1.1 Introduction

There are numerous processing techniques developed for the synthesis of nanocrystalline materials, such as ball milling, electrodeposition, sol-gel, inert gas condensation, combustion synthesis, sputtering, rapid solidification and physical and chemical vapor deposition (Landolt, 1994; Chou, 1991; Suwa, 1986; Savage, 1984; Ramasamy, 2005). Compared to other methods, electrodeposition has many advantages, e.g., a large number of pure metals, alloys and composite systems can be deposited with grain sizes less than 100 nm, the low initial capital investment required to synthesize these materials, high production rates, few size and shape limitations, and ability to produce fully dense nanostructures free of extraneous porosity (Alfantazi, 1996). Compared to direct current plating, pulse current (PC) electrodeposition can yield ultra-fine-grained structures and more homogeneous surface appearance of deposits with improved properties, such as ductility, hardness, strength, corrosion resistance and wear resistance (Ding, 1998; Kang, 2002; Berkh, 1995; Natter, 1996; Natter, 2008; Despa, 1999; Musiani, 2000). More diversified microstructures can be developed, since in PC deposition one can control the microstructure and composition of deposited metals and alloys more effectively by varying the pulse on time, pulse off time, duty cycle and pulse frequency (Alfantazi, 1994).

Electroplated Ni has been widely used for over a century now. However, considerable interest remains in further improving its mechanical, magnetic, electrical, and corrosion properties. These properties are strongly influenced by many parameters such as composition, morphology, internal stresses, preferred orientation and grain size of the deposits (Kaja, 1986). A large variety of micrometer or nano sized ceramic powders, such as SiC, Al_2O_3, ZrO_2, Li_2O_3 and TiO_2 have been incorporated in the Ni coatings to improve their hardness and wear resistance (Kaisheva, 2004; Webb, 1994; Hou, 2002; Reddy, 2008; Orlovskaja, 1999; Xue, 2004; Ferkel, 1997; Thiemig, 2008).

Recently, there has been a lot of interest in the nano CeO_2 particle reinforced Ni coatings because of their good corrosion resistance and potential applications in surface protection. Qu et al. (2006) synthesized the Ni–CeO_2 nanocomposite coatings by electrodeposition process with a variation in current density (1 and 3 Adm^{-2}) and concentration of CeO_2 in the electrolyte (10 and 30 g/l). They reported that the Ni–CeO_2 nanocomposite coating had higher hardness (446.2 HV), enhanced corrosion resistance and improved wear property compared to nanocrystalline pure Ni. Aruna et al. (2006) synthesized the Ni–

CeO_2 composite coatings by electrodeposition process from the electrolyte containing 100 g/l CeO_2 with the variation in current densities (0.23, 0.77, 1.55, 3.1 and 5.4 Adm^{-2}) and investigated the corrosion behavior. They also reported that the hardness and corrosion resistance of Ni–CeO_2 composite coatings were better than nanocrystalline pure Ni. Xue et al. (2006, 2010) studied the tribological properties of Ni–CeO_2 composite coatings synthesized by electrodeposition method from a nickel sulfamate solution containing CeO_2 particles and found that the composite coating containing 2.3 wt. % CeO_2 showed somewhat increased wear resistance compared to pure Ni coating.

From the literature survey, it appears that no systematic studies have been done to evaluate the effect of electrodeposition parameters (surfactants, additives, current density, bath temperature, duty cycle and electrolyte agitation) on the microstructures and properties (hardness, thermal stability, grain growth behavior, wear, scratch, corrosion and electrical resistivity) of Ni–CeO_2 nanocomposite coatings. Keeping this in mind, an attempt has been made to evaluate the effect of deposition parameters on the microstructure and hardness of the Ni-CeO_2 nanocomposite coatings. A number of coatings with varying CeO_2 concentration have been prepared by pulse electrodeposition. A through investigation is carried out to evaluate the properties, i.e., hardness, thermal stability, wear, scratch, and corrosion resistance, and electrical resistivity of these coatings.

1.2 Objectives

The specific objectives of the present investigation are:

(1) Synthesis of nano-sized CeO_2 reinforcements.

(2) Study the effect of high energy ball milling on powder characteristics like lattice parameters, size, strain and type of dislocation.

(3) Study the effect of different electrodeposition parameters (amount of surfactant and additive, current density, bath temperature, duty cycle, and stirring rate) on the microstructure and hardness of Ni-CeO_2 nanocomposite coating.

(4) Synthesis of pure Ni and Ni-CeO_2 nanocomposites containing different concentrations of CeO_2 by pulse electrodeposition method and subsequent microstructural characterization.

(5) Evaluation of hardness, thermal stability, wear, scratch and corrosion resistance, and electrical resistivity of the pure Ni and Ni-CeO_2 nanocomposite coatings.