

STATE ESTIMATION AND BATH CONTROL FOR THE ELECTROLESS NICKEL-PLATING PROCESS

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Keywords: Electroless plating, modelling, estimation, control.

Abstract

A mixed-potential model is proposed for the electroless nickel-plating process. It is tested against measured data and used for monitoring of the thickness and phosphorous content of plating film. The bath-loading process with strong effect on chemical reactions is evaluated as Markov pure jump process and applied in control. The thickness and phosphorous content are stabilized at desired levels using electrochemically balanced tracking trajectory for pH-index and nickel concentration. The latter processes are controlled at the balanced trajectory using the precise controls developed.

1 Introduction

Electroless nickel plating is an established process in many industries including electronics, oil and gas industry, motor industry, machinery, etc. Usually it does not require advanced control like in microelectronics (e.g. PTH-board and micro-processors manufacturing) where contactor thickness is measured in microns and they are packed tightly together. They are made electrically isolated using precise technology.

The purpose of the paper is to develop precise controls for the electroless nickel-plating technology for stabilisation of the thickness and phosphorous content of the plating film at desired levels using a mixed-potential model developed for evaluation of the unobservable processes.

The electroless nickel plating has been analysed in both the chemical reaction theory and electrochemical reaction theory [8]. The mixed-potential model [6] developed in the latter is similar to the electrochemical cell model originally developed for batteries [4]. In addition to the mixed-potential model, the cell model is applied in this paper for plating process estimation and control.

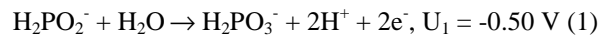
The plating chemistry is affected strongly by bath loading which is an unobservable jumping process. The estimation and control theory for a linear process with jump parameters in a non-linear relationship has been analysed in [3]. However, the model of plating process is different – it is non-linear by observable state variables and linear by unobservable jump parameters. In this case, non-linear filtration theory developed for a finite-state Markov process [7] can be applied to convert

the partially observable plating process into completely observable process and solved approximately as a regulator problem as shown in [1], [2].

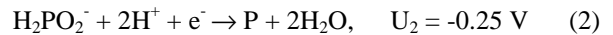
2 Model

Several reaction mechanisms have been proposed for plating chemistry [8], including an electrochemical reaction mechanism, which was found to match with measured data (also confirmed in this paper). This mechanism can be represented as the following system of anodic and cathodic reactions.

Anodic reaction - hypophosphite oxidation



Cathodic reactions - phosphorous deposition, hydrogen evolution, nickel deposition



The pH-index is controlled in plating process through feeding of ammonia, which is a weak base – its dissolution with water gives a strong conjugate acid ammonium that does not ionize completely in water.

The discharge of hydrated nickel ions in aqueous solution is subject to a series of transformations and displacements. Although these steps play an important role in the electrochemical behavior, they are not rate determining. The discharge reaction is controlled by the deposition process. This reaction can be represented as the following electrochemical cell model, originally developed for a porous electrode [4].

2.1. Electrochemical cell model

The discharge reaction on lattice metal is controlled by a mixed potential formed from the equilibrium potentials of the partial reactions through the electrode reaction. The current densities of the electrode reactions (1)-(4) can be represented as single-directional model processes: anodic reaction model for oxidation and cathodic reaction model for others

$$i_1 = i_{01}\mu_1 \exp(\alpha_1 2k\eta_1), \quad i_n = -i_{0n}\mu_n \exp(-\alpha_n p_n k\eta_n), \quad (5)$$

where

n - reaction number: 1 - oxidation, ..., 4 - nickel deposition,

i_n - current density, A/cm²,

i_{0n} - exchange current density, A/cm²,

η_n - overpotential, $\eta_n = \phi - u_n$, ϕ - mixed potential, V,

u_n - thermodynamic equilibrium potential, V,

μ_n - dimensionless concentration of species,

k - temperature voltage, $k = F/RT$, $1/V$, T - temperature, K,

R - universal gas constant, 8.3145 J/mol-K,

F - Faraday's constant, 96487 C/mol,

p_n - number of exchanged electrons, $p_n = 2$, $p_2 = 1$,

α_1 - anodic, α_n - cathodic apparent transfer coefficients.

