CONTRIBUTIONS TO ELABORATE A MATERIAL BALANCE, AN ENERGETIC BALANCE AND AN OPTIMIZATION METHOD FOR NICKEL ELECTROPLATING PROCESS

[1]Violeta Vasilache, [2]Traian Vasilache, [1]Gheorghe Gutt

[1]University Stefan cel Mare of Suceava, [2]non affiliated e-mail: violetaren@yahoo.com, tel. 0723018740

Abstrac: *The paper set out the theoretical balance sheet of materials in the case of galvanic settlements, particularized for nickel electroplating process. The balance sheet equations have been introduced for mass balance and for energy balance, respectively. In order to optimize the mass and energy parameters for the situation of nickel electroplating process, the Taguchi method was employed. To this end, seven factors were taken into account, factors that influence the process, and a Taguchi-type experimental plan was envisaged. The obtained results have been displayed in a table plus graph, in order to facilitate interpretation and best work approach.*

Keywords: *nickel electroplating, material balance and energy balance, optimization method.*

Introduction

In the domain of anticorrosive protection, the galvanic proceedings have an important role. Generally, there are processes with a high cost of materials and implies a great consumption of energy.

The acquaintance of the electrolytic processes helps the efficient manage of the technological process. At the present time the domain of thin layers (or thin films), of the nanotechnologies represents an important chance. It is possible to obtain new electronic devices or new materials very resistant for utensils and machines. The electrolytic depose have to be well controlled as from the quantitative and the qualitative point of view. The granulation of materials, the porous structure, and the shine are essentials [Ba'02].

At the end the environment protection is important. The electrolytic processes imply a high consumption of energy which implies a great quantity of gases of greenhouse effect (ex. carbon dioxide). There are the gases resulted directly from the electrolytic processes, too, and the toxic substances which remain [Gr'05].

The optimization of galvanic proceedings implies two ways, which means the qualitative and the quantitative aspects of the phenomena. From the qualitative point of view there is the problem of conception of a mathematical model which could describe as well it is possible the intimate of processes, for controlling the granulation, the rough and the shine. The quantitative aspect is treating with the balances of materials and the balances of energy, which means a better output of the industrial process.

The correlation of the quantities of the substances separated at the electrodes of a galvanic cell with the electric current output, intensity and time, the composition of the nickel bath, it is possible to realize with the balance of materials on the components.

In this work we show the theoretical bases for the elaboration of material balances in the case of electrochemical nickel cover and a model for the optimization of the quality of cover.

Materials and methods

To describe quantitative the chemical process it is necessary to create the balance for any component and for any phase in the reaction mass, the thermal balance and the balance for energy. All the equations could be written as differential equations. For the simplicity in finding the solutions the mathematical computing is used [Ca'72].

The stoichiometric equation of a chemical process could be written as follow:

$$\nu_{A_1} A_1 + \nu_{A_2} A_2 + \ldots + \nu_{A_n} A_n = \nu_{A_1} A_1^{'} + \nu_{A_2} A_2^{'} + \ldots + \nu_{A_n} A_n^{'} \tag{1}$$

or in the follow form:

$$\sum_{i=1}^{n} \nu_{A_i} A_i = \sum_{i=1}^{m} \nu_{A_i^{\prime}} A_i^{\prime} \tag{2}$$

where: ν_{A_i} - the stoechiometric coefficient of the reactants;

$\nu_{A_i^{'}}$ - the stoechiometric coefficient of the reaction products;

$A_i, A_i^{'}$ - the reactants and the reaction products.

In technical calculus it is useful as the reactant which transformation is followed, to be précised with the name *valuable reactant*. Such of kind of reactant is written A_K. Then it will be note as follow: n_{A_K} - the mol number of A_K reactant at one moment of time and through $n_{A_K}^0$ at initial time. The initial chemical composition of the reaction mass is expressed the mol numbers of reactant components and of the inert. Then:

$$n_{A_1}^0, \ldots, n_{A_n}^0; n_{A_1^{\prime}}^0, \ldots, n_{A_m^{\prime}}^0; n_{A_1^{\prime\prime}}^0, \ldots, n_{A_s^{\prime\prime}}^0 \tag{3}$$

and the composition of the reaction mass at one moment in time will be noted by:

$$n_{A_1}, \ldots, n_{A_n}; n_{A_1^{\prime}}, \ldots, n_{A_m^{\prime}}; n_{A_1^{\prime\prime}}, \ldots, n_{A_s^{\prime\prime}} \tag{4}$$

To expressed the composition of mass of reaction in one moment, it is used the notion *level of transformation for the valuable reactant* (η_{A_K}), or of one another reactant (η_{A_i}).

