# Hydrogen Desorption and Internal Stress in Nickel Coatings Obtained by Periodic Electrodeposition

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### ABSTRACT

Periodic electrodeposition is performed in order to determine to what extent a change in conditions for the desorption of hydrogen from nickel deposits, which is occluded during plating, can affect their internal stress. The duration of the pause (zero current),  $\tau_{off}$ , is varied in the range 0-900 s and the plating period,  $\tau_{on} = 120$  s, is kept constant, maintaining the structure unchanged. The variations of internal stress, microhardness, and coercivity of the electrodeposits obtained at different  $\tau_{off}$ , are investigated. The amounts of hydrogen leaving the coating, both during the pauses and after the deposition is completed, are determined. A proof of the mechanical nature of intensive hydrogen codeposition at the beginning of nickel electroplating is obtained. It is shown that the desorption of hydrogen is responsible to a considerable extent for the high tensile internal stress observed in similar coatings.

It is presumed that the electrodeposition of nickel from sulfate solutions is preceded by hydrogen evolution and starts as soon as hydrogen ion (H<sup>+</sup>) discharge becomes diffusion controlled.<sup>1-4</sup> The codeposition of hydrogen during the electrocrystallization of nickel is important because it has an influence on the structure and properties of the product film. On the one hand, H<sup>+</sup> discharge is the reason for the appearance of different inhibiting species (*e.g.*, hydroxides), which affect the structure and especially the preferred orientation of the crystallites.<sup>1,5</sup> On the other hand, a part of the codeposited hydrogen can be occluded into the coating and may, in this way, affect some of its properties. The presence of 2-butyne-1,4-diol enhances hydrogenation, a fact which has been proven by measurements after the plating is completed.<sup>6</sup> The effect of hydrogen occlusion during deposition is not investigated *in situ* in the process of nickel plating, however.

The presence of 2-butyne-1,4-diol in the electrolyte increases the internal stress (IS) in nickel coatings. It is of interest to determine to what extent change in the conditions for hydrogen desorption from the deposit can affect its IS. This is the reason for applying a periodic (interrupted) electrodeposition (PE), which is similar but not identical to pulse plating at very low frequency (Fig. 1). The plating period,  $\tau_{on} = 120$  s, is kept constant; the duration of the pauses (zero current),  $\tau_{\rm off}$ , is variable in the range 0-900 s: thus the duty cycle  $[\tau_{on}/(\tau_{on} + \tau_{off})]$  varies from 1 to 0.118. High current densities, at which the mass transport of the reacting ion becomes rate controlling, are not used here, however, and the values of  $\tau_{on}$  and  $\tau_{off}$  are considerably longer than those usually applied during pulsed plating.<sup>7-10</sup> This selection is made in order to meet the requirement that PE should not change substantially the structure of the coating but should only affect the hydrogen desorption conditions. In the case of cobalt the same PE ensures an increase of the IS without an alteration of the deposit structure (phase composition, texture, and grain size).<sup>11</sup> It has been shown that the reason for the variation of the IS is the desorption of hydrogen, codeposited during cobalt plating.

Applying PE and studying IS during nickel electrodeposition is aimed in this work at investigating the particular features of hydrogen occlusion in the coatings and its desorption *in situ*, as well as the influence of these phenomena on the properties.

#### Experimental

Plating and properties studies.—Nickel coatings  $\approx 23 \,\mu m$ thick were deposited from a Watts bath containing (mol/ dm<sup>3</sup>): NiSO<sub>4</sub> · 6H<sub>2</sub>O, 1.0; NiCl<sub>2</sub> · 6H<sub>2</sub>O, 0.21; H<sub>3</sub>BO<sub>3</sub>, 0.56; 2-butyne-1,4-diol,  $5.8 \times 10^{-4}$  (0.05 g/liter) at a cathodic current density (during  $\tau_{on}$ ) of 5 A/dm<sup>2</sup>,  $T = 56^{\circ}$ C, and pH 3.7.

<sup>a</sup> Present address: Department of Materials Science and Engineering, The Johns Hopkins University, Baltimore, MD 21218. For each deposition a newly prepared solution was used to make sure that the 2-butyne-1,4-diol concentration remains fixed and all experiments are conducted under identical conditions. The electrodeposition was carried out in a double chamber electrolytical cell with a volume of 700 cm<sup>3</sup>, kept under continuous circulation of the electrolyte.