The anodic current density is equal to the sum of cathodic current densities, i.e. the electrical neutrality requirement

$$i_1 + i_2 + i_3 + i_4 = 0. \quad (6)$$

The mixed potential is a self-adjusting parameter - it maintains the electrical neutrality (6) irrespective to changes in the partial reactions like changes in concentrations or equilibrium potentials.

The equilibrium potentials of the partial reactions can be evaluated from the Nernst equation using unit activity for solid materials (deposited nickel and phosphorous) and water, and unit partial pressure of hydrogen

$$u_1 = U_1 + \kappa(\log c_1 - \log c_5 + 2\text{pH}), \quad (7)$$

$$u_2 = U_2 + 2\kappa(\log c_1 - 2\text{pH}), \quad (8)$$

$$u_3 = U_3 - 2\kappa \text{pH}, \quad (9)$$

$$u_4 = U_4 + \kappa \log c_4, \quad (10)$$

where c_n - ion concentration of species, mol/cm³, $\kappa^{-1} = 2k \log e$.

The phosphorous deposition rate decreases if the pH-index increases. The nickel deposition rate decreases if the nickel concentration decreases. This inhibition of the deposition rates can be accounted for as a limitation due to low concentration

$$\mu_1 = \frac{c_1}{c_{1\text{ref}}}, \quad \mu_3 = \frac{c_3}{c_{3\text{ref}}}, \quad \mu_2 = \mu_1 \mu_3, \quad \mu_4 = \frac{c_4}{c_{4\text{ref}}}, \quad (11)$$

where $c_{n,\text{ref}}$ - reference concentration of species, mol/cm³.

The nickel deposition rate is proportional to the current density (5) of the deposition reaction (4)

$$\frac{dx_{\text{Ni}}}{dt} = -\frac{i_4}{C_{\text{Ni}}}, \quad (12)$$

where

t - immersion time, sec,

x_{Ni} - partial thickness of nickel, i.e. ratio volume per plate area,

i_4 - current density of nickel deposition, A/cm²,
 C_{Ni} - charge density of nickel ions, 29258 C/cm³.

The deposition rate of phosphorous is proportional to the current density (5) of the deposition reaction (2)

$$\frac{dx_{\text{P}}}{dt} = -i_2 \frac{i_2}{C_{\text{P}}}, \quad (13)$$

where

x_{P} - partial thickness of phosphorous, cm,

i_2 - current density of phosphorous deposition, A/cm²,

C_{P} - charge density of phosphorous ions, 5665 C/cm³.

The thickness $x_a = x_{\text{Ni}} + x_{\text{P}}$ of Ni-P-alloy film can be calculated by integration of (12), (13) over the plating period (22 min) with respect to the deposition reaction current densities.

2.2. Concentration dynamics

Feeding and utilisation of the reagents along with formation of the by-products induces related reactions with effect on the concentrations of species.

1. Hypophosphite consumption

$$dc_1 = [A(2i_2 - i_1)/2F + Q_{1f}c_{1f}]dt + \sigma_1 dW, \quad c_1(t_0) = c_{1\text{ref}}, \quad (14)$$

c_1 - hypophosphite concentration, $c_{1\text{ref}} = 0.27$ mol/dm³,

c_{1f} - feeding solution concentration, 5.2 mol/dm³,

A - bath loading, 50-100 cm²/dm³,

Q_{1f} - hypophosphite dilution rate, 1/s,

t_0 - makeup moment for a new bath,

σ_1 - model inaccuracy,

W - model-prediction error: Wiener process.

2. Hydrogen formation, consumption and pH neutralization

$$dc_3 = [A(i_1 + 2i_2 + i_3)/F - K_r Q_{3f} c_{3f}]dt + \sigma_3 dW, \quad c_3(t_0) = c_{3\text{ref}}, \quad (15)$$

c_3 - hydrogen concentration, $c_{3\text{ref}} = 10^{4.8}$ mol/dm³,

c_{3f} - feeding concentration, 6 mol/dm³,

Q_{3f} - ammonia dilution rate, 1/s,

K_r - dissociation constant.

3. Nickel consumption

$$dc_4 = [Ai_4/2F + Q_{1f}c_{4f}]dt + \sigma_4 dW, \quad c_4(t_0) = c_{4\text{ref}}, \quad (16)$$

c_4 - nickel concentration, $c_{4\text{ref}} = 0.1$ mol/dm³, $c_{4f} = 1.9$ mol/dm³.

