



### **Science Arts & Métiers (SAM)**

is an open access repository that collects the work of Arts et Métiers Institute of Technology researchers and makes it freely available over the web where possible.

This is an author-deposited version published in: <https://sam.ensam.eu>  
Handle ID: [.http://hdl.handle.net/10985/10090](http://hdl.handle.net/10985/10090)

#### **To cite this version :**

Hachemi Ben TEMAM, Larbi ZEROUAL, Abdelouahad CHALA, Saad RAHMANE, Corinne NOUVEAU - Microhardness and Corrosion Behavior of Ni-SiC Electrodeposited Coatings - Plasma Processes and Polymers - Vol. 4, p.618-621 - 2007

Any correspondence concerning this service should be sent to the repository

Administrator : [scienceouverte@ensam.eu](mailto:scienceouverte@ensam.eu)



# Microhardness and Corrosion Behavior of Ni-SiC Electrodeposited Coatings

Hachemi Ben Temam, Larbi Zeroual, Abdelouahad Chala,\* Saad Rahmane, Corinne Nouveau

Composite coatings suitable for protection against corrosion were prepared by electrodeposition of chloride-nickel coating containing silicon carbide particles maintained in the suspension. The Ni-SiC composite coatings showed a better corrosion resistance in 0.6 M NaCl solution and high hardness than nickel, electrodeposited under the same conditions. The coatings deposited were uniform and adherent to the substrates. X-Ray diffraction (XRD) studies showed that the nickel coatings grow with (111) preferred orientation.

## Introduction

A new family of electrodeposited coatings containing a dispersion of second phase particles has been developed in recent years to combine some of the desirable properties of the metals with that of solid particles. Such particles can be hard oxide or carbide particles like SiC, Al<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub>, TiO<sub>2</sub>, WC, or diamond, solid lubricants like polytetrafluoroethylene (PTFE), graphite or MoS<sub>2</sub>, or even liquid-containing microcapsules<sup>[1]</sup> to improve corrosion or wear resistance and/or to reduce the friction.

Electrodeposited composite coatings containing micron-sized particles are used as corrosion-resistance coatings<sup>[2,3]</sup>, e.g., nickel-SiC in car engines.<sup>[4-7]</sup> With the increasing availability of nanoparticles, the interest for electrolytic and electroless composite coatings containing nanoparticles is growing.<sup>[8]</sup> Recent advances in electrochemical processing of materials and the fabrication of nanostructured materials have begun to establish electrodeposition as a valuable technique for thin film deposition. According to the literature, the rate of particle entrapment

depends on many factors related either to the particles (size, density, composition, zeta potential, conductivity) or to the electrolytic solution (composition, pH, temperature, the presence of additives). Agitation of the bath appears to be an important factor but its effects are somewhat ambiguous. In most of the cases, particle sizes are larger than 0.1  $\mu\text{m}$  and, therefore, vigorous stirring of the electrolyte is necessary to obtain a homogeneous suspension.<sup>[9]</sup> Electrodeposition has a number of advantages over other physical deposition techniques since it is in low cost and can be used for many practical applications. Corrosion and wear resistance of mechanical parts are a major concerns and the common way of increasing wear resistance is the co-deposition of hard particles on a metallic matrix, i.e., Ni-SiC composites.<sup>[10]</sup> However, in many cases the mechanical components may work in severe conditions (environment containing water or aggressive compounds) regarding not only wear, but also concerning corrosion resistance. In these cases, wear, hardness, and corrosion resistance can play an important role in the durability of metallic components. In this work, electrolytic co-deposition of micron and submicron SiC particles form a chloride nickel solution in order to develop electrodeposition of Ni-SiC composite coatings to achieve high hardness and corrosion resistance.<sup>[11]</sup>

## Experimental Part

A three-electrode cell was utilized to carry out electrochemical experiments. The volume of the electrodeposition bath was

H. B. Temam, A. Chala, S. Rahmane  
Laboratoire de Chimie Appliquée, Université de Biskra, BP 145 RP,  
07000 Biskra, Algeria  
Fax: (+213) 33 73 32 05; E-mail: ab\_chala@yahoo.fr  
L. Zeroual  
Département de Génie des procédés, Université de Sétif, 19000  
Sétif, Algeria  
C. Nouveau  
LaBoMaP, ENSAM-Cluny, Rue Porte de Paris, 71250 Cluny, France

approximately 200 ml. The Ni-SiC films were electrodeposited onto chrome-vanadium stainless steel foils and were used as the cathode with an exposed area of 4 cm<sup>2</sup>. The substrates were mechanically polished, degreased in alkaline solution and treated in HCl (10% by volume), prior to deposition. The anode used in all the experiments was pure nickel (99.99%). Electrodeposition was carried out at 45 °C temperature from a solution containing NiCl<sub>2</sub> · 6H<sub>2</sub>O 0.1 mol · l<sup>-1</sup>, NH<sub>4</sub>Cl 0.4 mol · l<sup>-1</sup>, NaCl 0.1 mol · l<sup>-1</sup>, and boric acid 0.3 mol · l<sup>-1</sup>. The pH of the solution was regulated to 3.5. The current density used in all the experiments was 2 A · dm<sup>-2</sup> and the time of co-deposition was 60 min. We added different quantities of SiC and stirred for 48 h in order to ensure a good dispersion. The solution pH was observed to increase slightly with the addition of the SiC particles and was adjusted by the addition of the aqueous HCl or NaOH solution.