Samples, electrodeposited onto copper substrates ( $20 \times 10 \text{ mm}$ ) were subjected consecutively to three types of investigations: structural studies and magnetic and microhardness measurements. Before being used the substrates were mechanically polished, degreased, and plated with fine-grain bright copper ( $\approx 25 \text{ µm}$  thick) with practically random crystalline orientation. In order to obtain uniform thickness of the nickel coating over the sample area, an additional screen of copper wire was used. It follows the rectangular form of substrate being located at a distance 2 mm from it. A similar screen was applied when IS were dipped in 10% H<sub>2</sub>SO<sub>4</sub>.

The Vickers microhardness was determined perpendicular to the coating surface with a Leitz Wetzlar unit applying a 0.050 kg load. The magnetic properties were investigated using a B-H loop meter described in Ref. 13. Several experiments under the same conditions were conducted to ensure the required reproducibility. The preferred crystalline orientation was checked analyzing the pole chart, traced by the Schulz method.<sup>14</sup>

Determination of the internal stress and the amount of desorbed hydrogen.—IS was determined by the dilatometric (length change) method using an instrument described in details in Ref. 12 and 15. The cathode was a stretched platinum ribbon with thickness  $t_0$  (0.2 mm) and initial before deposition) length  $l_0$  (85 mm), clamped at one end. The other end was free to change its position. On both sides of the ribbon a coating with thickness t was deposited (Fig. 2).



Fig. 1. Periodic electrodeposition (PE).

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Fig. 2. Strained state and length change of the coating-substrate system in the case of dilatometric method for IS measurement: 1, the coating is imagined as being separated from the substrate; 2, elastic interaction between substrate and coating; (a) tensile IS; (b) compressive IS.

If tensile IS is present in the coating, and we imagine that it is detached from the substrate, it should reduce its length since it has been stretched before (Fig. 2a, 1). In the real case due to the IS in the coating on both sides of the ribbon, a force, F, is initiated. The balance of the forces is achieved, reducing the initial length of the substrate. In the case of compressive IS in the coating an elongation of the substrate should be observed (Fig. 2b). The change of the substrate length at the moment when plating is over,  $\lambda$ , is used for IS determination. The average stress value through the thickness during electrodeposition,  $\overline{\sigma}$ , is determined as follows (see Ref. 12 and 15 for details)

$$\overline{\sigma} = -\frac{\widetilde{E}_o t_o \lambda}{2t l_o}$$
[1]

where  $\widetilde{E}_{o} = E_{o}/(1 - v_{o})$  is the reduced elasticity modulus of the platinum substrate ( $v_{o}$  is its Poisson coefficient).

An increase of the IS after switching off the electrodeposition current was established when plating cobalt, nickel, and palladium (see Ref. 11 and references therein). This effect has been related to the desorption of hydrogen, occluded in the coating during deposition. The essence of this phenomenon is that, after the hydrogen desorption, a free volume appears in the coating. This causes a decrease of the coating size<sup>16</sup> and is equivalent to a preliminary stretching. [Using transmission electron microscopy (TEM) a part of the hydrogen, which is occluded during the electroless deposition of copper, can be observed: small (20-300 Å) gas bubbles incorporated uniformly throughout the film and large (~2000 Å) bubbles trapped at the grain boundaries.<sup>17</sup> The rest of the hydrogen is dissolved in the crystal lattice, expanding it.] The dilatometric technique is very convenient to trace postelectrodeposition IS changes.

Let us suppose that tensile IS appears in the coating, *i.e.*, it is expanded and tends to contract. Under this influence, the length of the substrate will decrease (Fig. 2). As mentioned above, in some cases the electrodeposition of metals is accompanied by hydrogen evolution. Hydrogen, when occluded in the coating, will cause its length to increase. This effect is in the reverse direction to the tensile IS in the plate, and it is similar to the initiation of compressive IS in it. In the same way deliberate hydrogenation causes compressive IS in the coatings.<sup>19,20</sup> Monitoring the kinetics of postelectrolysis increase of IS, it is possible to determine the effective diffusivity of hydrogen.<sup>18</sup> For electrodeposited nickel typical values:  $(2.3 \pm 0.7) \times 10^{-9}$  cm<sup>2</sup> s<sup>-1</sup> are in good agreement with published data, obtained by alternative methods. Evaluation of the amount of hydrogen lawing the coating immediately after the plating is also possible.<sup>18</sup>

