

# Effect of parameters: on the electroplating of Ni-Co alloys in an ammonium citrate bath on Yellow Brass (C27200).

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<b>Abstract</b>	Ni-Co alloy coatings are electroplated from a solution of nickel sulfate with Co suspension on a copper substrate. Then study the factors affecting the precipitation of Ni-Co alloys from a citric acid and ammonium citrate bath (The impact of the bath's carbon dioxide content and current density on the sediment's carbon dioxide content). The optimal conditions were C.D.2A/dm <sup>2</sup> , temperature 55°C, time 60 minutes, pH 8. Characterization of the coating layer in terms of (surface shape, composition of the coated layer, hardness measurements).by studying sedimentary surfaces by SEM, thin films by X-ray diffraction, and Vickers microhardness. It was found that the largest surface hardness value was at the current density of 2 A/dm <sup>2</sup> (470HV).
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<b>Keywords:</b>	Ni-Co alloy, citrate bath, copper substrate
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### Plating Bath Preparation and Cell Construction

Cu Zn alloy sheet metal with dimensions of 0.6 mm x 30 mm x 30 mm, a pure nickel anode with an effective area of 100 mm x 50 mm x 10 mm, a quantitative examination of the samples was carried out. Model AAAnalyst400 No2380 Atomic Absorption Spectrophotometer. the specimens' C27200 quantitative chemical analysis Golden Brass. an ammonium citrate bath was utilized as the plating medium in the investigation. The composition of the plating bath is shown in Table (2).

Table (2) Nickel-Cobalt plating bath chemical composition using Ammonium Citrate

Bath Chemical composition	Content
Nickel Sulphate	30g/l
Cobalt Sulphate	4g/l-15g/l
Ammonium Citrate	60g/l

### Bath Control

The temperature of the solution was kept at 55 °C ± 1°C during the plating process using hot plate type HS10-2 a. pH of the bath was controlled using ammonium hydroxide (NH<sub>4</sub>) OH to sustain pH 8. The experiments were done using DC power supply type SPS-3610 for providing the plating bath with 2-6 Amp.

### Experimental procedures

1. Specimen Preparation

Fig (1) shows the different steps for specimen preparation and plating.

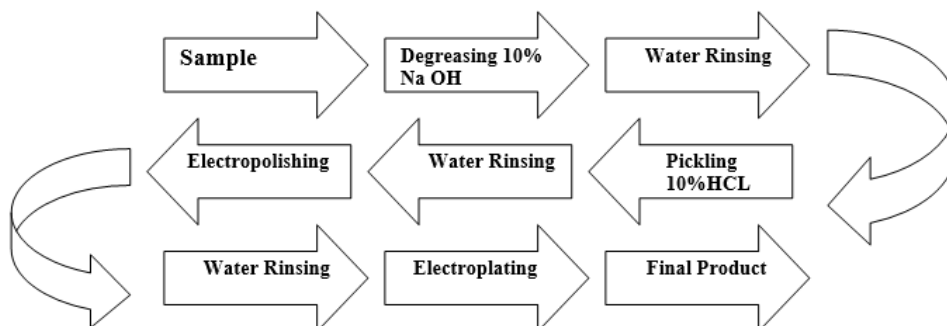


Fig. (1) Schematic Diagram and plating Representing Specimen preparation

2. The obtained coating thickness was measured by taking a cross-section of the plated sample and measuring the applied layer with an optical microscope (neophyte 21, Germany).
3. XRD analysis, which was carried out to follow up on the surface structure and phase variations of the various specimens, was used to ascertain the chemical structure of various coated specimens. X-ray diffractometers with copper targets and nickel filters were used to measure a sample from each group. [2]. XRD data were based on the following Bragg's equation:  $\lambda n = 2 d \sin \theta$ , where:  $n$  = integral number,  $\lambda$  = wave length,  $\theta$  = diffraction angle [3]



Fig. (2) (XRD) Unit used measurement

4. The SEM was utilized to display the crystal size and surface morphology. The examination was carried out on different samples. The results are obtained as computer printouts. Fig. (3) [4] shows the photo for SEM used for specimen surface demonstration.

