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ScienceDirect

Procedia Materials Science 8 (2015) 91 – 100

Procedia
Materials Science

www.elsevier.com/locate/procedia

International Congress of Science and Technology of Metallurgy and Materials, SAM - CONAMET 2013

Electrodeposition of Ni-Co Alloys. Determination of Properties to be Used as Coins

Diego S. Schweckandt ^(a), Maria del Carmen Aguirre ^{(b)*}^a *Ciencias de los Materiales, FAMAF- Universidad Nacional de Córdoba, Calle Medina Allende s/n, 5016-Córdoba, Argentina*^b *Subgerencia de Laboratorio, Banco Central de la Republica Argentina, Reconquista 266, C1003ABF Buenos Aires, Argentina*

Abstract

Our purpose is to look for new materials of Ni, Co and their combination, useful for blanks for coins. Thus, the objective is to change the old techniques in the fabrication of blanks, of higher cost, by direct current methods. On the subject suitable material for circulating coins, we focused on the synthesis of new alloys of NiCo over brass, by potentiostatic methods, studying the physicochemical and mechanical properties. Ni_xCo_{100-x} alloys and pure Ni and Co, were electrodeposited over foil brass (CuZn) p.a (width: 0,03mm), by cyclic voltammetry (CV) and constant potential (CP). The measurements by spectrometry of X-Ray fluorescence (EDXRF), microprobe (EDS) and SEM, showed Ni_xCo_{100-x} alloys but Co enrichment with smoothed surface and a granular morphology. The material was also characterized by X-ray diffraction showing (fcc) and (hcp) phases in accordance with the Ni_xCo_{100-x} composition, and the surface hardness assay, roughness and conductivity tests were also performed, obtaining specific properties, suitable for a coinage materials.

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Selection and peer-review under responsibility of the scientific committee of SAM - CONAMET 2013

Keywords: Ni-Co alloys, money material, characterization

1. Introduction

Technological advances achieved during the last decades have enabled the study of materials and devices of different dimensions. One methodology to build and study these small systems is electrochemistry [Schlesinger and Paunovic, 2010]. Nowadays, materials based on Cu, Ni, Al, and their alloys CuAl8, CuAl6Ni2, CuNi25 have been used for decades as a material in circulation coin blanks

[Odjj 2010; Watts 1887; Varea et al 2012]. Within these non-precious metals, nickel is one the most noble. Besides, its typical brightness and resistance to wear and corrosion are notable. Copper-nickel alloys results cheaper, where even though the quality of this material lies below that of pure nickel; their qualities are satisfactory for circulation coin [VDM. AG 1987]. However, with the rising costs of metals for decades have been introducing coins made of electroplated materials. Most of them are steel substrates, electroplated with Cu, Ni, brass, bronze, and combinations [Schlesinger and Paunovic, 2010; Odjj 2010; Watts 1887; Varea et al 2012; German Copper Institute Booklet 2013]. In turn, the development of this type of blanks -name that is given to the part before its coinage- requires different technologies, which may involve the use of one or more layers of the aforementioned elements. However, with the rising costs of traditional metals through the years, industry began to manufacture them in various ways. Today basic literature tells of alloys prepared by sputtering, thermal casting type (CuAl8, CuNi25) or electrodeposition (CuNi) [Varea et al 2012; German Copper Institute Booklet 2013]. The alloy containing 88% Cu and 12% Ni material was introduced as currency in USA in 1886 [German Copper Institute Booklet 2013]. Initially, the use of electrodeposited blanks was reserved for low denomination coins but gradually has spread to middle names and even today in some countries, all the coins are made of steel electroplated [Odjj 2010]. Being the electrochemistry an area of great diversity, we investigated new materials based on Ni, Co, Cu and combinations thereof, feasible to be used in the minting of coins so it can replace old techniques of "casting" in the manufacture of coins, of higher cost, by direct current methods: potentiostatic/galvanostatic. Ni, Co and their alloys are important materials in industry for many applications due to their unique properties such as magnetism, corrosion resistance, hardness, conductivity. There are various references in literature about electrodeposition Ni_xCo_{100-x} alloys by various methods and characterization[Bai et al (2002) ;Bai et al (2003), Bai et al (2005); Kim et al (2003);Wang et al (2005); Tury et al (2006)]. Considering this reference material we investigated around the topic of manufacturing large-scale materials, the synthesis of new NiCo alloys based on brass substrates by potentiostatic methods characterizing their mechanical, electrical and magnetic effects for use in coins. It is worth mentioning that although all properties are important and together determine the suitability of a material for minting, are considered especially transcendent relatively low hardness and grain size -as thin as possible- for a good quality of minting in the finished coin [VDM .AG 1987].