When PE is used,  $\lambda = \Sigma \lambda_{off}^{i} + \Sigma \lambda_{off}^{i}$  (Fig. 3), the length change of the substrate-coating system as time elapses during the plating periods  $\tau_{on}$  ( $\Sigma \lambda_{on}^{i}$ ) is due to IS, created as a result of the electrodeposition  $-\overline{\sigma}_{on}$ . During the  $\tau_{off}$  periods



Fig. 3. Experimental curves of the cathode length change,  $\lambda$ , vs. time,  $\tau$ , during periodic electrodeposition of nickel coatings.  $\lambda$  < 0 due to the presence of tensile IS.

 $\Sigma\lambda_{off}^i$  is connected with IS caused only by the desorption of hydrogen,  $\overline{\sigma}_{off}$ . The average IS during PE is:  $\overline{\sigma} = \overline{\sigma}_{on} + \overline{\sigma}_{off}$ . The stationary average stress,  $\overline{\sigma}^{*,21}$  is determined by replacing  $\lambda^*$  instead of  $\lambda$  in Eq. 1, where  $\lambda^* = \lambda + \lambda_{\infty}$  and  $\lambda_{\infty}$  is the postelectrolysis length change until the stationary state of this process is reached.  $^{12,15}$ 

In order to evaluate the amount of hydrogen desorbed during the pauses at PE, expressed as atomic concentration (hydrogen atoms per nickel atoms),  $C_{\rm H}^{\rm off}$ , we must consider its fractions desorbed during each pause *i*:  $C_{\rm H}^{\rm off}$ . This quantity is desorbed from the entire coating deposited up to this moment. Thus on the basis of the equation for the total amount of hydrogen, desorbed from the coating after continuous plating,<sup>18,22</sup> for the quantity leaving the coating during PE pauses we obtain

$$C_{\rm H}^{\rm off} = \sum_{i=1}^{k-1} C_{\rm H_1}^{\rm off} = \sum_{i=1}^{k-1} \left[ \frac{3}{\Delta\Omega/\Omega} \frac{\lambda_{\rm off}^{\rm i}}{l_{\rm o}} \left( 1 + \frac{\widetilde{E}_{\rm o} t_{\rm o}}{\widetilde{E} \ 2t_{\rm i}} \right) \right]$$
[2]

where  $\Omega$  is the atomic volume of the coating material,  $\Delta\Omega$  is its change due to the addition of a hydrogen atom, k is the number of plating periods, (k-1), the number of pauses,  $2t_i$ =  $2t_{on}i$ , and  $t_{on}$  is the thickness of the coating deposited during  $\tau_{on}$ ,  $\tilde{E} = E/(1-\nu)$  is the reduced elasticity modulus of the coating ( $\nu$  is its Poisson coefficient).

If there is no substantial difference between the experimental values  $\lambda_{off}^{i}$  (±5-10%), one may replace  $\lambda_{off}^{i}$  by an averaged value  $\lambda_{off}^{a}$  = constant =  $1/(k-1)\sum_{i=1}^{k-1} \lambda_{off}^{i} = 1/(k-1) \lambda_{off}^{\Sigma}$  and obtain

$$C_{\rm H}^{\rm off} = \frac{3}{\Delta\Omega/\Omega} \frac{1}{l_{\rm o}} \left( 1 + \frac{\widetilde{E}_{\rm o} t_{\rm o}}{\widetilde{E}} \frac{1}{2t_{\rm on}(k-1)} \sum_{i=1}^{k-1} \frac{1}{i} \right) \lambda_{\rm off}^{\rm z}$$
[3]

In the latter case the values calculated using Eq. 2 and 3 are quite similar (the difference is a few percent), and the application of Eq. 3 is recommended. Knowing  $C_{\rm H}^{\rm off}$ , it is easy to calculate the amounts of hydrogen leaving the coating during the pauses, expressed in cm<sup>3</sup>/g Ni:  $v_{\rm H}^{\rm off}$  (see Ref. 11, 18, 22). The quantity of hydrogen, desorbed from the coating after the deposition (cm<sup>3</sup>/g Ni),  $v_{\rm H}$ , can also be evaluated.<sup>11,18,22</sup>