This measure is defined as the transform fractious of the reactant:

$$\eta_{A_K} = \frac{n_{A_K}^0 - n_{A_K}}{n_{A_K}^0} = \frac{n_{A_i}^0 - n_{A_i}}{\frac{\nu_{A_i}}{\nu_{A_k}} n_{A_k}^0} = \frac{n_{A_i^{\prime}} - n_{A_i^{\prime}}^0}{\frac{\nu_{A_i^{\prime}}}{\nu_{A_K}} n_{A_k}^0} \tag{5}$$

Passing at real values it has to know the initial composition and the final composition of the reaction mass and the level of transformation for the valuable reactant η_{A_K}. The balance of materials is a real balance in the situation when η_{A_K} - represent the level of transformation in one moment in time and it is a theoretical balance in the situation when η_{A_K} is the level of transformation at equilibrium. The numeric value for the level of transformation could be obtained through equilibrium calculus and also experimental. The theoretic balance could permit to establish the coefficients of consumption [Gu'86].

We shall consider a Watt bath with a nickel anode (the soluble anode variant), and as cathode the piece which will be nickel covered. In the bath there are

solutions: H_2SO_4, $NiCl_2$, $NiSO_4$, and HCl.
The reactions at the cathode are:

$$NiSO_4 + NiCl_2 + 6H^+ + 6e^- \rightarrow 2Ni + H_2SO_4 + 2HCl + H_2 \tag{6}$$

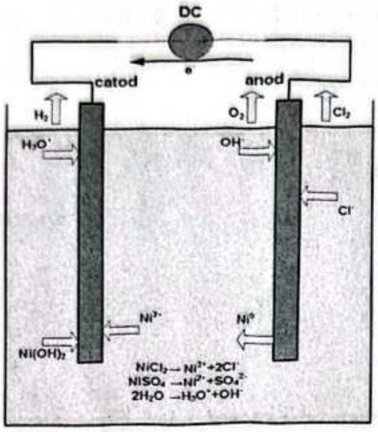

Figure 1 Nickel electroplating: representation of the processes.

Table1 Anode energy balance

Anode energy balance	$H_2SO_4+2HCl+H_2O+2Ni \rightarrow NiSO_4+NiCl_2+1/2O_2+6H^++6e^-$
$\eta_{Ni} = \frac{m^0_{Ni} - m_{Ni}}{\frac{1}{2} m^0_{Ni}} = \frac{m^0_{H_2SO_4} - m_{H_2SO_4}}{\frac{M_{H_2SO_4}}{2M_{Ni}} \cdot m^0_{Ni}} = \frac{m^0_{HCl} - m_{Cl}}{\frac{M_{HCl}}{M_{Ni}} \cdot m^0_{Ni}} = \frac{m^0_{H_2O} - m_{H_2O}}{\frac{M_{H_2O}}{M_{Ni}} \cdot m^0_{Ni}} =$ $= \frac{m_{NiSO_4}}{\frac{M_{NiSO_4}}{2M_{Ni}} \cdot m^0_{Ni}} = \frac{m_{NiCl_2}}{\frac{M_{NiCl_2}}{M_{Ni}} \cdot m^0_{Ni}} = \frac{m_{O_2}}{\frac{M_{O_2}}{4M_{Ni}} \cdot m^0_{Ni}} = \frac{m_{H^+}}{\frac{3M_{H^+}}{M_{Ni}} \cdot m^0_{Ni}}$	
$m_{Ni} = m^0_{Ni} - \frac{1}{2} m^0_{Ni} \eta_{Ni} =$ $= m^0_{Ni} - \frac{M_{Ni} \cdot I \cdot t}{F} \cdot \eta_{O_2}$	$\frac{W_{Ni}}{m_{Ni}} = \frac{U_{celula} \cdot I \cdot t \cdot F}{m^0_{Ni} F - M_{Ni} \cdot I \cdot t \cdot \eta_{O_2}}$
$m_{H_2SO_4} = m^0_{H_2SO_4} - \frac{1}{2} \frac{M_{H_2SO_4}}{M_{Ni}} m^0_{Ni} \cdot \eta_{Ni} =$ $= m^0_{H_2SO_4} - \frac{M_{H_2SO_4} \cdot I \cdot t}{F} \cdot \eta_{O_2}$	$\frac{W_{H_2SO_4}}{m_{H_2SO_4}} =$ $= \frac{U_{celula} \cdot I \cdot t \cdot F}{m^0_{H_2SO_4} \cdot F - M_{H_2SO_4} \cdot I \cdot t \cdot \eta_{O_2}}$
$m_{HCl} = m^0_{HCl} - \frac{M_{HCl}}{M_{Ni}} m^0_{Ni} \cdot \eta_{Ni} =$ $= m^0_{HCl} - \frac{2M_{HCl} \cdot I \cdot t}{F} \cdot \eta_{O_2}$	$\frac{W_{HCl}}{m_{HCl}} = \frac{U_{celula} \cdot I \cdot t \cdot F}{m^0_{HCl} F - 2M_{HCl} \cdot I \cdot t \cdot \eta_{O_2}}$