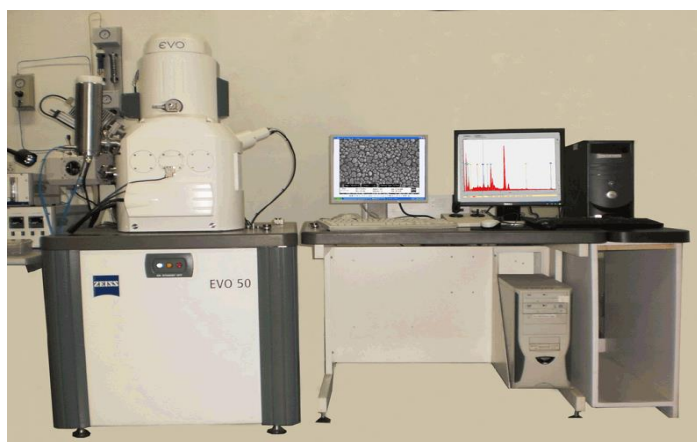


Fig. (3) (SEM) Unit

5. The EDX method was used to analyze the deposited layer composition on the specimens. It is a technique used for identifying the elemental composition of the specimen, The EDX analysis system works as an integrated feature of a scanning electron microscope (SEM), and cannot operate on its own without the latter. During EDX Analysis, the specimen is bombarded with an electron beam inside the scanning electron microscope. The bombarding electrons collide with the specimen atoms' own electrons, knocking some of them off in the process. A position vacated by an ejected inner shell electron is eventually occupied by a higher-energy electron from an outer shell. here, however, the transferring outer electron must give up some of its energy by emitting an X-ray.[5]
6. The specimens after coating were enclosed in a bakelite mount. Then polished using emery papers with different sizes 220, 500, 800, and 1000. finally, the specimens were polished using alumina solution.
7. Vickers microhardness test of the deposit was measured under load 10 grams for 15 seconds using Shimdsu micro-hardness tester see Fig (4). The average of the two diameters length of the diagonal was measured and consequently, the microhardness value in HV was obtained automatically by the hardness tester.[6]



Fig. (4) Micro-hardness tester with Optical microscope

## Results and Discussion

### Current density's impact on an ammonium citrate bath's deposition rate

Fig. (5) shows the average deposition rate with current density change from 2 to 6 A/dm<sup>2</sup> in (mg/dm<sup>2</sup>.min). The values of deposition rate increased from 17 (mg/dm<sup>2</sup>.min) at current density 2 A/dm<sup>2</sup> to 49 (mg/dm<sup>2</sup>.min) at 6 A/dm<sup>2</sup>, the bath temperature was held at 55°C, pH 8, and time 60 min at 4g/l Co. It is clear that the deposited film thickness increased as the current density increased from 2 to 6 A/dm<sup>2</sup>.

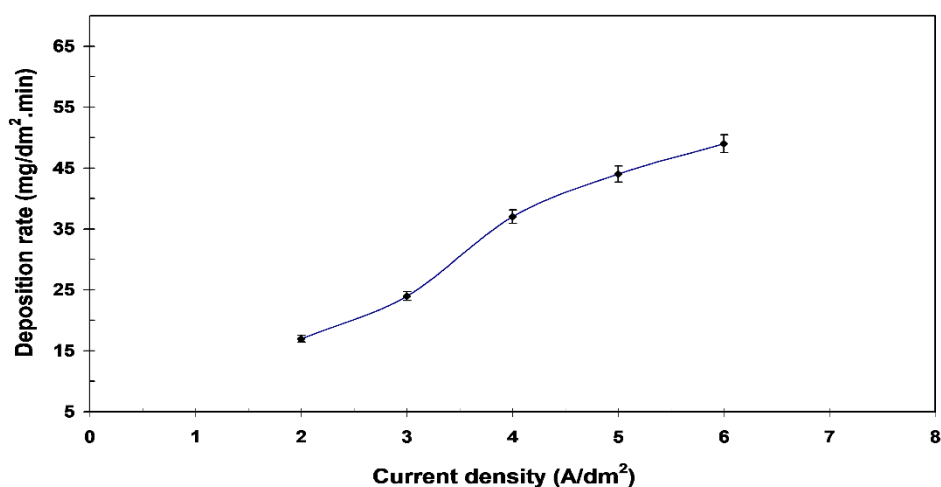
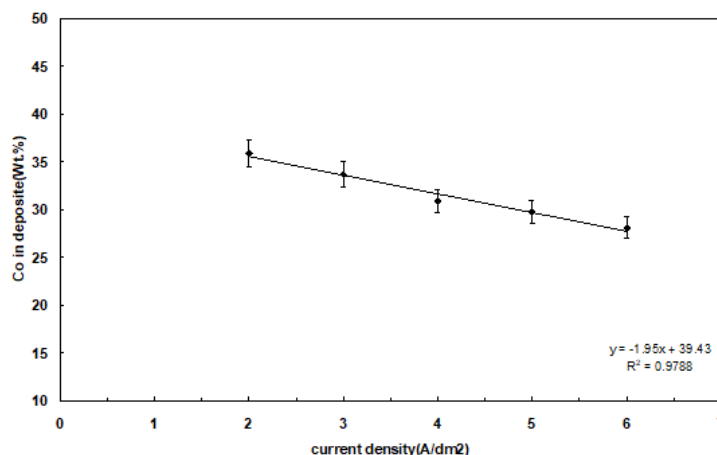