Accumulation of the orthophosphite as a by-product can be represented as

$$dc_5 = Ai_1 dt/2F + \sigma_5 dW, \quad c_5(t_0) = 0, \quad (17)$$

c_5 - orthophosphate concentration, mol/dm³.

The plating chemistry is rather complex and still not com-

pletely understood [8], especially with respect to the hydrogen (15), the formation, utilisation and evolution of which are more complicated than shown in the reactions (1)-(4). The Weiner processes are introduced to deal with the inaccuracy of the models – they are applied to render the estimation and control strategy insensitive to inaccuracy.

2.3. Bath loading

The bath loading is defined as a total area of plates immersed in the solution per bath volume

$$A = zA_p, \quad (18)$$

where

A_p - overall plates area per bath volume (constant), cm^2/dm^3 ,
 z - loading level: $z = Z/N$, Z - number of baskets immersed,
 N - maximum number of baskets in use, $N = 2$ or 3 .

2.4. Measurements

Typically two types of analysers are used in a plating process control in industry.

1. Nickel analyser

$$\xi_4 = 10^3(c_4 + r_4\epsilon), \quad (19)$$

where

ξ_4 - measured nickel concentration in relative units, %,
 r_4 - analysis accuracy,
 ϵ - analysis error: standard Gaussian variable.

2. pH analyzer

$$\xi_3 = 10^{-\text{pH}} = c_3 + r_3\epsilon, \quad (20)$$

where ξ_3 - measured hydrogen concentration, $\text{pH} = -\log \xi_3$.

Beside these standard analyses the flow rates of ammonia Q_{3f} and hypophosphite/nickel Q_{1f} are measured.

3 State estimation

The deposition rate depends on the bath loading, which is a finite-state pure jump process that can be specified as a simple birth and death process [5] with two parameters characterizing loading and interruption (equipment service) rates. These parameters can be estimated statistically from the measured loading process. In the simplest case of single basket they can be evaluated from the average plating time $\tau = 22$ min and mean loading $Mz = 0.8$ using the simplest relationship $\tau = \mu^{-1}$, $Mz = \mu/(\mu + \lambda)$ as the loading rate $\lambda = 5.5 \text{ h}^{-1}$ and interruption rate $\mu = 2.7 \text{ h}^{-1}$. In general other properties of the birth and death process should be applied. They can be specified as properties of a Markov pure jump process with $N+1$ -non-absorbing states. This process satisfy the following Kolmogorov forward equation [5] for the transition probabilities

$$\frac{dp_{xy}}{dt} = q_{y,y-1}p_{x,y-1} + q_{yy}p_{xy} + q_{y,y+1}p_{x,y+1}, p_{xy}(0) = \delta(x-y), \quad (21)$$

where

x, y - loading levels: $0, 1, \dots, N$,

p_{xy} - transition probability that loading process started in the state x at moment t will be in the state y at moment $s \geq t$,

q_{yx} - infinitesimal operator parameters defined through the loading and interruption rates as the 3-diagonal matrix elements:

Lower diagonal: $q_{y,y-1} = \lambda, 1 \leq y \leq N$,

Upper diagonal: $q_{y,y+1} = \mu, 0 \leq y < N$,

Diagonal: $q_{00} = -\lambda, q_{yy} = -\lambda - \mu, 1 \leq y < N, q_{NN} = -\mu$.

The loading process beside other unobservable processes can be evaluated from measured data as the following partially observable process with observable (19), (20) and unobservable (14), (17) components represented as the system

$$Z_t - \text{finite-state Markov process}, \quad (22)$$

$$d\theta = [a_1 u_1(t) + a_2(i)Z_t]dt + bdw, \quad (23)$$

$$d\xi = [G(\xi_t)u_t + A(i)Z_t]dt + Bdv, \quad (24)$$

where

Z_t, θ_t - unobservable processes,

ξ_t - observable process,

a_1, a_2, G, A - system elements shown in Tables 1, 2, $\omega = A_p/NF$,

b_1 - model accuracy: diagonal matrix in Table 1,

B - model and measurements accuracy: matrix in Table 2.