2. Experimental procedures

Electrochemical assays were performed using an Autolab302N unit in an electrochemical cell with a system of three electrodes: the working electrode (Brass substrate), reference electrode (Ag/AgCl) and auxiliary electrode (graphite). The electrolyte consisted in the respective salts $NiCl_2 \cdot 6H_2O$ (Tetrahedron) (0.2 M solution) and $CoCl_2 \cdot 6H_2O$ (Tetrahedron) solution (0.2 M) to pH 3 with the addition of H_3BO_3 (Cicarelli) (solution 0,4M). The total volume in the cell was kept at 60 ml varying volumes of Ni and Co salts for different nominal deposit relationship. The different solutions were electroplated on CuZn brass 70/30, (Alfa Aesar 99.995%) of 0.3 mm thickness. The brass pieces were mechanically polished (diamond paste 1 μ m) and chemically treated prior to the electrodeposition. This was conducted with gentle bubbling N_2 to the electrochemical cell. Cyclic voltammetry was performed in a potential range of -0.32V to -1.2V at 20 mV/s, and 30 cycles, and the molar compositions of the NiCo electrolytic bath was: 0/100, 10/ 90, 25/75, 50/50, 75/25, 90/10 and 100/0. Electrodeposited samples at a constant potential of -0.80V for 1800 sec, maintained the molar composition NiCo of the electrolytic bath in: 0/100, 50/50, 65/35, 75/25, 80/20, 90/10 and 100/0. It also prepared to -0.65 V for 30 min a NiCo composition 50/50. The composition and morphology of the samples were determined by energy dispersive X-ray spectroscopy (EDS) on a JEOL JXA-8230, and the composition was evaluated comparatively also with an X-ray fluorescence spectrometer (EDXRF) Shimadzu EDX900HS model at 50kV, Rh anode, 10 mm collimator and vacuum atmosphere. The resulting structures were characterized by X-ray diffraction (XRD) measured on a Philips PW3830

diffractometer using Cu K α radiation ($\lambda = 1.5418 \text{ \AA}$), in the 2θ range from 30° to 90° in Bragg-Brentano configuration. In addition, we carried out measurements of Rockwell hardness (HR-15T) with a team EMCOTEST, preload time 1s, 3s total load time, analyzed the roughness (Ra 4mm) with a SURTRONIC25 Taylor Hobson equipment and conductivity (% IACS at 60 kHz) with a SIGMASCOPE SMP10 Fischer, Cu pattern 100.8 % IACS.

3. Results and Discussion

The Co electrochemical properties are intermediate between those of Fe and Ni. The system standard potential: $\text{Co}^{2+} + 2e^- = \text{Co}^0$, is -0.28V vs SHE, whereas the (E°) of the system: $\text{Ni}^{2+} + 2e^- = \text{Ni}^0$, is -0.25V vs SHE, that is, the Ni is more reducible than Co, but the potentials are very close, so it is expected that both elements can be incorporated into the alloy in its nominal composition. Fig. 1a and 1b show the 30 cycles of cyclic voltammetry in a potential range of -0.32V to -1.2V at a scan rate of 20 mV/s in the deposition of Co and Ni pure, respectively. From Fig. 1a, by pure Co it is observed that in positive sweep, there is anodic re-dissolution at potentials less negative, which is not seen in Fig. 1b, by pure Ni. Inside 1a, one cycle was plotted of the preparation of pure Co sample. In it exist an intercrossing at -0.6V (nucleation potential) during the cathodic scanning. In Figs. 1c, 1d, 1e, 1f, and 1g are showed the cyclic voltammeteries for compositions in the electrolyte bath: c) $\text{Ni}_{10}\text{Co}_{90}$, d) $\text{Ni}_{25}\text{Co}_{75}$, e) $\text{Ni}_{50}\text{Co}_{50}$, f) $\text{Ni}_{75}\text{Co}_{25}$, and g) $\text{Ni}_{90}\text{Co}_{10}$. All profiles of samples are similar in cathodic sweep, with a unique reduction wave commencing at -0.6V , except the anodic reverse cycle, that changes according to the Co content in the sample.