The morphology and the microstructure of the coatings were analyzed using scanning electron microscopy (SEM) and X-ray diffraction (XRD) with Bragg–Brentano configuration using CoK $\alpha$  X-ray. The coating's hardness was measured using microhardness Vickers. To study the corrosion behavior of electrodeposited films, we used the lost weight of Ni-SiC alloy specimen (cored area is 2.5 cm<sup>2</sup>) immersed in 0.6 M NaCl solution for 30 days.

## Results and Discussion

Many authors<sup>[1,2,4–7,12]</sup> think that the variation of electrolyzing parameters (current density, bath pH, granulometry of particles, particles concentration, and temperature) can improve the quality of coatings. In the same way, and in order to reduce the costs, which tends to occupy the second place after the quality of deposition, we tried to find the optimum conditions that can satisfy this objective. After the realization of some coatings with different current densities (1–10 A · dm<sup>-2</sup>), we found that the better results were obtained for current densities between 1.20 and 2.0 A · dm<sup>-2</sup>. It was verified that the quality of coating (adherence, efficiency, brightness, thickness, and corrosion behavior) is better in this range of current densities. Beyond this interval, the coatings become dendritic and their adherence to substrates becomes very bad. Generally, the number of crystallites increases with increment in current density. The increase in temperature decreases the voltage and increases the formation speed of the crystals, which can be explained by dendrite deposition. The best coatings are obtained between 40 and 48 °C. The electrodeposition of the film of Ni-SiC is obtained with a very good cathodic efficiency, which increases with the addition of solid particles. The good adherence obtained in our coatings was confirmed by the test of thermic shock.

The metallic structure of our films is shown in Figure 1. We can easily see that it is homogeneous and compact and without any dendrites. Figure 1(b) and 1(c) show that the coatings obtained by the addition of SiC present a high

degree of co-deposition. The surface is covered by solid particles incorporated in the matrix of pure nickel. We also note that the co-deposition degree is improved by increasing the quantity of solid particles, which is in good agreement with literature.<sup>[11,13]</sup>

The thickness of different films determined by optical microscopy increases on increasing the quantity solid particles (SiC) added to the solution [Figure 2(a)]. Figure 2(b) shows that coatings prepared in selected bath present good hardness. We can also see that the addition of solid particles (SiC) in the nickel matrix increases the hardness. This is essentially due to the heterogeneity of

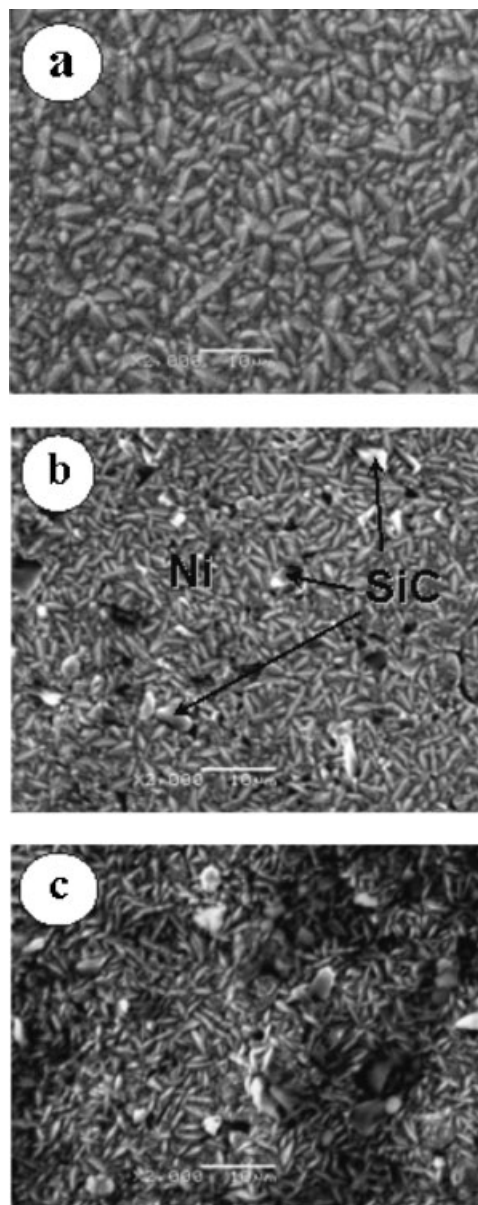


Figure 1. SEM images of films of pure nickel (a), nickel +10 g · l<sup>-1</sup> SiC (b), and nickel +20 g · l<sup>-1</sup> SiC (c).