	θ	a_1	u	$2a_2/\omega$	diag b
Hypophosphite	c_1	c_{1f}	Q_{1f}	$2i_2 - i_1$	σ_1
Orthophosphite	c_5	0	0	i_1	σ_5

Table 1. Specification of the unobservable processes.

	ξ	diag G	u	diag A/ω	diag BB
Hydrogen	ξ_3	$-K_1 c_{3f}$	Q_{3f}	$i_1 + 2i_2 + i_3 - i_4$	$\sigma_3^2 + r_3^2$
Nickel	ξ_4	c_{4f}	Q_{4f}	$2i_4$	$\sigma_4^2 + r_4^2$

Table 2. Specification of the observable processes.

The current densities shown in Tables 1, 2 are dependent on each other through the mixed potential; they depend also on the concentrations of used chemicals through the limitation coefficients (11) and equilibrium potentials (7)-(10). This is an implicit relationship that can be solved numerically with respect to the current densities. Analytically it can be represented as a structure. For example, the nickel deposition current can be represented as the following structure

$$i_4 = F\{i_1(c_1, c_3, c_5), i_2(c_1, c_3), i_3(c_3), i_4(c_4), c_4\}$$

that can be simplified by replacement of the observable processes c_3, c_4 with available measurements ξ_3, ξ_4 and unobservable processes $\theta = [c_1, c_5]^T$ with conditional average value $m = \mathbf{M}\{\theta_t | F_t^\xi\}$. As a result of these replacements the nickel deposition current can be represented as a progressively measur-

able process with respect to σ -algebra of Wiener and Poisson processes and therefore adapted to the measured processes. Similarly adapted processes are the system coefficients $a_2(i) = a_2(t, \xi)$, $A(i) = A(t, \xi)$ because the same ideas of measurable functions can be applied with respect to the other current densities in Tables 1, 2. Eventually the system (22)-(24) can be represented as a Markov process with respect to the unobservable process (Z_t, θ_t) and non-anticipative process with respect to the observable process (ξ_t)

$$d\theta = [a_1 u_t + a_2(t, \xi) Z_t] dt + b dw, \quad (25)$$

$$d\xi = [G(\xi_t) u_t + A(t, \xi) Z_t] dt + B dv. \quad (26)$$

The estimation problem is to evaluate the state of loading process and the hypo- and orthophosphite concentrations using available measurements of the pH-index and nickel concentration as well as the ammonia and nickel feeding rates.

The state of loading process can be evaluated based on the filtrated probabilities; they satisfy [7] the following filtration equation obtained from the partially observable processes (25), (26)

$$d\pi = \Lambda \pi_t dt + (D - k_t I) \pi_t A^T(t, \xi) B^{-1} dW^u, \quad \pi(0) = \delta(y - MZ), \quad (27)$$

where

π_y - probability that y baskets are immersed at moment t ,
 W^u - innovation process with new measurements: Weiner process

$$dW^u = B^{-1} \{d\xi - [G(\xi_t) u_t + A(t, \xi) k_t] dt\}, \quad (28)$$

Λ - infinitesimal matrix operator, $\Lambda = \{q_{yx}: x, y = 0, 1, \dots, N\}$,
 I - unity matrix, D - diagonal matrix: $D = \text{diag}(0, 1, \dots, N)$,
 k_t - loading level: conditional average value, $k_t = \mathbf{M}\{Z_t | F_t^\xi\}$.

The loading process can be evaluated as a least-square estimate or maximum likelihood estimate. The least-square estimate is an average value of the loading process calculated as a sum of the filtrated probabilities $k_t = L^T \pi_t$ weighed with loading levels $L = [0, 1, \dots, N]^T$. The maximum likelihood estimate is equal to the loading level corresponding to the biggest filtrated probability.

A future loading program required for control can be forecasted on average $k_{s/t} = L^T \pi_{s/t}$, $s \geq t$ using extrapolated probabilities $\pi_{s/t} = e^{\Lambda(s-t)} \pi_t$.