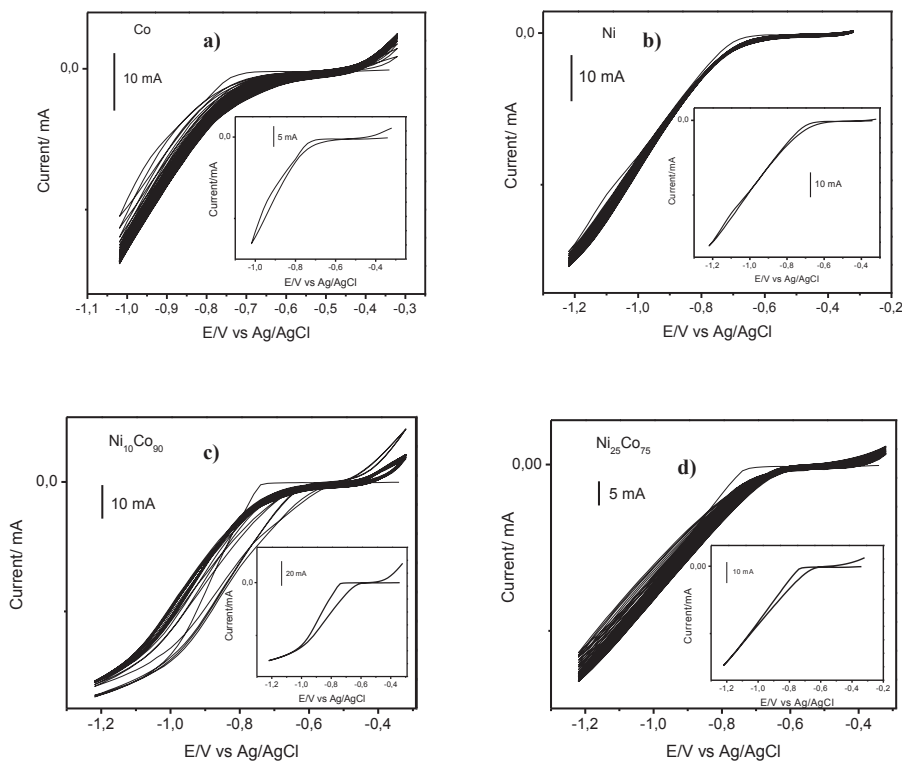


Figure 1. CV at 20 mV/sec , 30 cycles, in electrolytic bath composition of: a) pure Co, b) pure Ni, c) $\text{Ni}_{10}\text{Co}_{90}$, d) $\text{Ni}_{25}\text{Co}_{75}$, Inset is graphed the first cycle.

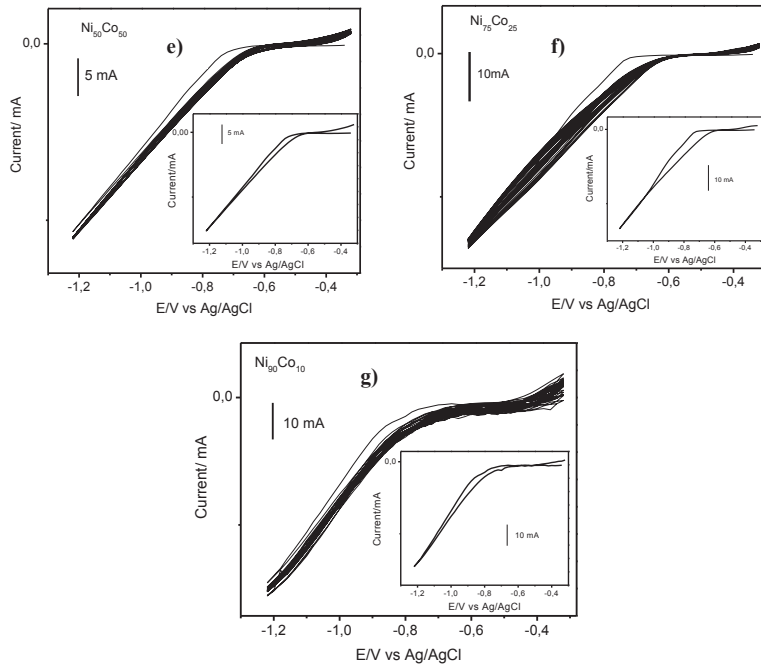


Figure 1. CV at 20 mV/sec, 30 cycles, in electrolytic bath composition of: e) $\text{Ni}_{50}\text{Co}_{50}$, f) $\text{Ni}_{75}\text{Co}_{25}$ and g) $\text{Ni}_{90}\text{Co}_{10}$. Inset is graphed the first cycle.

The samples synthesized at constant potential are showed in Fig. 2. It was graphed the current density versus deposition time. It can be observed an increase in the deposition rate with the theoretical Co content in the sample, with well-defined maximums at nearly 100 sec, until the composition of the electrolytic bath $\text{Ni}_{50}\text{Co}_{50}$. At lower overpotentials (-0.65 V) in $\text{Ni}_{50}\text{Co}_{50}$ an abrupt decrease in deposition rate and shift in the maximum current to longer times are observed, indicating a free growth without diffusion-controlled interaction cores up to 400 sec, then the superposition of the growth centres prevent further growth and slightly decrease the current.

