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### **ACCEPTED MANUSCRIPT**

# The effect of tungsten addition on metallurgical state and solute content in nanocrystalline electrodeposited nickel

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#### **Abstract**

It is usually difficult to control the incorporation of foreign species in electrodeposited coatings originating from the solvent or the chemical species used for the electrodeposition bath. However, the presence of these impurities can modify their physicochemical properties. In the present study, complementary analytical techniques were used to evaluate the chemical contamination in nickel and nickel-tungsten alloys, electrodeposited from additive free baths. In order to better understand the relationship between impurity content and grain size refinement, the concentration of light elements (H, O and N) was systematically quantified by hot extraction analysis. Also, the distribution of contaminants was evaluated by SIMS analysis. We have shown that in nanocrystalline electrodeposited nickel the grain size refinement and the impurity contents are strongly related. However, in Ni-W alloys the evolution of the contamination is more complex, with a maximum amount for W contents around 10 at.%.

#### 1. Introduction

Nanocrystalline materials have been the subject of many studies due to their specific physicochemical properties, generally correlated to the grain size refinement. However, as the grain size decreases, other parameters as crystallographic orientation, solute content, internal stresses, grain boundary or misorientation and density of defects are also modified. Several studies reported that even small amount of impurities can have a dramatic effect on the properties of nanocrystalline and polycrystalline metals (ex. strength and ductility or corrosion) [1-8], which shows the importance of evaluating these elements.

Electrodeposition of metals and alloys are usually accompanied by the incorporation of foreign species originating from the solvent (often water, leading to hydrogen co-deposition) or from the chemicals species used for the bath (metallic salts, complexing agents, additives ...). Metallic impurities can be analyzed using elementary analysis techniques such as X-Ray Fluorescence, GDOES (Glow Discharge Optical Emission Spectroscopy) or GDMS (Glow Discharge Mass Spectrometry). However, the detection and quantification of light elements, as H, C, N, and O, is quite difficult explaining why these elements are generally investigated in bulk materials rather than in electrodeposited metals.

Few studies have investigated the non-metallic contamination in electrodeposited nickel and nickel alloys using hot extraction method [9-11]. The nitrogen content in nanocrystalline electrodeposited nickel was influenced by the bath composition and increased up to 620 wt. ppm in coatings elaborated from a bath containing tartrate (complexing agent) and Na-saccharin (inhibitor) [11]. Chassaing *et al.* [9] have shown that the amounts of H and O are more important in electrodeposited Ni-Mo alloys than in pure nickel deposited from the same bath.

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