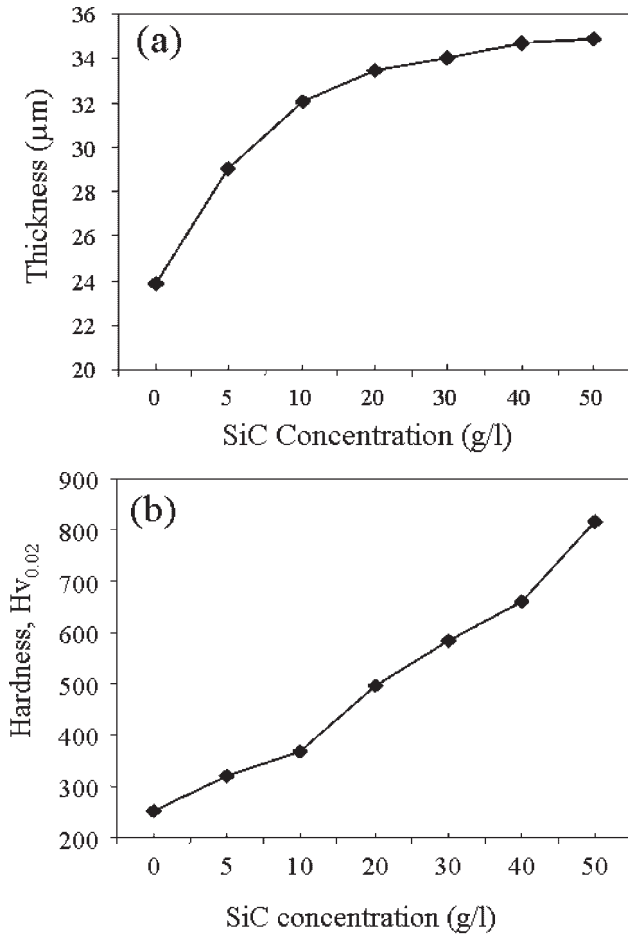


Figure 2. Variation of electrodeposited film's thickness (a) and microhardness (b) with SiC concentration.

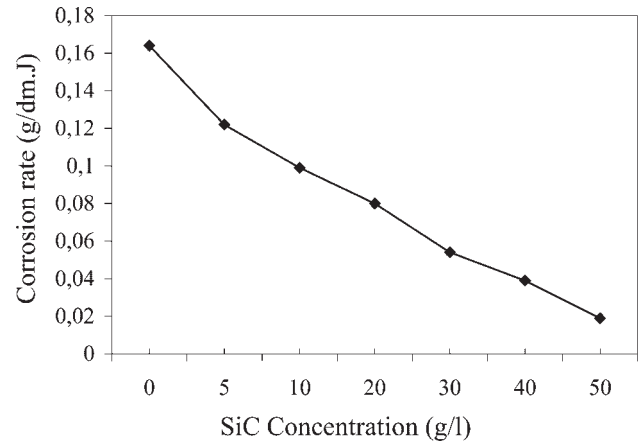


Figure 3. Variation of corrosion rate with SiC concentration.

coatings and it improves, consequently, the mechanical properties of films.<sup>[10,11]</sup>

The corrosion tests carried out with the lost weight method (Figure 3) and examined in an aggressive environment (NaCl 3.5%) for 30 days at an ambient temperature show that the corrosion rate decreases with increasing the quantity of silicon carbide. We can say that the incorporation of solid particles in the metallic matrix makes the films to resist to corrosion phenomena better, which is in good agreement with literature.<sup>[12,13]</sup>

XRD (Figure 4) shows that all the films present good, crystalline, and simple cubic structure of nickel matrix. The preferred orientations of pure nickel are (111), (200), and (311). A light shift of these peaks' positions is observed

