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Abstract

The object of this study was the explanation of the participation of the methanol molecules in the formation process of the methoxy pseudo-passive layer during the cathodic polarisation on the nickel surface in anhydrous methanolic solutions of electrolytes. The aim of this work was also understanding the mechanisms and kinetics of the methoxide film formation. The study was conducted in anhydrous methanol solutions of lithium and tetrabutylammonium sodium methoxide (CH₃ONa) chloride (LiCl), tetrafluoroborate (Bu₄NBF₄). There were used the classical polarization techniques: cyclic voltammetry (CV), chronoamperometry at constant potential and electrochemical impedance spectroscopy (EIS). After the electrochemical tests the structure of the methoxy layer formed on the nickel was analyzed using the innovative in situ spectroelectrochemical method (ATR-FTIR), and ex situ techniques (Raman spectroscopy, SEM-EDX, XPS, XRD, confocal microscopy).

Due to the complexing properties of the Cl ions, the studies conducted in the CH₃OH – LiCl solutions in the cathodic range of potential has not revealed the solid product on the nickel surface. The mechanism of the surface product creation has been proposed in "alkaline" CH₃OH – CH₃ONa solution, where the methoxide film formation and growth is observed. The creation of Ni(OCH₃)₂ layer is the result of a chemical oxidation of intermediate product (NiOCH_{3,surf}). In anhydrous solutions of Bu₄N(BF₄) it has been found the surface carbon layer as a result of cathodic polarization of nickel.

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