The characterization of the microstructures $\text{Ni}_x\text{Co}_{100-x}$ was made by electron microprobe, (EDS), in comparison with energy dispersive X-ray fluorescence (ED-XRF), scanning electron microscopy (SEM) and X-ray diffraction (XRD). In general, in the analysis of $\text{Co}_x\text{Ni}_{100-x}$ composition, there was an acceptable correlation between the values obtained by electron microprobe (EDS) and energy dispersive X-ray fluorescence (ED-XRF). Table 1 shows the composition results by EDS of the alloys synthesized by cyclic voltammetry. Table 2 shows the comparative results between EDS and ED-XRF for alloys synthesized at -0.8 V/30 min. In samples analyzed by EDS, none of the samples detected the presence of oxygen. The relationship $\text{Ni}/(\text{Co}+\text{Ni})$ between the composition of the electrolyte and the alloy obtained by measurement (EDS) in the samples synthesized by cyclic voltammetry is shown in Fig. 3a. It is observed an enrichment in Co all compositions $\text{Ni}_x\text{Co}_{100-x}$, which agrees with published literature [Bai and Hu 2002], because the experimental conditions used in this work. The composition ratio in the alloy versus the electrolyte, for $\text{Ni}_x\text{Co}_{100-x}$ synthesized at -0.8 V for 30 min are shown in Fig. 3b. According to EDS, the curve follows the same trend as the 3a but with less incorporation of Ni that samples synthesized by cyclic voltammetry. The sample with the electrolytic bath composition $\text{Ni}_{50}\text{Co}_{50}$, synthesized at -0.65 V for 30 min showed an alloy composition of $\text{Ni}_{12.25}\text{Co}_{87.75}$ very similar that one synthesized at -0.8 V for 30 min. Thus a less sobrepotential in the electrodeposition didn't improve the Ni incorporation in the sample because the H_2 evolution was not even inhibited.

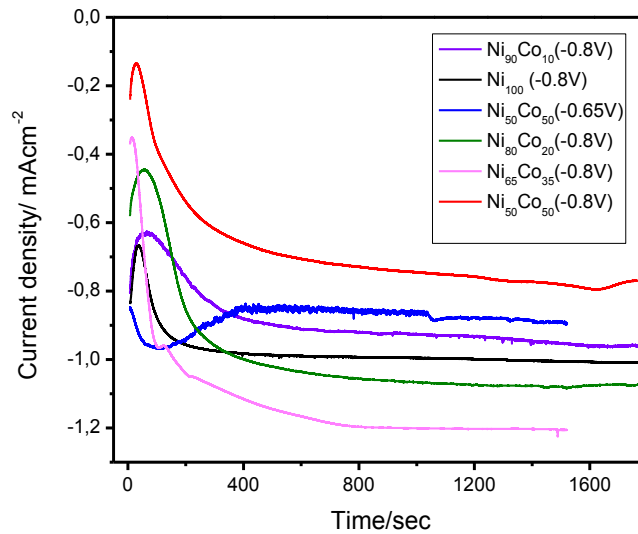


Figure 2. Ni₁₀₀(black line), Ni₉₀Co₁₀(pink line), Ni₈₀Co₂₀(olive green line), Ni₆₅Co₃₅(light blue line), Ni₅₀Co₅₀(red line), at -0.8V/30min, and Ni₅₀Co₅₀(dark blue line) at -0.65V/30min.

Table 1. Composition of Ni_xCo_{100-x} alloys with EDS and ED-XRF assays, obtained by CV

Electrolyte composition	EDS		ED-XRF	
	Ni	Co	Ni	Co
Ni	100	0		
NiCo 90/10	72.50	27.50		
NiCo 75/25	42.35	57.65	37.43	62.57
NiCo 50/50	14.84	85.16	14.80	85.20
NiCo 25/75	4.85	95.15	6.55	93.45
NiCo10/90	0.60	99.40		
Co	0	100		

Table 2. Composition of Ni_xCo_{100-x} alloys with EDS and ED-XRF assays, obtained at -0.8V/30'

Electrolyte Composition	EDS		ED-XRF	
	Ni	Co	Ni	Co
Ni	100	0		
NiCo 90/10	41.82	58.12	41.93	58.07
NiCo 80/20	25.62	74.38	30.48	69.52
NiCo 75/25	20.57	79.43	20.51	79.49
NiCo 65/35	16.06	83.94		
NiCo50/50	12.47	87.53	21.54	78.46
Co	0	100	35.55	64.45
Brass				Cu68Zn32

During electrodeposition by cyclic voltammetry in the range -0.32 V to -1.2 V, and at constant potential (-0.8 V), the hydrogen evolution was observed, whereby a side reaction was the reduction of H⁺ to potential very negative. The release of H₂ would cause an increase in pH around the electrode surface with the formation of Co(OH)⁺ and Ni(OH)⁺ [Bai et al 2002]. These authors [Bai et al 2003; Bai et al 2005]