$m_{H_2O} = m^0_{H_2O} - \frac{1}{2}\frac{M_{H_2O}}{M_{Ni}} \cdot m^0_{Ni} \cdot \eta_{Ni} =$ $= m^0_{H_2O} - \frac{M_{H_2O} \cdot I \cdot t}{F} \cdot \eta_{O_2}$	$\frac{W_{H_2O}}{m_{H_2O}} = \frac{U_{celula} \cdot I \cdot t \cdot F}{m^0_{H_2O}F - M_{H_2O} \cdot I \cdot t \cdot \eta_{O_2}}$
$m_{NiSO_4} = \frac{1}{2}\frac{M_{NiSO_4}}{M_{Ni}} m^0_{Ni} \cdot \eta_{Ni} = \frac{M_{NiSO_4} \cdot I \cdot t}{F} \cdot \eta_{O_2}$	$\frac{W_{NiSO_4}}{m_{NiSO_4}} = \frac{U_{celula} \cdot F}{M_{NiSO_4} \cdot \eta_{O_2}}$
$m_{NiCl_2} = \frac{1}{2}\frac{M_{NiCl_2}}{M_{Ni}} m^0_{Ni} \cdot \eta_{Ni} = \frac{M_{NiCl_2} \cdot I \cdot t}{F} \cdot \eta_{O_2}$	$\frac{W_{NiCl_2}}{m_{Ni}} = \frac{U_{celula} \cdot F}{M_{NiCl_2} \cdot \eta_{O_2}}$