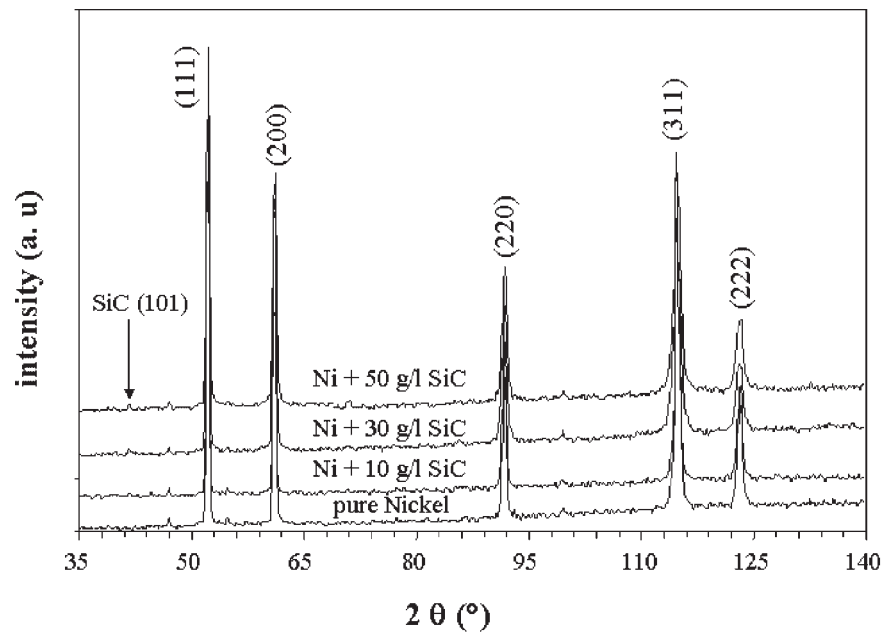


Figure 4. XRD patterns as a function of SiC concentration.

(2–3°) and we think that it is due to the insertion of SiC particles. The orientation of the peak (222) becomes slight when increasing the SiC concentration. This is probably due to the compact structure resulted by the insertion of SiC solid particles. Because of their very little quantity and very small size (0.1–0.3 μm), the SiC particles are not observed by XRD analyses. We need to use a specific method of characterization like XPS or EDX to confirm their existence. However, SiC (101) peak with very small intensity can be detected at 40.86°.

## Conclusion

The elaboration of nickel and composite films of Ni-SiC by electrolytic method onto steel substrates has been realized successfully in our laboratory. The present study shows that SiC particles can be co-deposited with nickel to improve the mechanical and the corrosion behavior of steel substrates.

The morphology of different films shows that the structure is homogeneous and compact. A high co-deposition degree is obtained when optimum conditions of elaboration are applied and the surface is covered by SiC particles.

The thermal shock test proves that films prepared with the optimum conditions present very good adherence to substrates. The determination of electrodeposited film's thickness indicates that it increases gradually with the addition of solid particles.

The corrosion tests realized using the lost weight method show that the corrosion rate decreases with increasing SiC concentration which is in a good concordance with literature.

The microhardness measurements indicate that all the films have good mechanical properties. The microhardness increases with the addition of silicon carbide particles in nickel matrix which is due to the heterogeneity of coatings.

The XRD indicates good nickel crystalline structure with (111), (200), and (311) preferred orientations.

Received: September 3, 2006; Revised: November 29, 2006; Accepted: December 21, 2006; DOI: 10.1002/ppap.200731417

Keywords: corrosion; electrodeposition; microhardness; Ni-SiC composite

- [1] S. Alexandridou, C. Kiparissides, J. Fransaer, J. P. Celis, *Surf. Coat. Technol.* **1995**, *71*, 267.
- [2] J. Zahavi, J. Hazan, *Plat. Surf. Finish.* **1983**, *2*, 57.
- [3] L. Jun, W. Yiyong, W. Dianlong, H. Xinguo, *J. Mater. Sci.* **2000**, *35*, 1751.
- [4] J. R. Roos, J. P. Celis, J. Fransaer, C. Buelens, *J. Metals* **1990**, *42*, 60.
- [5] M. Ghouse, *Metal. Finish.* **1984**, *3*, 33.
- [6] X. M. Ding, N. Merk, *Scripta Mater.* **1997**, *37*, 685.
- [7] D. H. Cheng, W. Y. Xu, L. Q. Hua, Z. Y. Zhang, X. J. Wan, *Plat. Surf. Finish.* **1998**, *2*, 61.
- [8] Y. V. Timoshkov, L. S. Molchan, S. V. Labunov, V. I. Kurmashev, T. M. Gubarevich, J. Fransaer, J. P. Celis, *Surf. Modif. Technol.*, **1998**, 991.
- [9] G. Maurin, A. Lavanant, *J. Appl. Electrochem.* **1995**, *25*, 1113.
- [10] C. Müller, M. Sarret, M. Benballa, *Surf. Coat. Technol.* **2002**, *162*, 49.
- [11] Y. S. Huang, X. T. Zeng, I. Annergren, F. M. Liu, *Surf. Coat. Technol.* **2003**, *167*, 207.
- [12] J. P. Celis, J. Fransaer, J. R. Roos, *Mater. Tech.* **1995**, *1–2*, 5.
- [13] I. Garcia, A. Conde, G. Langelaan, J. Fransaer, J. Celis, *Corros. Sci.* **2003**, *45*, 1173.