Electrodeposition of Nickel-Polytetrafluoroethylene (PTFE) Polymer Composites

By G.N.K. Ramesh Bapu and S. Mohan

A nickel-fluoropolymer composite was produced by a technique using a Watts nickel bath containing PTFE in suspension. The effects of the PTFE concentration, the current density, the pH and the temperature on the volume percent of PTFE incorporation in the deposit were investigated. It was found that the PTFE content increased with PTFE concentration and current density. An optimum PTFE content (14.6 vol. percent) was possible by operating the bath at 6 A/dm² at pH 3.0 and 50 °C.

Fluoropolymers such as polytetrafluoroethylene (PTFE) and poly carbon monofluoride (CF), have a unique repellent property for water and oils. These compounds are used for coatings with non-stick characteristics, low coefficient of friction and reduced wear as self-lubricants.^{1,2} Low hardness of the PTFE compounds makes the coating wear rapidly. Ni-PTFE electrocomposites can overcome this problem and are attractive. PTFE-incorporated bronze bearings have been developed for many industrial applications and the composite is being used in the preparation of molds for the release of plastics and rubber without release agents.³ Recently, Ni-PTFE composites on a nickel substrate have been reported as a novel electrode for synthesis of organic compounds.⁴⁻⁶ Despite the industrial importance of the composite, no systematic study of its preparation has been attempted. In the current investigation, nickel-PTFE composite was prepared by electrodeposition from a Watts nickel bath containing PTFE in suspension. The effects of PTFE content in the bath, current density, bath pH and temperature on the volume percent of PTFE in the composite were studied and electrolysis conditions optimized to get uniformly dispersed PTFE in the nickel matrix.

Experimental Procedure

The electrodeposition of nickel-PTFE composite was performed in a Watts nickel bath consisting of 250 g/L nickel sulfate, 45 g/L nickel chloride, 30 g/L boric acid and 0.1 g/L sodium lauryl sulfate. The PTFE polymer used for codeposition is in the form of a suspension containing 60 percent polymer by weight; the average size of the particles ranged from 1.5 to 2.0 µm. The same experimental set-up, as described elsewhere, was employed.7 A known volume of 60-percent PTFE suspension was added to the Watts bath. By means of a mechanically controlled glass stirrer, the PTFE suspension was stirred in the bath for eight hr. Plating was carried out in a 1-L glass beaker. As anodes, two 5-mm-thick nickel pieces were used. Stainless steel specimens 7.5 x 5.0 x 0.1 cm size served as cathodes from which the coatings could be easily stripped for analysis. The extent of PTFE incorporation in the nickel coating was studied with respect to PTFE content of the bath. Current density ranged from 2 to 8 A/dm², bath pH from 2 to 5, bath temperature from 30 to 60 °C.

The plated stainless steel samples with coating thickness of 50 μ m were weighed (w₁) with an accuracy of 0.1 mg, then stripped in 50 mL of 20-percent warm nitric acid. The resulting solutions were made up to 100 mL in standard volumetric flasks. After stripping the deposits, the stainless steel substrates were again weighed (w₂). The difference (w₁ - w₂) gave the mass of the composite deposit. The nickel content of the composites was determined by analysis of the solutions, using atomic absorption spectrophotometry.⁸ These steps determined the PTFE content. The volume percent of PTFE was evaluated as follows:

Volume of nickel in composite $(V_1) = \frac{\text{Mass of nickel}}{\text{Density of nickel}}$ Volume of PTFE in the composite $(V_2) = \frac{\text{Mass of PTFE}}{\text{Density of PTFE}}$ Total volume, $V = V_1 + V_2$ Volume percent of PTFE $= \frac{V_2}{V} \ge 100$

Triplicate analyses were performed in each case and the average values recorded.

Results and Discussion

Effect of PTFE Suspension On Volume Percent PTFE

The extent of PTFE incorporation with 5 to 80 mL/L PTFE suspension in the bath over a wide current density range is shown in Fig. 1. Regardless of operating current density, PTFE incorporation increases sharply and attains an opti-



Fig. 1—Effect of PTFE suspension on incorporation of PTFE in composite.