Table2 Cathode energy balance

Cathode energy balance	$NiSO_4 + NiCl_2 + 6H^+ + 6e^- \rightarrow 2Ni + H_2SO_4 + 2HCl + H_2$
$\eta_{NiSO_4} = \frac{m^0_{NiSO_4} - m_{NiSO_4}}{m^0_{NiSO_4}} = \frac{m^0_{NiCl_2} - m_{NiCl_2}}{\frac{M_{NiCl_2}}{M_{NiSO_4}} \cdot m^0_{NiSO_4}} = \frac{m^0_{H^+} - m_{H^+}}{\frac{6M_{H^+}}{M_{NiSO_4}} \cdot m^0_{NiSO_4}} = \frac{m_{H_2SO_4}}{\frac{M_{H_2SO_4}}{M_{NiSO_4}} \cdot m^0_{NiSO_4}} =$ $= \frac{m_{Ni}}{\frac{2M_{Ni}}{M_{NiSO_4}} \cdot m^0_{NiSO_4}} = \frac{m_{HCl}}{\frac{2M_{HCl}}{M_{NiSO_4}} \cdot m^0_{NiSO_4}} = \frac{m_{H_2}}{\frac{M_{H_2}}{M_{NiSO_4}} \cdot m^0_{NiSO_4}}$	
$m_{NiSO_4} = m^0_{NiSO_4} - m^0_{NiSO_4} \cdot \eta_{NiSO_4} =$ $= m^0_{NiSO_4} - \frac{M_{NiSO_4} \cdot I \cdot t}{2F} \cdot \eta_{Ni}$	$\frac{W_{NiSO_4}}{m_{NiSO_4}} =$ $= \frac{2U_{celula} \cdot I \cdot t \cdot F}{2m^0_{NiSO_4} \cdot F - M_{NISO_4} \cdot I \cdot t \cdot \eta_{Ni}}$
$m_{NiCl_2} = m^0_{NICl_2} - m^0_{NiSO_4} \cdot \frac{M_{NiCl_2}}{M_{NiSO_4}} \cdot \eta_{NiSO_4} =$ $= m^0_{NiCl_2} - \frac{M_{NiCl_2} \cdot I \cdot t}{2F} \cdot \eta_{Ni}$	$\frac{W_{NiCl_2}}{m_{NiCl_2}} = \frac{2U_{celula} \cdot I \cdot t \cdot F}{2m^0_{NiCl} \cdot F - M_{NiCl_2} \cdot I \cdot t \cdot \eta_{Ni}}$
$m_{H^+} = m^o_{H^+} - m^o_{NiSO_4} \cdot \frac{6M_{H^+}}{M_{NiSO_4}} \cdot \eta_{NiSO_4} =$ $= m^0_{H^+} - \frac{3M_{H^+} \cdot I \cdot t}{F} \cdot \eta_{Ni}$	$\frac{W_{H^+}}{m_{H^+}} = \frac{U_{celula} \cdot I \cdot t \cdot F}{m^0_{H^+} \cdot F - 3M_{H^+} \cdot I \cdot t \cdot \eta_{Ni}}$
$\frac{W_{H^+}}{m_{H^+}} = \frac{U_{celula} \cdot I \cdot t \cdot F}{m^0_{H^+} \cdot F - 3M_{H^+} \cdot I \cdot t \cdot \eta_{Ni}}$	$\frac{W_{H_2SO_4}}{m_{H_2SO_4}} = \frac{2U_{celula} \cdot F}{M_{H_2SO_4} \cdot \eta_{Ni}}$
$\frac{W_{H_2SO_4}}{m_{H_2SO_4}} = \frac{2U_{celula} \cdot F}{M_{H_2SO_4} \cdot \eta_{Ni}}$	$\frac{W_{H_2SO_4}}{m_{H_2SO_4}} = \frac{2U_{celula} \cdot F}{M_{H_2SO_4} \cdot \eta_{Ni}}$

$\frac{W_{H_2SO_4}}{m_{H_2SO_4}}=\frac{2U_{celula}\cdot F}{M_{H_2SO_4}\cdot\eta_{Ni}}$	$\frac{W_{H_2SO_4}}{m_{H_2SO_4}}=\frac{2U_{celula}\cdot F}{M_{H_2SO_4}\cdot\eta_{Ni}}$
$\frac{W_{H_2SO_4}}{m_{H_2SO_4}}=\frac{2U_{celula}\cdot F}{M_{H_2SO_4}\cdot\eta_{Ni}}$	$\frac{W_{H_2SO_4}}{m_{H_2SO_4}}=\frac{2U_{celula}\cdot F}{M_{H_2SO_4}\cdot\eta_{Ni}}$

Corresponding to these relations there will be the relations for the electrical energies used in the electrolytic cell. These results consider the cell like a resistive element which uses the energy:

$$W = U_{cell}\cdot I\cdot t \qquad (7)$$

For our case it is important to write the energy on the mass unit, then *W/kg*.

The nickel mass deposed at the cathode will be:

$$m_{Ni}=\frac{M_{Ni}\cdot I\cdot t}{F}\cdot\eta_{Ni} \qquad (8)$$

and the energy on mass unit:

$$\frac{W_{Ni}}{m_{Ni}}=\frac{U_{cell}\cdot F}{M_{Ni}\cdot\eta_{Ni}} \qquad (9)$$

As it can see, excepting time, the variables are current intensity *I*, the voltage of the cell U_{cell} and the transformation fraction. This it has to be calculating couldn't be initially predicted. The other elements like the geometry of the cell, the geometry of the electrodes, the concentration of the solution are in the transformation fraction [Fi'83]. In a given time, the mass deposed could grow with grow of the intensity, but this is limited by secondary phenomena (chemical reaction undesired, grow of the temperature etc.). Also, the specific energy used could reduce with the voltage (U_{cell}). But this voltage cannot be so small. It has to be over the specific potential of the electrodes, so the cell will function like an electrolytic cell, no like a source.

We shall use a method Taguchi to study the influence of all facts: the electric intensity, the voltage of the cell, electrolytic temperature, concentration of $NiSO_4$, concentration of $NiCl_2$ and the purity of the Ni anode.