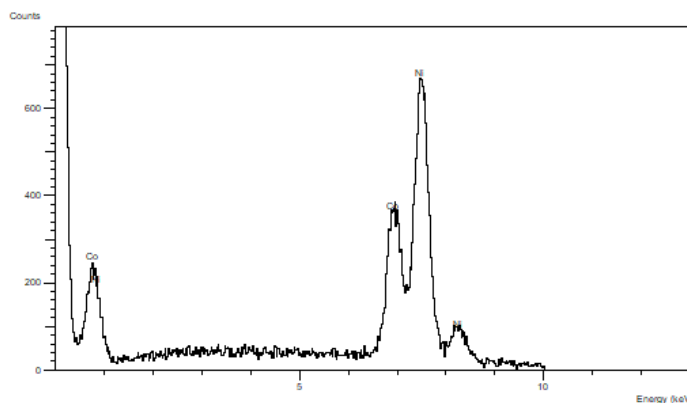
Fig. (5) Deposition rate in an ammonium citrate bath is influenced by current

**Current density's impact on the amount of Co deposited in an ammonium citrate bath**

Figure (6) shows the change of Co content in Ni-Co alloy coating with current density. The bath temperature is maintained at 55°C, time = 60 minutes, and the pH value is maintained at 8. It is obvious that the Co percentage decreases with increasing current density. EDX analysis showed a 36 wt % reduction in Co content. At a bath load of 4 g/L, the current density increased from 2 A/dm<sup>2</sup> to 6 A/dm<sup>2</sup> and the weight percentage increased to 28%. This outcome is consistent with earlier findings for CO-Ni-Al<sub>2</sub>O<sub>3</sub> made by Gang Wu et al.

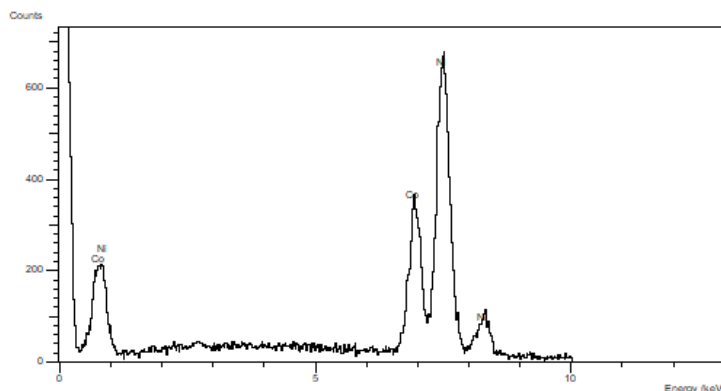


**Fig.6. Effect of current density on Co% in deposit**



**Fig. 7. Chemical analysis by X-ray analysis using electron dispersive (EDX) at current density 2 A/dm**

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**Fig.8. Chemical analysis by X-ray analysis using electron dispersive (EDX) at current density 3A/dm<sup>2</sup>**