### **Results and Discussion**

One can see in Fig. 3 that there is a compressive IS at the initial stage of nickel plating, using continuous current ( $\tau_{off} = 0$ ). When we apply PE, this effect is repeated. Presumably the influence of severe hydrogenation reaches its peak after about 60 s  $\approx 1/2 \tau_{on}$ . Tensile IS appears during the pauses due to hydrogen desorption. The appearance of compressive IS prevents the vanishing of steps and smoothening of the  $\lambda vs$ . time curve, which is observed in the case of cobalt for short  $\tau_{off}$ , where only tensile IS is



Fig. 4. Relationships of (a) total amount of desorbed hydrogen, v<sub>H</sub>; (b) amount of hydrogen desorbed after electrodeposition is over, v<sub>H</sub>; (c) average IS during coating formation,  $\overline{\sigma}$ , average IS during plating periods only,  $\overline{\sigma}_{on}$ , stationary average IS,  $\overline{\sigma}^*$ , average IS during pauses,  $\overline{\sigma}_{off}$ , coercivity,  $H_c$ , and microhardness,  $H_v$ , vs. the duration of pause,  $\tau_{off}$ , during the periodic electrodeposition of nickel. The duty cycle varies from 1 to 0.118 for  $\tau_{on} = 0$  and 900 s, respectively.

present.<sup>11</sup> This fact suggests that a severe hydrogenation of nickel occurs before metal deposition starts. This is further evidence of mechanical nature for the existence of this phenomenon, as discussed in the literature.<sup>1-5</sup> When cobalt is plated, hydrogen evolution and metal deposition proceed simultaneously. A similar effect is also observed in the case of nickel, but the initial hydrogenation in the presence of 2-butyne-1,4-diol is predominant.

It is seen in Fig. 4 that the hydrogen desorbed during the pauses,  $v_{\rm H}^{\rm eff}$ , is the major portion of the total amount leaving the deposit,  $v_{\rm H}^{*}$ ,  $(v_{\rm H}^{*} = v_{\rm H}^{\rm eff} + v_{\rm H})$ . During the pauses a passivation or poisoning of the growth sites due to adsorption of anions (or other foreign species) is presumed. Such a deactivation of the growth sites implies that a renucleation process should occur, initiating the deposition during the next  $\tau_{\rm on}^{23}$  Thus the initial evolution of hydrogen is repeated, increasing its amount evolved during PE as compared with continuous current plating.

The quantity of hydrogen desorbed after the deposition is over is reduced (by 30-40%), when PE is applied (Fig. 4b), since hydrogen has the chance to leave the coating during the pauses. This value remains practically unchanged after  $\tau_{off} > 240$  s, whereas in the case of cobalt  $v_{H}$  is gradually decreased in the entire pause interval.<sup>11</sup> Generally, the difference between cobalt and nickel when plated using PE is that the properties of the former *vs.*  $\tau_{off}$  display a gradual change (no kinks in the curves),<sup>11</sup> whereas in the case of the latter the corresponding curves have kinks (Fig. 4). We suppose the reason for this is the larger amount of hydrogen codeposited with nickel at the beginning of plating and after every pause, when PE is applied.

Also due to the hydrogenation  $\overline{\sigma}_{on}$  becomes compressive (negative), and it is only the desorption of hydrogen during the pauses that makes the overall IS,  $\overline{\sigma}$ , tensile:  $\overline{\sigma} = \overline{\sigma}_{on} + \overline{\sigma}_{off}$  (Fig. 4c). The compensation of  $\overline{\sigma}_{on}$  by  $\overline{\sigma}_{off}$  leads to a very slight increase of  $\overline{\sigma}$  with pause duration. The decrease of  $\overline{\sigma}_{on}$  with  $\tau_{off}$  is due to the enhanced desorption of hydrogen during longer pauses ( $\overline{\sigma}_{off}$  rises): then there is more room for new hydrogenation, and  $\overline{\sigma}_{on}$  becomes more negative.