The hypo- and orthophosphite concentrations can be evaluated on average as a solution of the following filtration equation obtained from the processes (22), (25), (26)

$$dm = [a_1 u_1(t) + a_2(t, \xi) k_t] dt + \gamma_{21} A_1^T(t, \xi) B^{-1} dW^u, \quad (29)$$

where m_t - vector of hypo- and orthophosphite concentrations. This is the least-square estimate $m = \mathbf{M}\{\theta_t | F_t^\xi\}$ with covariance $\gamma_{21} = \text{cov}\{Z_t, \theta_t | F_t^\xi\}$ constant for simplicity. $A_1 = [A, 0]^T$.

4 Optimal tracking trajectory

The geometrical and physical properties of a PTH-board are defined with several parameters. Among them, the thickness of Ni-P-alloy film and phosphorous content are crucial. In plating process they are controlled through choice of references for pH-index and nickel concentration. The latter processes are kept at desired references with adjustment of the ammonia and hypophosphite/nickel feeding rates.

The desired thickness of plating film is a constant $x_a = 4 \mu\text{m}$ as well as the desired phosphorous content $P_{\text{vol}} = 31 \%$. The partial thicknesses of nickel and phosphorous can be calculated as target values for control from the desired levels $x_{\text{Ni}}^* = x_a - x_p^*$, $x_p^* = x_a P_{\text{vol}}/100$. These references can be achieved if an electrochemically balanced tracking trajectory is used for the pH-index and nickel concentration adjustment along with the mixed potential. This triple, if optimal, satisfies the electrochemical system, which includes the electrical neutrality equation (6) solved with respect to electrode reactions (5) and thermodynamic equilibrium potentials (7)-(10). It also includes the deposition rate requirements derived from the deposition rates (12), (13) based on the target values for thickness of nickel x_{Ni}^* and phosphorous x_p^*

$$-\frac{i_4}{C_{\text{Ni}}} = \frac{x_{\text{Ni}}^*}{\tau}, \quad -\frac{i_2}{C_p} = \frac{x_p^*}{\tau}, \quad (30)$$

The balanced trajectory is a triple for the pH-index, nickel concentration and mixed potential calculated as adjoint processes in dependence on the hypo- and orthophosphite concentrations or on their estimated values

$$\zeta_3 = c_3(m_1, m_5), \quad \zeta_4 = c_4(m_1, m_5), \quad \phi = \phi(m_1, m_5). \quad (31)$$

The plating process is controllable as long as this relationship between target values holds. Otherwise an automatic change of the mixed potential takes place with effect on all partial reactions, even if the pH-index and nickel concentration are kept constant.

5 Bath control

The thickness and phosphorous content can be maintained at the desired levels through stabilization of the pH-index and nickel concentration at the electrochemically balanced trajectory. This tracking problem can be formulated as a minimization problem of the square functional

$$v^u = \mathbf{M}^u \int_{t_0}^{T_0} \{(\xi_t - \zeta_t)^T (\xi_t - \zeta_t) + (u_t - v_t)^T \Psi (u_t - v_t)\} dt \quad (32)$$

with respect to the partially observable stochastic process (22), (25), (26).

Here

ζ - desired trajectory for pH-index and nickel percentage,
 u - controls: ammonia and nickel feeding rates,

v - setpoint for controls,

Ψ - cost of agitation: positively defined matrix of weights,

T_0 - control horizon equal to the bath age of 2-3 days.

This incomplete data control problem can be converted to complete data and solved as an equivalent control problem of minimising the square functional (32) with respect to the completely observable process (27)-(29) as solved in a separated control problem with estimation independent of the controls. Although minimization of (32) with respect to the filtrated (27)-(29) and original processes (22), (25), (26) are equivalent, some simplifications are required to obtain controls in an explicit form suitable for practical implementation. They are introduced to ignore the nonlinear and random behavior of the system (27)-(29) partially.

1. The electrode reaction equation (6) is approximated with linear model with respect to the controllable processes. This allows linearization of the matrix function $A(t, \xi) = k_t(f_t + F_t \xi_t)$.
2. The future values of the current densities are assumed to be predictable through the current values $\mathbf{M}\{i(s) | F_t^{\xi}\} \approx i(t)$, $s \geq t$. This inaccuracy is small, because the current densities are relatively constant. For the same reason the future values of the non-stationary coefficients f_s , F_s are also predictable through the current values f_t , F_t , $s \geq t$.
3. The vector f_t and matrix F_t functions are considered as deterministic - independent of Poisson and Weiner processes.