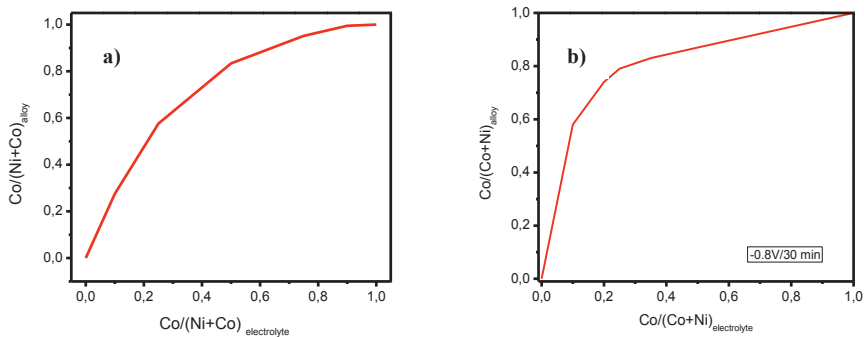


Figure 3. Co/(Ni+Co)alloys vs Co/(Ni+Co)electrolyte, mol ratio, by samples of a) CV, and b) -0,8V/30min

have published that in the electrodeposition of Ni-Co alloys there is an anomalous codeposition with enrichment Co and it has been described as an abnormal phenomenon in the adsorption competition between metal hydroxides, monohydroxides or metal ions. Since the adsorption ability of a less noble metal hydroxide, $\text{Co}(\text{OH})^+$, is considered more than a nobler metal $\text{Ni}(\text{OH})^+$, NiCo alloys are often enriched with Co. To overcome this difficulty NiCo alloys could be deposited from molten salts (more complex method) or changing the conditions in the method of electrodeposition. Cyclic voltammetry can enhance the incorporation of Ni modifying the potential range from 0 V to -1 V and conducting the pH of the electrolyte to 1 to avoid the presence of $\text{Ni}(\text{OH})^+$. The CV method is somewhat more malleable than CP, since in the latter case, a very small overpotential using a potential more positive than -0.65 V, partly would avoid the presence of H_2 , but with the difficulty that if it couldn't exceed the critical nucleation overpotential the electrodeposition does not occur. However, a pulse method, can improve the interfacial sorption properties, because the molecular species such as H_2 or $\text{Ni}(\text{OH})_2$ are easily desorbed during the relaxation time, improving the electrocrystallization process.

The characterization by XRD of the materials prepared by cyclic voltammetry (Fig. 4) and a constant potential (Fig. 5) showed good agreement with the crystalline phases (fcc) and (hcp) according to the alloy composition. In Fig. 4, for the phase pure Ni (fcc) matches patterns (JCPDS-00-001-1260,00-001-1266,00-003-1051) with the plane (111) highest intensity at 44.77° , a cell parameter of 3.5028 \AA and a crystal size of 22.60 nm [Kim et al. 2003; Wang et al. 2005, Tury et al. 2006]. In the alloy $\text{Ni}_{72.5}\text{Co}_{27.5}$, the crystal size was 29 nm, with a cubic cell parameter of 3.5073 \AA . Alloys $\text{Ni}_{72.5}\text{Co}_{27.5}$, $\text{Ni}_{42.35}\text{Co}_{57.65}$ and $\text{Ni}_{14.84}\text{Co}_{85.16}$ contained phases (hcp) of Co (JCPDS00-005-0727,00-001-1278) (fcc) of Co (JCPDS00-015-0806,00-001-1259) and (fcc) Ni (JCPDS-00-001-1260,00-001-1266,00-004-0850). For $\text{Ni}_{4.85}\text{Co}_{95.15}$ alloy, the phases observed are (hcp) of Co and (fcc) of Co with peaks of more intensity at 41.83° and 76.276° and a average crystal size of 27 nm. The pure Co is little crystalline, the phases (fcc) or (hcp) are detected slightly, with a peak at 44.31° and crystal size of 23 nm. The maximum intensity at $2\theta = 72.57^\circ$ corresponds to the main line of the substrate CuZn. $\text{Ni}_x\text{Co}_{100-x}$ alloys gradually change from a (fcc) to (hcp) structure with increasing the content of Co, which is also seen in Figure 5 for samples prepared at constant potential.