As in the case when PE is applied to plate cobalt,<sup>11</sup> there are no severe structural changes in nickel electrodeposited at different  $\tau_{off}$ . The texture remains <100> + <211> as previously observed with continuous current plating.<sup>24</sup> The hysteresis loop is very narrow and inclined: typical for the presence of tensile stress in materials with negative magnetostriction.<sup>25</sup> After applying PE, an increase of the average IS ( $\approx 14\%$ ) occurs as well as a corresponding rise of coercivity ( $\approx 15\%$ ), since magnetoelastic energy is proportional to the IS value (Fig. 4c). The hardness slightly decreases when PE is applied, but nevertheless it does not depend on  $\tau_{off}$ .

So the possibility for hydrogen to go in and out of the deposit during pulse plating can modify additionally some of its properties. This particular feature has to be taken into account when we consider the alteration of properties with frequency during pulse plating. In nickel deposited from Watts and chloride baths an increase of IS has been observed in pulse plating with low duty cycles.<sup>9</sup> The picture is similar for nickel plated from sulfamate bath with a duty cycle of 0.0909 and at frequency 9.2 Hz.<sup>9,26</sup> Zabludovskij<sup>8</sup> believes that at a given rate of voltage increase (20-25 V/s) during pulse deposition of nickel coatings the hydrogenation is possible at frequencies lower than 30-40 Hz. The microhardness of nickel electrodeposits prepared under pulse current reversal (PCR) at 0.1 Hz and duty cycle 0.95 is almost twice as high, as compared with those plated at frequencies  $\geq 1$  Hz.<sup>27</sup> It can be presumed that the short anodic pulse (0.5 s) clears the surface, and then the process starts with hydrogenation. At 0.1 Hz hydrogen has 9.5 s for the penetration in the deposit, *i.e.*, ten times longer than at 1 Hz. The increase both of hardness and hydrogen content in electrodeposited Ni-P amorphous alloys after applying PCR as compared with those obtained under dc was observed recently.28

Thus the desorption of hydrogen, occluded during nickel plating, is responsible to a considerable extent for the observed high tensile IS in similar coatings. Another evidence for a similar statement is the influence of different surfactants upon the hydrogenation and the IS value. It was established<sup>6</sup> that the I class brighteners (*e.g.*, saccharin) known as IS reducers,<sup>29</sup> inhibit the hydrogenation, and the II class ones (*e.g.*, 2-butyne-1,4-diol), which increase tensile IS,<sup>30</sup> promote the hydrogenation of coatings. It was found also that the addition of saccharin in the bath decreases by one-fifth the content of hydrogen occluded in nickel deposits.<sup>31</sup>

The depletion of 2-butyne-1,4-diol during the operation of our bath without correction of its concentration leads to a decrease both of the hardness and the coercivity of deposits obtained by PE (Fig. 5). The reduction of surfactant content decreases hydrogenation. There is less hydrogen in the coating, which decreases hardness as well as hydrogen desorption, reducing correspondingly IS and coercivity.

## Conclusions

Evidence (of nonelectrochemical but rather of a mechanical nature) is obtained for the intensive hydrogen evolution during the initial stage of nickel electroplating. Each of the two modes of hydrogen codeposition during plating (especially when not continuous) can predominate: before starting metal electrodeposition or during the process. In the case of nickel plating, in the presence of 2-butyne-1,4diol in the electrolyte the first mode is prevailing. This is the reason for the absence of a strong dependence of properties on the pause duration (duty cycle) after PE is applied, but nevertheless there is a difference between the continuous and the PE. In the case of cobalt the second mode is dominating, and there is a smooth relationship between properties and pause duration. For the value of hardness the amount of hydrogen absorbed in the deposit is significant. For the tensile IS level the quantity of hydrogen desorbed from the coating is important. Thus the phenomena of hydrogen occlusion and desorption may have an additional effect on the mechanical properties of deposits obtained by pulse plating at low duty cycles and low frequencies as well as when PCR with low frequencies and high duty cycles is applied.



BATH OPERATION DURATION [A\*h/I]

Fig. 5. Influence of the bath operation duration (amperes  $\times$  hours/ liter) without correction of 2-butyne-1,4-diol concentration on the microhardness (a) and the coercivity (b) of obtained coatings.

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