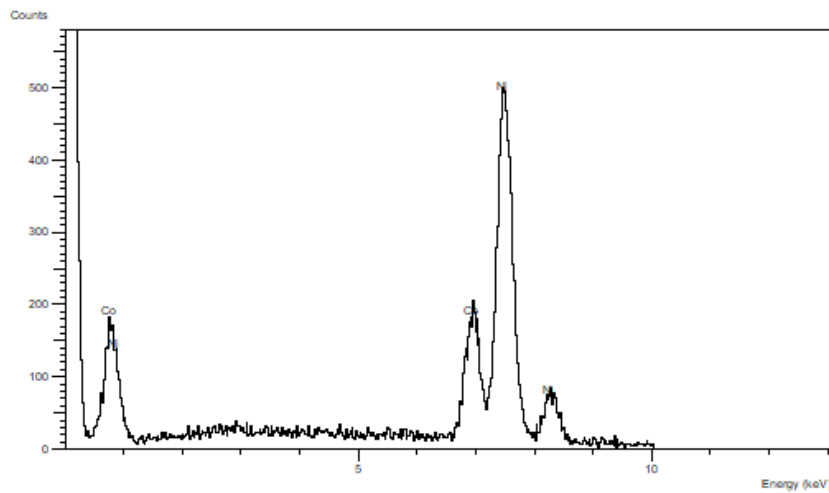


Fig. 9. Chemical analysis by X-ray analysis using electron dispersive (EDX) at current density 4A/dm<sup>2</sup>

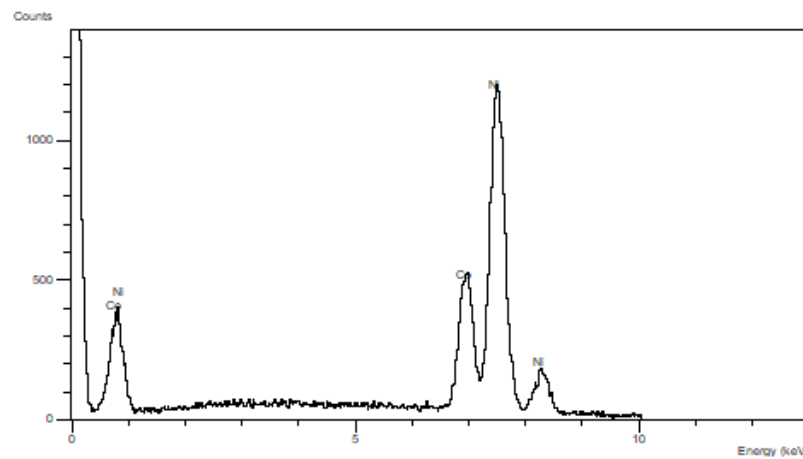


Fig. 10. Chemical analysis by X-ray analysis using electron dispersive (EDX) at current density 5A/dm<sup>2</sup>

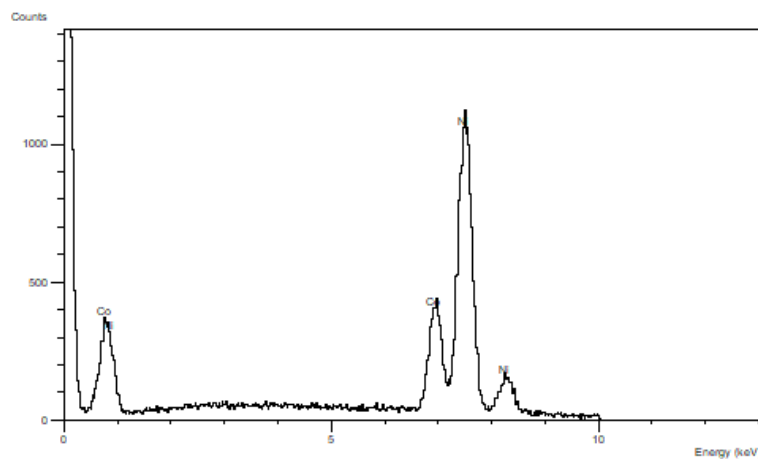
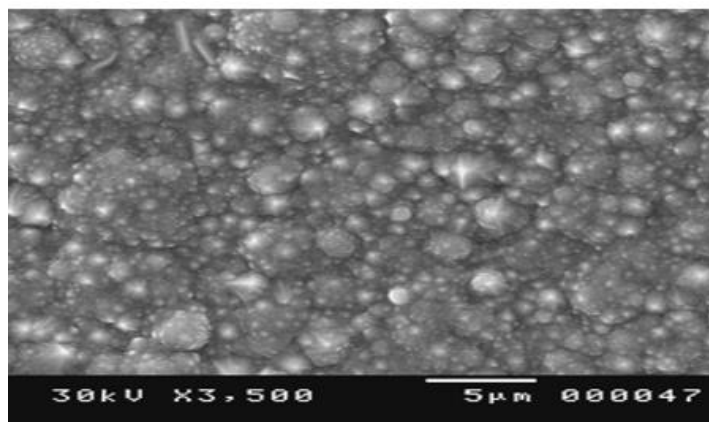


Fig. 11. Chemical analysis by X-ray analysis using electron dispersive (EDX) at current density 6A/dm<sup>2</sup>