The micrographs of the samples synthesized by cyclic voltammetry are shown in Figs. 6a, b, c, d, e and f. The morphology and grain size of the alloys vary with the Co content and the structure of the phase. Structures $\text{Ni}_x\text{Co}_{100-x}$ alloys gradually changed from (fcc) to (hcp) with increasing content of Co. In the sample of pure Ni, Fig. 6a, the regular bit morphology is globular. High content nickel alloys, $\text{Ni}_{72.5}\text{Co}_{27.5}$. Fig. 6b, there are fine and coarse grains roughly spherical intermixed arranged on a closed smooth surface. Sample $\text{Ni}_{42.35}\text{Co}_{57.65}$ (showed as $\text{Ni}_{42}\text{Co}_{58}$ by simplicity in Fig.6c) presents sizes coarser uniform closed surface. In Fig. 6c, a large number of grains of the same size and approximately spherical shape. Co enriched alloys as $\text{Ni}_{4.85}\text{Co}_{95.15}$, $\text{Ni}_{14.85}\text{Co}_{85.16}$, simplified in Figs. 6d and 6e shown open surfaces,

roughened with decreasing grain size. In $\text{Ni}_{0.6}\text{Co}_{99.4}$ and pure Co (Figs. 6f and 6g) provides a flatter surface and consistent, fine-grained morphology, circular and average size 200 nm. In pure Ni sample prepared at $-0.8\text{ V}/30\text{ min}$, closed surface is formed by fine grains regularly as shown in (Fig. 7a). Fig. 7b shows, the micrograph of pure Co morphology similar that obtained by cyclic voltammetry. Thicknesses $\text{Ni}_x\text{Co}_{100-x}$ films synthesized by cyclic voltammetry are between 5.47 and 7.59 μm , and they were calculated according to the equation: $d = Q \text{ Mw}/zA\rho$, where (Q: measured load during electrodeposition, Mw: average atomic weight of the mixture Co-Ni, $z = 2$, A: sample area, and ρ : average density mixture Co-Ni).

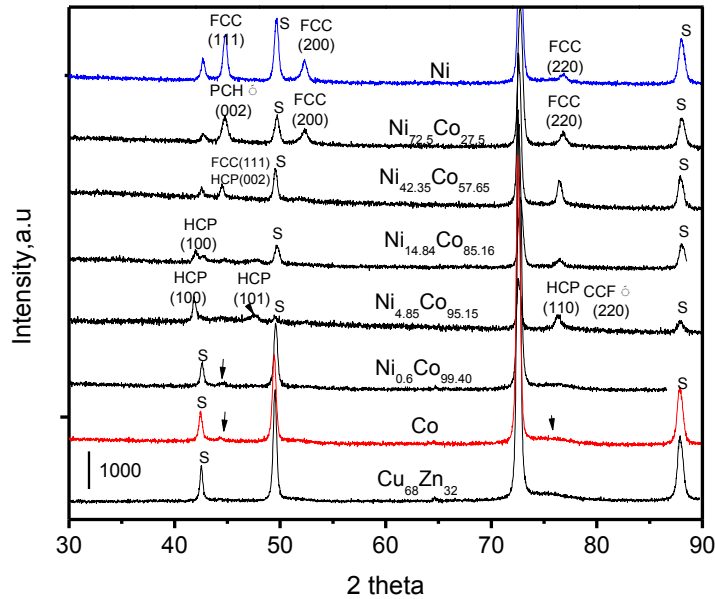


Figure 4. XRD of $\text{Ni}_x\text{Co}_{100-x}$ alloys synthesized by CV.

