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Electrodeposition and Morphology Analysis of Nickel Nanoparticles from Sulphate Bath

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The electrodeposition of Nickel in the presence of organic compound was studied systematically as function of the nature of the organic additive. The effects of various parameters such as nickel sulphate concentration, bath temperature, current density have been investigated on deposit physical appearance. The tolerance limits and the effect of combined organic additive have been identified under different experimental conditions. We have used scanning electron microscopy (SEM) to characterize the surface morphology of deposits. The SEM pictures show the formation of domain growth of nickel in which nanoparticles are mostly concentrated at domain boundaries and incorporated in the coating matrix to improve the surface mechanical properties. The coatings are found to be highly adherent and uniform by adding calixarene derivative as organic additive at very low concentration. Three-dimensional AFM images confirm that the presence of calixarene derivative improves the surface smoothness. The microhardness measurements were performed and the structure of deposits was examined by X-ray diffraction. [DOI: 10.1380/ejssnt.2010.227]

Keywords: Electrodeposition; Ni; Calixarene derivative, Surface morphology; Scanning electron microscopy; AFM

I. INTRODUCTION

The usage of nickel for electroplating has increased substantially in the last two decades and appears likely to grow because of greater consumption in the developing countries of Asia [1–3]. Nickel electroplating has gained major importance as a cheap and versatile surface finishing process for decorative applications [4–23]. Hence, nickel coating by electroplating is of special interest especially in the electronic industry, due to the possession of a combination of properties, such as good corrosion and wear resistance, deposit uniformity, electrical and thermal conductivity [24].

Previous studies of nickel electrodeposition mainly focused on the electrochemical behaviour and on the micrometre-scale morphology of nickel deposits on carbon and various metallic substrates and their dependence on the deposition conditions, such as temperature and stirring, the nature of the substrate and the electrolyte composition [25]. Regarding the latter, in particular the role of boric acid as a component in the widely-utilized Watts electrolyte has been studied, which is conventionally believed to act as a buffer [26].

Electrodeposition involves the reduction at the cathode surface of nickel ions arriving from the electrolyte to produce a deposit. The nickel coating is very thin and, often, so poorly adherent if organic additives are not been added. The presence of tiny amounts of additives or of complexing agents, can have enormous consequences for the resulting microstructure and composition of the deposited materials and hence for their properties. The kinetics and mechanisms of single metal deposition were studied extensively and reaction paths for many systems were established [27–29]. Complexing species play an important role for the discharge mechanism because they change the thermodynamic equilibrium and the rate determining step of the charge transfer reaction at the electrode surface. The additives are not only needed for levelling and super filling, but they also affect the structure and roughness of the deposits.

Electron microscopy is ideal for examining surface characteristics and structure since it enables high magnifications to be utilised up to ×100 000. The depth of focus is far superior to that of optical microscopy and the resolution possible is of the order of 5-10 Å for the transmission electron microscope and 150-250 Å for the Scanning Electron Microscopy scope [30]. Several groups used the Scanning Electron Microscopy (SEM) or the Atomic Force Microscopy (AFM) to study the effect of additives on the formation and movement of atomic steps during the growth of electrodeposits. In recent study, Ganesh *et al.* [31] were found that electrodeposition of nickel from a nickel sulphamate bath in the presence of a magnetic field applied at an angle of 45° to the cathode surface produces a nickel deposit with a fine grain structure. Gelatine was added to nickel electroplating bath as organic additive to control the deposition rate, crystallization, leveling and brightness of the deposit [32]. Less knowledge therefore is available on the mechanism of nickel deposition by using organic additives and how deposition conditions affect deposit structure and properties. Nickel electrodeposition exhibits complex potential-dependent growth be-

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