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In situ investigation of electrodeposition at liquid-mercury interfaces by X-ray reflectivity

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Thanks to the high quality crystalline form of obtainable nanostructured material, due to the lack of substrate-induced stress or strain, and to the possibility of controlling growth parameters by applying an interface potential in electrodeposition, the interest in the study of growth processes at liquid-liquid interfaces has been lately renewed. With the aim of understanding nucleation and subsequent growth of crystals at Hg-electrolyte interfaces, experiments using a combination of electrodeposition, in situ XRR and time-resolved XRD were performed, employing the high resolution diffractometer LISA [4,5], at P08, PETRA III synchrotron, Hamburg. In previous studies, electrolyte containing 0.01M NaF+0.01M NaBr+0.5mM $PbBr_2$ was found to exhibit an adlayer growth [6,7]. Changing the potential from values < -0.8V vs Hg/Hg_2SO_4, where the Pb ions are amalgamated in the Hg, to values >0.7V, these ions are deamalgamated, leading to the growth of a monolayer followed by 3D nanocrystal formation of PbBrF.
Currently a fluorite free electrolyte is used: 0.01M NaBr+0.05mM $PbBr_2$. While XRR curves show no evidence of growth at this concentration, experiments at higher concentrations (0.5mM $PbBr_2$) clearly exhibit crystal growth. Also a different electrodeposition system was studied: Ge growth from 0.1M Na_2SO_4+0.05M GeO_2 electrolyte on Hg electrodes [1]. These investigations will aid understanding of the first stages of nucleation and growth by electrodeposition at liquid-liquid interfaces.
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