Eventually the controllable process can be represented as a linear stochastic process with jumping coefficients

$$d\xi = [k_t(f_t + F_t \xi_t) + Gu_t]dt + BdW^u. \quad (33)$$

This simplified control problem (32), (33) can be solved analytically as an optimal regulator problem for a linear completely observable stochastic system [1], [2]. The control law obtained can be represented as

$$u_t = -L^T \pi_t G^{-1} f_t - \Psi^{-1} G^T (P \xi_t + p) \quad (34)$$

where

ξ - measured pH-index and nickel percentage,
 p , P - solution of the Riccati equations for tracking gain

$$-\frac{dP}{ds} = k_{s/t}(PF_t + F_t^T P) - PG\Psi^{-1}G^T P + I, \quad (35)$$

and level

$$-\frac{dp}{ds} = (k_{s/t}F_t - G\Psi^{-1}G^T P)^T p - \zeta_t, \quad (36)$$

$k_{s/t}$ - loading forecast $k_{s/t} = L^T e^{\Lambda(s-t)} \pi_t$.

The control horizon that covers several days is a too long period for fast calculations. At the same time, solution of the Riccati equation is a constant for gain and tracking level if the forecast time exceeds the plating period τ . If the bath loading process is forecasted analytically then the Riccati equations (35), (36) can be initialised without any loss of accuracy with solutions $p(\tau)$, $P(\tau)$ of the algebraic equations

$$(L^T e^{\Lambda\tau} \pi_t F_t - G\Psi^{-1}G^T P)^T p - \gamma_t = 0, \quad (37)$$

$$L^T e^{\Lambda\tau} \pi_t (PF_t + F_t^T P) - PG\Psi^{-1}G^T P + I = 0 \quad (38)$$

adapted to the loading forecast at every moment $t_0 \leq t \leq T_0$. This makes the control algorithm fast; it is similar to the deterministic control but depends on the random coefficients through loading program. The industrial PID controller is less rapid in tracking of the bath loading and other changes in a plating process.

The proposed control is stable because the loading and tracking trajectories are bounded and the pair $(k_{s/t}F_t, G)$ is completely controllable for any predicted $s \geq t$ and current $t \geq t_0$ moments. The included simulation experiment is a simple proof of stability.

7 Experiments

The prediction accuracy evaluated from the mass balance as overall characteristic of all models applied is shown in Figs 1,2.

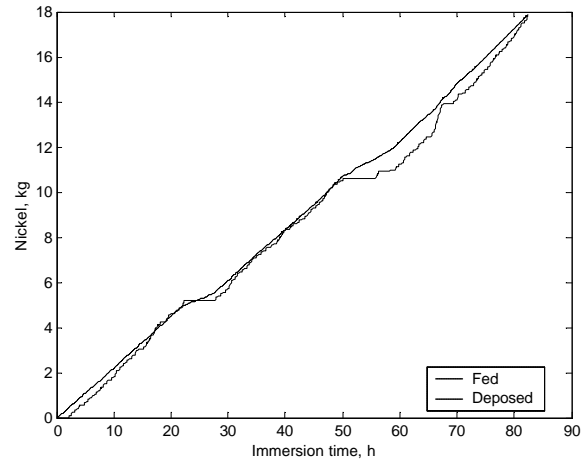


Fig. 1. Model-predicted and measured weight of nickel deposited on plates and fed in bath with nickel sulphate.

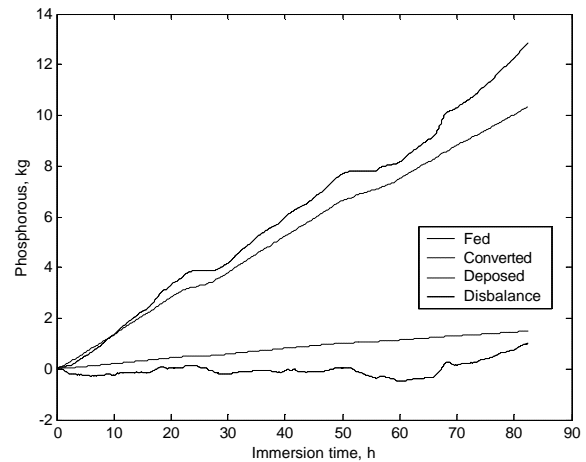


Fig. 2. Model-predicted and measured weight of phosphorous deposited on plates, converted in orthophosphite and fed in bath with hypophosphite.

