

STM Study of 2D and 3D Phase Formation during Electrodeposition of Ni, Ni_xAl_{1-x}, Co and Co_xAl_{1-x} from a Chloroaluminate Room Temperature Molten Salt

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AlCl₃-rich room temperature molten salts are useful electrolytes for the deposition of aluminium and transition metal-aluminium alloys. Several electrochemical and spectroscopic investigations of Ni_xAl_{1-x} [1] and Co_xAl_{1-x} [2] deposition have been performed, correlating the potential with the composition of the deposited alloy. However, up to now little is known about the initial stages of electrocrystallization and growth in these systems.

In this paper, we describe an in situ STM study of the electrodeposition of Ni, Co and their respective aluminium alloys from Lewis acid AlCl₃/1-butyl-3-methylimidazolium chloride on Au(111). Furthermore, voltage tunneling spectroscopy has been performed for the first time at the molten salt electrolyte/electrode interface. Ni deposition close to its equilibrium potential is characterized by a 2D layer-by-layer growth. Formation of 3D Ni-clusters starts almost exclusively along the step edges of the underlying substrate. Below -0.2 V vs. Ni/Ni(II) Al codeposition sets in leading to 3D cluster growth all over the electrode surface. Cobalt deposition leads to formation of randomly distributed almost monodisperse 2D islands over a wide potential range between 0 and -0.15 V vs. Co/Co(II). Codeposition of Al starts below -0.25 V.

Voltage tunneling spectra have been measured in both systems at various potentials and coverages. The effective tunneling barriers decrease with increasing aluminium content of the deposited alloy, as it is expected from UHV data.

[1] W.R. Pitner, C.L. Hussey, G.R. Stafford, J. Electrochem. Soc. 143, 130 (1996)

[2] R.T. Carlin, P.C. Trulove, H.C. De Long, J. Electrochem. Soc. 143, 2747 (1996)