1. Introduction

The properties of engineering materials are direct correlated to their microstructure. One of the microstructural parameters which influence the properties of polycrystalline metals and alloys is the grain size. Since the early 1980´s materials with the grain size in the nanocrystalline range have been extensively studied. These studies involve a wide variety of technological areas such as electronics, ceramics, magnetic data storage and structural components. The majority of the research in nanocrystalline materials is focused on the study of their electric, optic and magnetic properties; however, in the past decade the mechanical behavior of nanocrystalline metals has become more and more attractive to the research community. This latter interest is strongly related to the well known relationship between grain size and mechanical strength, typically described by the Hall-Petch relationship, which shows that the strength of the material increases with decreasing grain size. However, as the grain size reduces to the nanometer range, the variation of the strength with the grain size does not completely follow the Hall-Petch relationship.

The desire for understanding the “unusual” deformation response in nanocrystalline metals has stimulated the research on the mechanisms causing this effect. However, the microstructure characteristics of the studied nanocrystalline metals have not gained such attention by now. Most of the investigation on the mechanical deformation of nanocrystalline metals discussed the microstructure only in terms of the average grain size; however the response to mechanical deformation is not only related to this parameter but also to the distribution of the grain size and to the grain morphology, among others.

Nanocrystalline materials can be produced by several techniques as for example: inert gas consolidation, mechanical allowing, electrodeposition and severe plastic deformation. One of the techniques which attracted particular interest to the research field of mechanical behavior of nanocrystalline metals is electrodeposition. This interest is related to the knowledge that this process is capable of produce high density films with nanocrystalline grain size and high fraction of large angle grain boundaries at relative low cost.

Electrodeposition is a well established technique, which has been used for film preparation for more than 100 years. The research in this field is broad and encloses preparation of compound semiconductors, fabrication of coatings for corrosion and wear resistance and even for decorative purpose. The structure and properties of electrodeposited materials depends on a large variety of parameters, which extend from the process condition to the bath composition. This variety of factor influencing the deposit properties opens several research fields for this technique, and the majority of publication on electrodeposited metals deals with the influence of the deposition parameter on the properties of this materials. Although the microstructure, texture and surface characteristics of the deposits are often considered in these studies, there are fewer detailed analyzes on the microstructure, regarding for example the grain size distribution, the grain boundary character or texture evolution.

Since CoNi forms a solid solution over the entire concentration range, these alloys are interesting for their magnetic and mechanical properties. The wide solubility enables their potential use for microelectronical systems (MEMS), because it is possible to shift the magnetic characteristics from soft to hard magnetic by adjusting the Co content of the alloy. And the low stacking fault energy of Co allows tailoring the crystal structure, to control the stacking fault energy and the twin density of nanocrystalline CoNi alloys.

The lack in microstructure study of electrodeposited nanocrystalline metals has stimulate the present research, which is focused on a detailed characterization of the microstructure, texture and grain boundary character of electrodeposited CoNi samples. The requirement of studying some microstructure parameters as grain size distribution, grain morphology, grain boundary character distribution and of correlating these with the texture and with the film growth has motivate the use of electron backscatter diffraction (EBSD) for this research. The EBSD technique allows the microstructure related texture analysis of large sample areas with a spatial resolution of approximately 30 nm. The complete characterization of electrodeposits, however, requires further techniques. In this work x-ray diffraction, energy dispersive spectroscopy and transmission electron microscopy were applied to achieve further understanding of the microstructure features of the studied samples. Furthermore, a new emerging technique which is a combination of precise serial sectioning with focused ion beam (FIB) and EBSD based orientation microscopy (3D-EBSD) has been applied to extend the microstructure knowledge obtained in the two dimension to third dimension. The use of this latter technique has allowed the characterization of grain boundary planes, which play an important role in the microstructure formation and evolution of the studied films.

This work was mainly concentrated on the study of the microstructure, texture and grain boundary characteristic of an electrodeposited CoNi from a deposition bath with 0.02g/l of saccharin. This three sample characteristics are presented in three subchapters of the experimental results and are individually discussed. Based on the knowledge obtained by this characterization a three dimensional orientation study of this film was performed. The final subchapter of the experimental results discusses the influence of additive level in the deposition bath, on the film microstructure and texture characteristics, where three samples produced with different level of saccharin were characterized according to their microstructure, texture and grain boundary character. The entire characterization study was performed on three distinct sections of the samples, the substrate interface, the bath interface and the cross section. Only in this way the microstructure and texture evolution with the film growth could be studied and a fundamental understanding on the microstructure formation could be achieved.

This work presents and discusses the microstructure characteristics of three nanostructured electrodeposited samples. Since the electrodeposition process influences strongly the microstructure of these materials, this work does not only describe but also provides some fundamental understanding on the formation of these microstructures.