Fig. 2—SEM photographs of Ni-PTFE composite for different PTFE suspensions in the bath. Current density 6.0 A/dm², pH 3.0, temp. 50 °C: (a) 10 mL/L PTFE; (b) 20 mL/L PTFE; (c) 30 mL/L PTFE; (d) 80 mL/L PTFE.

mum value of 14.6 vol. percent at 25 mL/L PTFE suspension in the bath. With further additions, the particles appeared to agglomerate in the bath and a decreasing trend was observed. This trend beyond the optimum may be a result of insufficient stirring to maintain all the PTFE particles in suspension and to the greater degree of agglomeration in the bath.⁹

The SEM photographs of the Ni-PTFE composites obtained at different PTFE suspensions in the bath are shown in Figs. 2a-d. PTFE particles are uniformly reflected in white in the composite film. Adherent bonding is observed between the nickel matrix and the incorporated PTFE, with no voids. At a suspension of 10 mL/L PTFE (Fig. 2a), a part of the surface is covered with PTFE. Upon increasing concentration of PTFE (Figs. 2b and 2c), complete coverage, with uniform dispersion of PTFE in the nickel matrix is obtained. Figure 2d clearly reveals the growth of agglomerates with less PTFE incorporation at 80 mL/L of PTFE in the bath.

Effect of Current Density on Volume Percent

Figure 3 illustrates the effect of current density on the extent of PTFE incorporation in the composite. Regardless of PTFE concentration in the bath, maximum incorporation could be seen at 6 A/dm², above which the extent of codeposition decreased, resulting in a burnt deposit. This trend is consistent with other types of metal-particle composites¹⁰ where the metal is being deposited under conditions of charge transfer



Fig. 4—Effect of pH on incorporation of PTFE in the composite. Temp., 50 °C; current density, 6 A/dm²; PTFE conc., 25 mL/L.



Fig. 3—Effect of current density on incorporation of PTFE in the composite. Temp. 50 $^\circ\text{C}$, pH 3.0.

overpotential control. Above 6 A/dm², as the reduction of nickel ions is controlled by concentration overpotential, the amount of codeposited PTFE decreases gradually. The dependency of codeposition on charge transfer overpotential control indicates that the rate of codeposition is determined by initiation of real contact between the nickel ions adsorbed on PTFE particles and the cathode surface.¹⁰

Effects of pH and Temperature on Volume Percent The influence of pH on PTFE inclusion can be seen in Fig. 4. A smooth, uniform and semi-bright deposit containing 14.6 vol. percent PTFE was obtained at pH 3.0 for a PTFE suspension of 25 mL/L in the bath at 6.0 A/dm² and 50 °C. A brittle deposit was obtained at pH 5.0. The observed decrease in PTFE content between pH 2.0 and 3.0 may be a result of the decrease in the efficiency of nickel deposition and the increase in viscosity of the solution.⁹ The decrease in efficiency was found to lower the rate of nickel deposition, with a consequent decrease in incorporation of PTFE.



Fig. 5—Effect of temperature on incorporation of PTFE in the composite. Conc. PTFE, 25 mL/L, pH 3.0, current density, 6 A/dm².

It was found that the temperature of the plating bath influenced the amount of codeposition to a remarkable extent (Fig. 5). PTFE incorporation increased from 10.5 to 14.6 vol percent when the temperature was increased from 30 to 50 °C. Above this range, a slight decreasing trend was observed, probably resulting from the decrease in current efficiency of nickel deposition above 50 °C and at 6.0 A/dm².

Conclusions

Nickel-PTFE (14.6 vol. percent) composite has been obtained from a Watts nickel bath containing 25 mL/L PTFE suspension. The volume percent of PTFE in the composite increases with increase in PTFE concentration in the bath and current density. An adherent, smooth, uniform and semibright deposit could be obtained by operating the bath at 6.0 A/dm^2 at pH 3.0 and 50 °C.

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About the Authors



Dr. G.N.K. Ramesh Bapu is a scientist at the Central Electrochemical Research Institute, Karaikudi 623 006, Tamilnadu, India. He holds MSc and PhD degrees from Madurai Kamaraj University and has published more than 30 research papers. His technical interests include electrodeposition of metals, alloys and composites. He is an active member of

the Society for the Advancement of Electrochemical Science and Technology, Karaikudi, India.



Dr. S. Mohan is a scientist at the Central Electrochemical Research Institute, Karaikudi, India, working on electropolishing, electrodeposition of metals and oscillatory chemical reactions. He holds an MSc from Madras University and a PhD from the Indian Institute of Technology and has published more than 21 research papers.