Table 3 The definition of factors and their levels

	Controlled factors	**Level 1**	**Level 2**
A	*Intensity*	I_1	I_2
B	*Voltage*	U_1	U_2
C	*Temperature*	T_1	T_2
D	*Concentration $NiSO_4$*	C_1	C_2
E	*concentration $NiCl_2$*	C_1^1	C_2^1
F	*anode type*	F_1	F_2

We shall make a matrix L_8 with interaction AC, then between the intensity and the electrolytic temperature [Al'99].

Table 4 The matrix of experiments L_8

No. of tests trials	Controlled Factors						Interaction
	A	B	C	D	E	F	AC
1	1	1	1	1	1	1	1
2	1	1	1	2	2	2	2
3	1	2	2	1	2	2	1
4	1	2	2	2	1	1	2
5	2	1	2	1	1	2	2
6	2	1	2	2	2	1	1
7	2	2	1	1	2	1	2
8	2	2	1	2	1	2	1

The results of measurements are in the follow table (the thickness of nickel film, the quality of deposed film, shines).

Table 5 The results of measurements

No. of prove	Measured values					
	No1	No2	No3	No4	No5	Average
1	V_{11}	V_{12}	V_{13}	V_{14}	V_{15}	$\bar{v}_1$
2	V_{21}	V_{22}	V_{23}	V_{24}	V_{25}	$\bar{v}_2$
3	V_{31}	V_{32}	V_{33}	V_{34}	V_{35}	$\bar{v}_3$
4	V_{41}	V_{42}	V_{43}	V_{44}	V_{45}	$\bar{v}_4$
5	V_{51}	V_{52}	V_{53}	V_{54}	V_{55}	$\bar{v}_5$
6	V_{61}	V_{62}	V_{63}	V_{64}	V_{65}	$\bar{v}_6$
7	V_{71}	V_{72}	V_{73}	V_{74}	V_{75}	$\bar{v}_7$
8	V_{81}	V_{82}	V_{83}	V_{84}	V_{85}	$\bar{v}_8$
general average						$\bar{v}$

The average response for every level is the average results for all proves for every factor at that level.

$$\begin{aligned}
\overline{A}_1 &= \frac{\bar{v}_1+\bar{v}_2+\bar{v}_3+\bar{v}_4}{4}, \quad \overline{A}_2 = \frac{\bar{v}_5+\bar{v}_6+\bar{v}_7+\bar{v}_8}{4} \\
\overline{B}_1 &= \frac{\bar{v}_1+\bar{v}_2+\bar{v}_5+\bar{v}_6}{4}, \quad \overline{B}_2 = \frac{\bar{v}_3+\bar{v}_4+\bar{v}_7+\bar{v}_8}{4} \\
\overline{C}_1 &= \frac{\bar{v}_1+\bar{v}_2+\bar{v}_7+\bar{v}_8}{4}, \quad \overline{C}_2 = \frac{\bar{v}_3+\bar{v}_4+\bar{v}_5+\bar{v}_6}{4} \\
\overline{D}_1 &= \frac{\bar{v}_1+\bar{v}_3+\bar{v}_5+\bar{v}_7}{4}, \quad \overline{D}_2 = \frac{\bar{v}_2+\bar{v}_4+\bar{v}_6+\bar{v}_8}{4} \\
\overline{E}_1 &= \frac{\bar{v}_1+\bar{v}_4+\bar{v}_5+\bar{v}_8}{4}, \quad \overline{E}_2 = \frac{\bar{v}_2+\bar{v}_3+\bar{v}_6+\bar{v}_7}{4} \\
\overline{F}_1 &= \frac{\bar{v}_1+\bar{v}_4+\bar{v}_6+\bar{v}_7}{4}, \quad \overline{F}_2 = \frac{\bar{v}_2+\bar{v}_3+\bar{v}_5+\bar{v}_8}{4}
\end{aligned} \tag{10}$$

The general average $\overline{v}$ of all proves is represented by the central point of the average level for any factor:

$$\overline{v}=\frac{\overline{A}_1+\overline{A}_2}{2}=\frac{\overline{B}_1+\overline{B}_2}{2}=\frac{\overline{C}_1+\overline{C}_2}{2}=\frac{\overline{D}_1+\overline{D}_2}{2}=\frac{\overline{E}_1+\overline{E}_2}{2}=\frac{\overline{F}_1+\overline{F}_2}{2} \quad (11)$$

The medium effect for every level is appreciated through general average.

