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Porosity determination of nickel coatings on copper by anodic voltammetry

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Abstract

This paper presents the application of anodic voltammetry to determine the effective porosity in nickel coatings. The nickel coatings were obtained by electrodeposition from a Watts bath on copper substrate. This technique consists in a comparison of the charge density involved in the passivation process of the substrate without coating and that required to passivate the substrate covered with a nickel layer. The passivation solution was a 0.4 M sodium sulfide solution (Na_2SO_3) at 25 °C selected to maintain the coating inert in the potential region where substrate passivation occurs, thus preserving its integrity. The results indicate an exponential decay of the coating porosity, with respect to the deposit thickness, and a net porosity of 4 to 5%. The relation between porosity decay and deposition potential was investigated for nickel deposition from a Watts bath.

1. Introduction

Metallic coatings are frequently inspected visually to detect pores. However, a precise determination of the amount and distribution of the pores is required. Pores and cracks in metallic coatings are localized interruptions of the coating material that may be defined as follows: (a) *pores* are voids expanded predominantly in three dimensions, which are not filled with solid or liquid materials, and (b) *cracks* are faults with expansion predominantly in one dimension [1]. They can be classified as in Figure 1 [1], as (i) pores and cracks 'passer-bys' crossing the whole coating, from the surface to the substrate, and (ii) pores and cracks 'no-passer-bys', subdivided in opened 'no-passer-by' (opened to the surface but not reaching the substrate), and closed ones (no opening to the surface, totally restricted into the coating or substrate)˙ In this work, pores and cracks of type 1 were studied. In general, the porosity in the electrodeposits is analysed qualitatively by four techniques: ferrocyanate test, electrographic test, hot water test and salt-spray test. These techniques are based on the observation and counting of pores, and, consequently, they are not sensitive to pores of small dimensions, which prevents the detection of small cracks, as well as small pores [2–5].

With the increasing interest in thin films technologies [6–8], there is a need for more reliable measuring techniques of coating porosity. Basically, the porosity measuring techniques can be divided into two categories: (i) techniques in which individual pores are detected by physical, chemical or electrochemical observation, and (ii) techniques in which the total porosity is obtained through measurements of gas permeation, chemical or electrochemical analysis [9]. Electrochemical measurement seem more appropriated to determines the effective coating porosity, including exposed substrate through pores and cracks of type 1. These techniques can be divided in four categories: open circuit potential measurements [10, 11], anodic current measurements [12–16], polarization resistance measurements [16], measurements of the charge obtained during process of potentiodynamic anodic polarization [10, 17] and, recently, cyclic voltammetric charge measurements [8].

This work presents the results obtained from voltammetric anodic dissolution, in which the dissolution/passivation charges of the substrate were measured. One positive aspect of this technique is the short analysis time (about one minute), when compared with current works [9]. In addition, the charges involved in the porosity analysis by the proposed technique are about 100 times smaller than those necessary to determine the porosity by other techniques [9]. As a consequence, a smaller interference level on the sample could be obtained, as well as a larger reliability and better precision [8]. Meanwhile, it should be noted that the applied technique uses a small area for analysis not considering possible edge effects.