni matrix. In addition, to increased conductivity and boundary areas between WC and nickel matrix are also disadvantageous to corrosion resistance of the coating. Potential difference between WC and Ni may also promote Ni and Pb to increase in corrosion resistance. In this condition, the latter effect played a more important role, which is confirmed by increasing the coating resistance R_p of the nano composite coating.

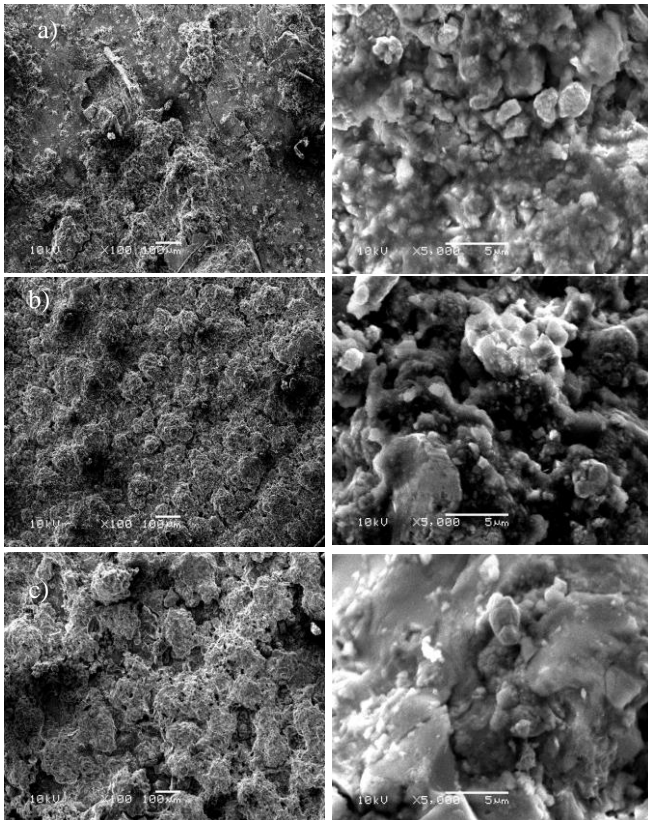


Fig 7: SEM images for the three samples after weight loss test in 3.5 wt % NaCl solution for 100 hours of a) Ni-WC 2gm/l, b) Ni-WC 4gm/l & c) Ni-WC 6gm/l

The coating deposition under the current density of 2 A/cm^2 showed greater R_p value than the coating obtained at 3 A/cm^2 , while the WC content in the latter was higher based on the result in **Fig. 5 & 6**.

As results shows the change in corrosion behavior of the nano composited coating with the addition of WC particles. Through electro-co-deposition of Ni, particle sizes of Ni deposition decreased with the increase of current density [11], it is helpful to corrosion resistance of the coatings.

From **Fig.7 (a-c)** shows the morphology of coating were shown after weight-loss test in 3.5 wt. % NaCl solution for hundred hours for specimens, the surface indicates presence of corrosion.

Presence of cracks and flakes reveals the degree of corrosion was higher in Ni-WC 2 g/l composites coatings on the surface of Ni-coating.

Ni-WC 4 g/l composites showed that shallow & pits, results indicated that the Ni-WC composite coating had superior resistance to corrosion, results obtained by weight loss and electrochemical measurement.

SEM micrographs of Ni-WC composite-coated specimens exhibits corrosion on the surface, but composite-coated specimens exhibits lesser corrosion. These results confirmed that the composite coated specimens maintained its original surface even after fifteen days of immersion in NaCl and retains superior corrosion resistance property.

IV. CONCLUSION

Corrosion rate were decreased in nano composite coated specimens. SEM micrographs show that the corrosion rate is lower in coating surface. By using the lost weight method it shows corrosion rate decreased with Ni-WC composite coatings. Ni-WC composite coating experimental studies revealed the optimum mechanical properties and corrosion resistance were obtained at 4 g/l WC, 55°C bath temperature and current density of 3 A/dm^2 .

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