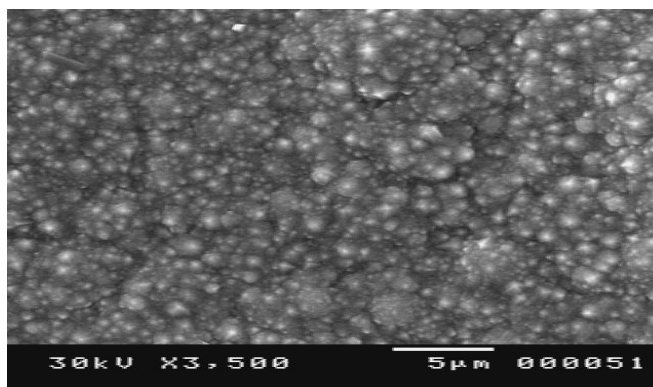
**Surface morphology**

Figure (12). As can be seen from the figure, pure nickel deposits have irregular polyhedral crystals. Bright spherical particles are what the agglomerates appear to be. Changes in cobalt content may be responsible for the morphological change; The size of grains increases as cobalt concentration rises. EDX analysis reveals a uniform distribution of Co in the alloy.



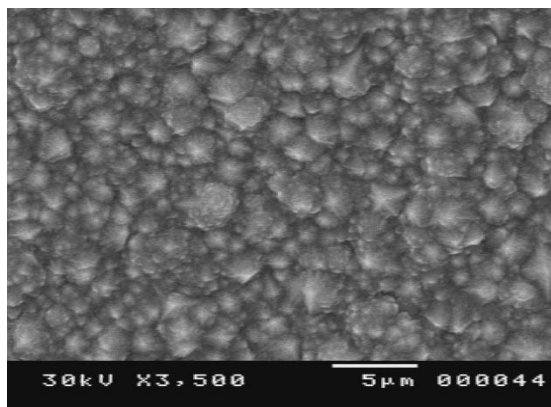
**Fig. 12a.** The surface morphology of the Ni-Co alloy at Cobalt Sulphate 10g/l.

The morphology of the Ni-Co coating consists of a uniform fine spherical structure containing approximately 39% Co particles



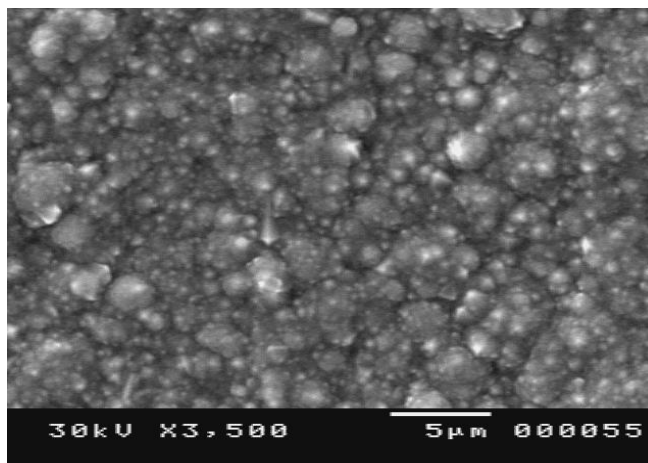
**Fig. 12b.** The surface morphology of the Ni-Co alloy at Cobalt Sulphate 15g/l.

The morphology of the Ni-Co coating consists of a uniform fine spherical structure with a Co particle content of approximately 48%



**Fig12c.** The surface morphology of the Ni-Co alloy at Cobalt Sulphate 20g/l.

The morphology of the Ni-Co coating consists of a uniform fine spherical structure containing approximately 47% Co particles.