It was evaluated as overall weight of nickel removed with produced plates (predicted weight) compared with nickel sulphate fed in bath (measured weight). The overall weights of phosphorous removed with plates and converted in orthophosphate (predicted weights) are compared with hypophosphite fed in bath (measured weight). The model-predicted weight calculated by several unobservable processes is close to measured weight in Figs 1, 2.

7 Control simulation

The bath-loading process simulated as a Markov pure jump process and filtrated from the measured data as the least-square estimate is shown in Fig. 3.

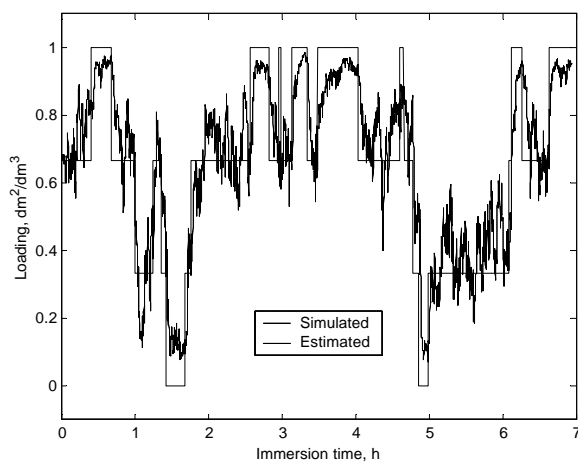


Fig. 3. Simulated and estimated bath loading.

Besides the bath loading the Wiener processes with 20 % intensity were included in every model to simulate inadequacy. Irrelevant to perturbations the plating film thickness and phosphorous content can be kept stable at the desired level with proposed controls as shown in Figs 4, 5.

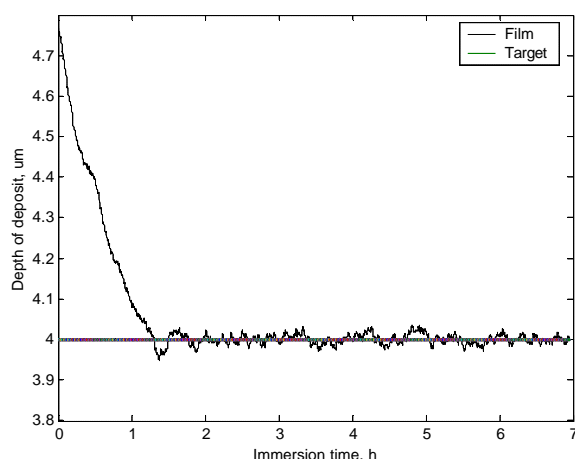


Fig. 4. Stabilised thickness of deposit.

The control effect obtained in the simulation experiment is essential. The real variation tested with industrial PID controller was about 10 times larger for thickness and 5 times large for phosphorous content.

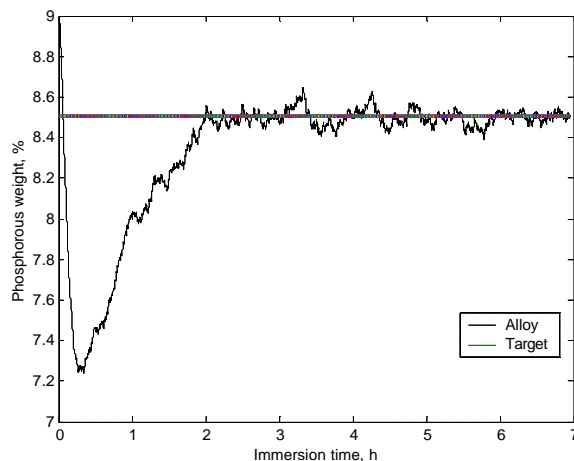


Fig. 5. Stabilised phosphorous content in alloy.

8 Conclusion

In this paper, it was shown that PTH-boards could be characterised online during nickel-plating process using standard analysers. The state can be controlled at the electrochemically balanced trajectory using improved control with respect to the industrial PID controller at the expense of use of more relevant plating process information (bath loading, chemical state, electrochemical state) than currently used.

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