$$E_{A_{11}}=\overline{A}_1-\overline{v},\ \ E_{A_2}=\overline{A}_2-\overline{v} \quad (12)$$

Evident: $E_{A_1}=-E_{A_2}$

Similar we shall have the following relations:

$$\begin{aligned}
&E_{B_1}=\overline{B}_1-\overline{v}, E_{B_2}=\overline{B}_2-\overline{v}, E_{B_1}=-E_{B_2}\\
&E_{C_1}=\overline{C}_1-\overline{v}, E_{C_1}=\overline{C}_2-\overline{v}, E_{C_1}=-E_{C_2}\\
&E_{D_1}=\overline{D}_1-\overline{v}, E_{D_1}=\overline{D}_2-\overline{v}, E_{D_1}=-E_{D_2}\\
&E_{E_1}=\overline{E}_1-\overline{v}, E_{E_2}=\overline{E}_2-\overline{v}, E_{E_1}=-E_{E_2}\\
&E_{F_1}=\overline{F}_1-\overline{v}, E_{F_2}=\overline{F}_2-\overline{v}, E_{F_1}=-E_{F_2}
\end{aligned} \quad (13)$$

The interaction by the factors A and C could be calculated from the effects of the factors A and C.

$$\begin{aligned}
&E_{A_1C_1}=E_{A_1}+E_{C_1}+I_{A_1C_1}\\
&E_{A_1C_1}=\overline{A_1C_1}-\overline{v}=\left(\overline{A}_1-\overline{v}\right)+\left(\overline{C}_1-\overline{v}\right)+I_{A_1C_1}
\end{aligned} \quad (14)$$

And it results,

$$\begin{aligned}
&I_{A_1C_1}=\overline{A_1C_1}-\overline{v}-\left(\overline{A}_1-\overline{v}\right)-\left(\overline{C}_1-\overline{v}\right)\\
&I_{A_1C_1}=\overline{A_1C_1}-\overline{v}-E_{A_1}-E_{C_1}
\end{aligned} \quad (15)$$

The medium response reported at every combination AC could be calculated following the same principle:

$$\begin{aligned}
&\overline{A_1C_1}=\frac{\overline{v}_1+\overline{v}_2}{2},\ \overline{A_1C_2}=\frac{\overline{v}_3+\overline{v}_4}{2}\\
&\overline{A_2C_1}=\frac{\overline{v}_7+\overline{v}_8}{2},\ \overline{A_2C_2}=\frac{\overline{v}_5+\overline{v}_6}{2}
\end{aligned} \quad (16)$$

Then it can be calculated all interactions $I_{A_1C_1}, I_{A_1C_2}, I_{A_2C_1}, I_{A_2C_2}$.

With the values $\overline{A}_1, \overline{A}_2, \ldots,\ E_{A_1}, E_{A_2}, \ldots$ the table with responses is made [Ho'00].

Table 6 Responses of the factors

No of factors	**The effect over two measured values**	
	level 1	level 2
A	E_{A_1}	E_{A_2}
B	E_{B_1}	E_{B_2}
C	E_{C_1}	E_{C_2}
D	E_{D_1}	E_{D_2}
E	E_{E_1}	E_{E_2}
F	E_{F_1}	E_{F_2}
AC	$E_{A_1C_1}$	$E_{A_2C_2}$

Then we draw the graph of the responses:

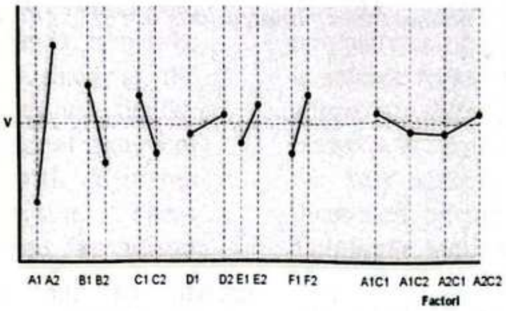

Figure 2 The graph of responses.

Conclusions

The value $\overline{v}$ represents a target thickness for the nickel film deposed which has the desired shine, granulation and desired adherence. Of course, if only quantity of nickel deposed interested us, the problem should be treated different *ab initio*. Here our intension is to touch as well it is possible the target value. Analyzing the results in the graph it is possible to decide the value of the parameters, and then searches prove for validation will be made. Of course there is possible to take intermediate parameters between A_1, A_2, B_1, B_2, etc.

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