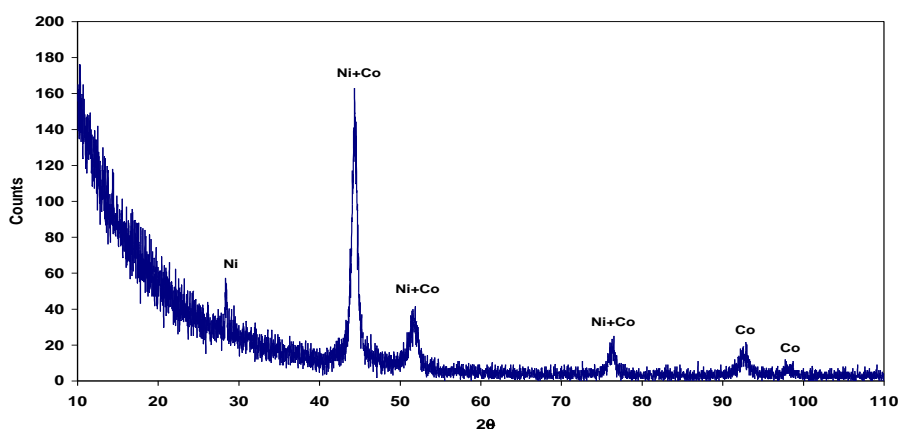


**Fig. 12d.** The surface morphology of the Ni-Co alloy at Cobalt Sulphate 25g/l.

The morphology of the Ni-Co coating consists of a uniform fine spherical structure containing approximately 47% Co particles.

#### Composition of the coated film

The composition of the coating film was determined using X-ray diffraction (XRD) analysis, Figure (13). XRD patterns are shown, and the XRD patterns confirm the presence of Co particles embedded in the nickel matrix.



**Fig. 13.** XRD patterns of the Ni/Co gradient deposit prepared at (2A/dm<sup>2</sup>).

#### Hardness measurements in ammonium citrate bath

Figure 14. Shows the microhardness of coated samples as a function of current density. The bath temperature was held at 55°C, pH at 8, and time 60 min. and the current density change from 2 to 6 A/dm<sup>2</sup>. The microhardness test was conducted on the surface and was taken as the average of 5 readings to represent the surface. Coating the brass with Ni-Co has increased its surface hardness from 130 HV (brass substrate) to 470 HV. Fig (14). indicates that the greater value of surface hardness was at the current density of 2 A/dm<sup>2</sup> and then the surface hardness decreased when the current density increased.

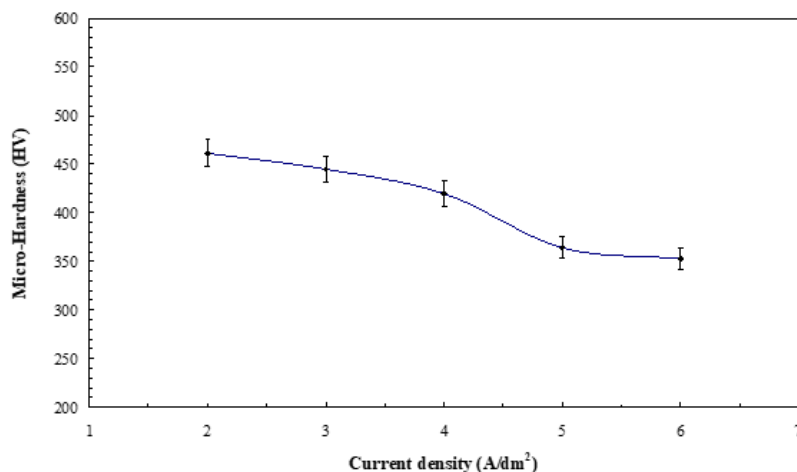


Fig. (14) Influence of current density on the microhardness of Ni–Co alloy coating.

### Efficiency in hardness

$$\text{Efficiency of coat} = \frac{H_1 - H_2}{H_2} \times 100$$

$$\frac{470 - 130}{130} \times 100 = 261.5\%$$

$H_1$  = Hardness of coated alloy at (C.D. 2A/dm<sup>2</sup>, pH 8, temperature 55°C and time 1hr).  $H_2$  = Hardness of base metal (Brass).

### Conclusions

1. In this work, a novel alloy Ni-Co coating method using an alkaline citrate bath is presented.
2. In comparison to commercial Watt's baths, alkaline citrate baths are less expensive and have a more favorable environmental impact.
3. The Ni-Co gradient deposit was with obtained a homogeneous distribution of the Co content up to 48 wt. % at conditions (2 A /dm<sup>2</sup>, temp.55°C, PH 8, agitation 250-300 r.p.m and Co 15 g / l.
4. The hardness of the deposited Ni-Co alloy coating increases from 130HV to 470HV. An increase of 261%
5. The shape and size of the Ni-Co alloy coating were observed and determined using a Scanning Electron Microscope (SEM). The percentage of deposits was determined using an energy-dispersed X-ray (EDX) coupled with the SEM. It was found the amount of Co particles depends on the current density, temperature, pH, and the amount of Co particles in the bath. By setting these parameters as (2-6A/dm<sup>2</sup>,55oC pH 8,60 min)

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