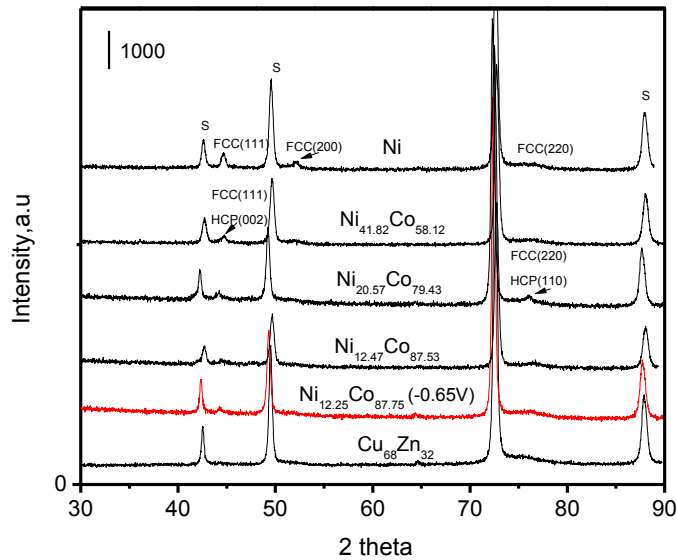
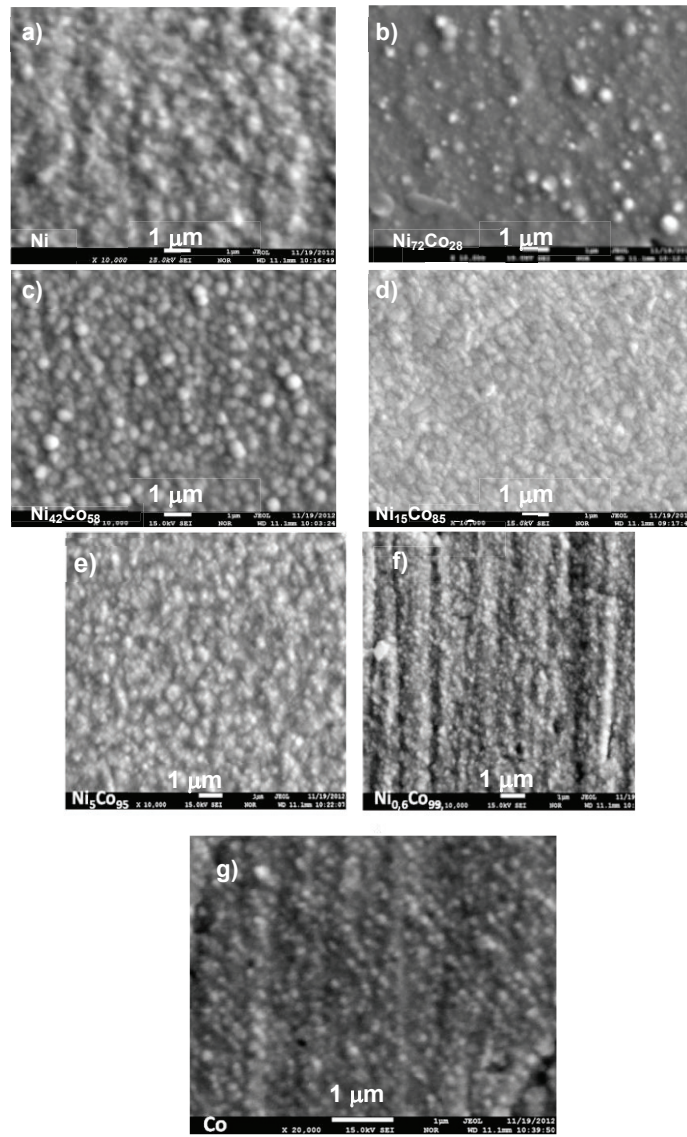
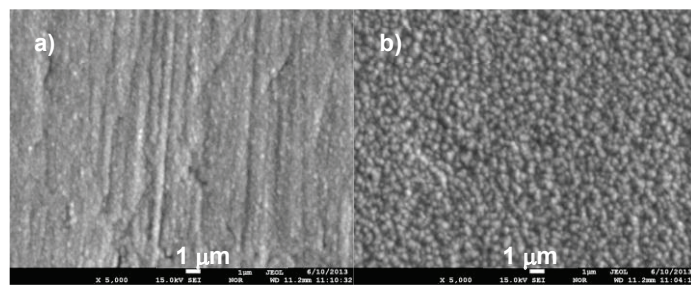


Figure 5. XRD of $\text{Ni}_x\text{Co}_{100-x}$ alloys synthesized at $-0.8\text{ V}/30\text{ min}$ and at $-0.65\text{ V}/30\text{ min}$ (red line).

Figure 6. SEM Image of Ni_xCo_{100-x} , synthesized by CV.Figure 7. SEM Image of pure a) Ni and b) Co, synthesized at $-0.8V/30 \text{ min}$

The mechanical properties of the Ni_xCo_{100-x} alloys are tabulated in 3, 4 and 5. Table 3 shows, the results of Rockwell surface hardness (HR-15T) with load applied of 15kg for 3 seconds. For Ni_xCo_{100-x} alloys made by CV and CP (-0.8 V/30 min) were observed in the composition near $Ni_{42}Co_{58}$, somewhat higher micro hardness values. This is accordance with date reported by Wang et al 2005, in electrodeposited NiCo samples, where the maximum value of micro hardness noted for 49 % of Co in the alloy, and then decayed with the increase and decrease of the Co content. Roughness properties (Table 4), are in correspondence to the strain resistance and micro hardness observed in Table 3.

Table 3. Surface Rockwell (HR-15T) hardness, load 15kg/3s, para Ni_xCo_{100-x} , prepared by CP and CV

Electrolyte composition	-0.8V/30' Alloys	Hardness HR	CV Alloys	Hardness HR
NiCo 25/75	-----	-----	$Ni_{4.85}Co_{95.15}$	81.05
NiCo 50/50	$Ni_{12.47}Co_{87.53}$	81.20	$Ni_{14.84}Co_{85.16}$	80.55
NiCo 65/35	$Ni_{16.06}Co_{83.94}$	79.20	-----	-----
NiCo 75/25	$Ni_{20.57}Co_{79.43}$	77.05	$Ni_{42.35}Co_{57.65}$	81.65
NiCo 80/20	$Ni_{25.62}Co_{74.38}$	74.85	-----	-----
NiCo90/10	$Ni_{41.82}Co_{58.12}$	81.45	$Ni_{72.5}Co_{27.5}$	81.36
Ni 100	Ni_{100}	80.90	Ni_{100}	80.90
Brass	$Cu_{68}Zn_{32}$	71.50	$Cu_{68}Zn_{32}$	71.50

