THE STRUCTURE AND GROWTH OF NICKEL ELECTRODEPOSITS ON ELECTROPOLISHED COPPER SINGLE-CRYSTAL SUBSTRATES

BY A. K. N. REDDY* AND H. WILMAN

(Applied Physical Chemistry of Surfaces Laboratory, Chemical Engineering Department, Imperial College of Science and Technology, London, S.W. 7, England)

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ABSTRACT

An exploratory study has been made of the orientation and growth of nickel electrodeposits on copper (110) and (111) substrates. The general nature of the variation of deposit structure with current density and thickness has been elucidated, and the results compared with previous work involving a deposit-substrate pair, where both metals belong to the f.c.c. system.

INTRODUCTION

THOUGH some of the first electron diffraction experiments were concerned with electro-deposits, it has only been in the past decade that the structure and growth of these deposits were studied systematically by varying parameters like thickness, current density, temperature, concentration, etc. Setty and Wilman1 were the first to carry out such an intensive study of cathodic crystal growth. They studied the electrodeposition of silver on silver single-crystal substrates. These authors drew the following conclusions:

1. That there was a minimum critical current density below which deposits grew in parallel orientation (relative to the substrate) up to large thicknesses (of the order 100,000 Å).

2. Deposits prepared at a current density greater than the minimum critical current density showed, with an increase in deposit thickness, the following sequence of changes in surface structure: parallel orientation→twinning→random polycrystalline structure→a preferred one-degree orientation.

* Present address: Post-doctoral Research Associate, Electrochemistry Laboratory, University of Pennsylvania, Philadelphia 4, Pa.
The higher the current density, the smaller was the thickness at which parallel orientation of the electrodeposit crystals gave way to octahedral twinning.

Kumar and Wilman extended these results to the case of copper grown electrolytically on silver single-crystal substrates, and to silver on copper.

These authors also observed a broadly similar pattern of change in surface structure with factors such as current density, thickness and temperature as was observed by Setty and Wilman. However, certain differences in the nature of growth were also observed. For instance, Kumar and Wilman found that when copper grows on a silver (110) face, there is a disorientation of the electrodeposit crystals which adopt a range of orientations about a particular lattice row. This phenomenon was termed directed disorientation and it was suggested that it resulted from tensile stresses developed due to the lack of exact fitting of the deposit and substrate lattices. The lattice constant difference in the Cu-Ag deposit-substrate pair was 0.4703 Å.

The present exploratory study was undertaken with two objects in view: (a) to ascertain the general form of dependence of deposit structure on thickness and current density in the case of nickel deposited on copper single-crystal substrates, (b) to study whether the type of directed disorientation (observed by Kumar and Wilman) generated by the deposit-substrate pair having a relatively large lattice constant difference would also be found in the case of nickel on copper where the lattice constant difference is only 0.0925 Å.

**Experimental Details**

Electropolished copper single-crystal faces were taken as substrates. The electroplating bath consisted of 240 g/pl of NiSO₄·7H₂O and 30 g/pl of H₃BO₃. The anode was a nickel sheet (7×5 cm.). The single-crystal face was made to touch the surface of the electrolyte and nickel was deposited on to it at a particular current density and at 20° C. The thickness of the electrodeposit was estimated utilizing Faraday's Laws and experimentally determined current efficiencies. Care was taken to minimize atmospheric oxidation after electrodeposition by flooding the crystal face with distilled water, absolute alcohol and isopropyl alcohol in rapid succession. The crystal was then quickly transferred to the electron diffraction camera keeping the crystal face of copper covered by a film of the high boiling-point alcohol. Electron diffraction examination of the surface structure of the electrodeposit was carried out in the usual way. Nickel deposits of different