Table 4. Roughness of Ni_xCo_{100-x} samples, synthesized by CP and CV

Electrolyte composition	-0.8V/30' Alloys	Roughness μm	CV Alloys	Roughness μm
NiCo 25/75	-----	-----	$Ni_{4.85}Co_{95.15}$	0.26-0.34
NiCo 50/50	$Ni_{12.47}Co_{87.53}$	0.34-0.60	$Ni_{14.84}Co_{85.16}$	0.40-0.46
NiCo 65/35	$Ni_{16.06}Co_{83.94}$	0.38-0.84	-----	-----
NiCo 75/25	$Ni_{20.57}Co_{79.43}$	0.50-0.52	$Ni_{42.35}Co_{57.65}$	0.20-0.26
NiCo 80/20	$Ni_{25.62}Co_{74.38}$	0.28-0.34	-----	-----
NiCo90/10	$Ni_{41.82}Co_{58.12}$	0.20-0.26	$Ni_{72.5}Co_{27.5}$	0.56-0.92
Ni 100	Ni_{100}	0.28-0.34	Ni_{100}	0.20-0.30
Brass	$Cu_{68}Zn_{32}$	0.12	$Cu_{68}Zn_{32}$	0.12

Table 5. Conductivity at 60 kHz with Cu 100,8%IACS pattern

Electrolyte Composition	-0.8V/30' Alloys	%IACS	CV Alloys	%IACS
NiCo 25/75	-----	-----	$Ni_{4.85}Co_{95.15}$	7.040
NiCo 50/50	$Ni_{12.47}Co_{87.53}$	4.920	$Ni_{14.84}Co_{85.16}$	6.338
NiCo 65/35	$Ni_{16.06}Co_{83.94}$	5.667	-----	-----
NiCo 75/25	$Ni_{20.57}Co_{79.43}$	5.445	$Ni_{42.35}Co_{57.65}$	5.330
NiCo 80/20	$Ni_{25.62}Co_{74.38}$	6.190	-----	-----
NiCo90/10	$Ni_{41.82}Co_{58.12}$	5.710	$Ni_{72.5}Co_{27.5}$	4.509
Ni 100	Ni_{100}	7.014	Ni_{100}	7.010
Brass	$Cu_{68}Zn_{32}$	7.696	$Cu_{68}Zn_{32}$	7.696

To measure the relative electrical conductivity of copper is used as the known unit % IACS (International “Standard unalloyed copper” which describes as 100% IACS electrical conductivity to a pattern corresponding to a copper wire 1 meter in length and 1 gram of weight, which gives a resistance of 0.15388Ω at 20°C . In developing an alloy widely used currencies is CuNi25. This presents an intense silver, and in general its conductivity is about 5 % IACS, relative value very close to those obtained in our materials $\text{Ni}_x\text{Co}_{100-x}$ (see Table 5), and comparison with $\text{Ni}_{42.35}\text{Co}_{57.65}$ alloy prepared by CV. The conductivity is one of the parameters used for recognition by machines for coins.

4. Conclusions

Cyclic voltammetry (CV) and constant potential test (CP), synthesized $\text{Ni}_x\text{Co}_{100-x}$ alloys (without formation of oxides/hydroxides) with fine grain morphologies and/or thick according to the content of Co in the alloy. All cases, was observed a major incorporation of Co to Ni with respect to the electrolyte composition. It is estimated that the inclusion of Ni will be improved changing the conditions in CV method or using double potentiostatic pulses. X-ray diffraction indicated good agreement crystalline phase (fcc) and (hcp) with the alloy composition and average grain sizes between 23-30 nm. Surface Rockwell hardness measurements were (NiCo 80 HR-15T, 15T Brass HR-70), roughness (Ra 0.3-0.8 μm) and conductivity (5-7% IACS), achieving particular properties, similar to materials suitable for coins. Any of the samples obtained could be used for making circulation coins because the measured properties are satisfactory for the projected application. However, it is suggested $\text{Ni}_{42}\text{Co}_{58}$ alloy for its hardness, roughness and conductivity very close to the coins in circulation. It also certain that although all properties are important and together determine the suitability of a material for the coinage, in the alloy proposal values of hardness and grain size that predict a good quality of finished coin minting. Finally, considering the economic factor, an alternative is to produce blanks of pure Ni electroplated because the low price/ton of material compared to Co and ease of production.

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