HYDROGEN PERMEATION AND NICKEL FILMS STRUCTURE CORRELATION

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Introduction

The galvanic nickel films of 3 – 8 mcm thickness have found the application in radioelectronic industry and in device-building. The electrochemical nickel coatings receiving method features are the high structural sensitivity and the properties dependence on the electrolyte chemical composition and electrolysis regimes [1, 2, 3]. So, the present paper aim has become the investigation of the electrodeposition regimes influence on the structure and hydrogen permeation of the nickel films under 4 mcm thickness of, received from sulphamate electrolyte [4]. The samples of 500 – 1000 Å thickness on the copper base material were used for the Ni deposits structure detection. The grain size was calculated and the histograms, characterized the grains distribution with their size, were built according the nickel deposits microstructures photographs (increasing in 82000). The microtensions value and the mosaic blocks size were determined by the (111)α и (200)α lines. The quantitative porosity magnitude is represented as porosity surfaces (n) – - the pores number, related to the 1 cm^2 of the galvanic coating [5]. The hydrogen permeation (V_{H2}, cm^3/ 100 g) was determined by the vacuum extraction method. The electrolysis regimes interval: the temperature t = 30 – 50 ^{0}C; the acidity value pH = 3,5 – 4,5.

Results and discussion

The investigation results are represented on the Fig. 1, 2, 3. The grains distribution with size from their number histogram for the nickel film, received at the t = 40 ^{0}C, is shown on the Fig. 1. We can conclude, that the dominating grains number (68 %) are of the size inside 0 - 400 Å, and the received structure can be attributed to the smallgrained one and the grains shape – to the equalaxial. The electrolyte temperature influence on the nickel films structure is evident from Fig. 1 and Fig. 2, 3 comparison. The electrolyte temperature decreasing to 30 ^{0}C leads to the histogram view changing. The maximums shift to the right at the low temperature, and the significant grains dispersion with their size is observed.

The small grains number of 100 – 400 Å diameters is ~ 66 %, the middle of 400 - 800 Å - ~ 40 % and the large with diameter higher than 800 Å - ~ ~ 13 % from the summary grains number. This testifies about nonequalgrained structure formation. When the electrolyte temperature increases up to 50 ^{0}C, the grain size is becoming even (Fig. 3). The portion of small grains with size up to 400 Å decreases (~ 60 %), and the grains number with size 800 – 1000 Å increases (~ 15 %). Beginning from the electrolyte temperature 40 ^{0}C the large crystallites with size more than 1000 Å disappear (Fig. 1, 3).

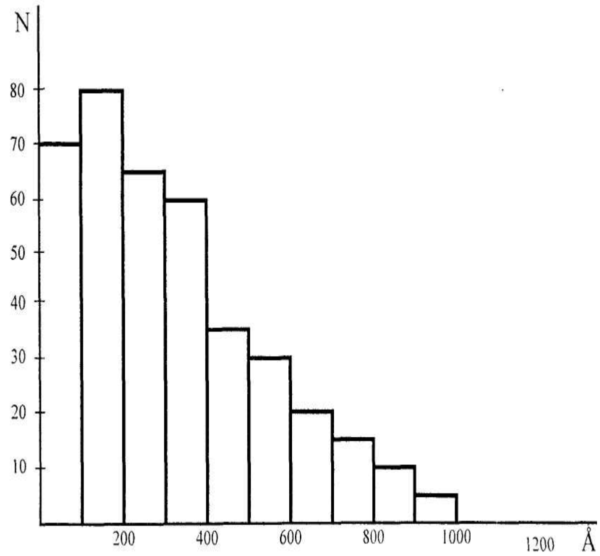

Fig. 1. The histogram of the grains distribution with size for nickel film
Electrolysis regimes:
i_k = 2A/dm^2; pH = 4,0; t = 40 °C

The electrolyte temperature influence on the microdistortions and the mosaic blocks dispersion of nickel films is interesting. When t decreases from 40 to 30 ^{0}C the microdistortions magnitude and the mosaic blocks size D_{HKL} is becoming smaller. This is connected, evidently, with more intense hydrogen evolution at low temperatures, and numerous pores, unentireties, macrodefects form, because of the simultaneous nickel deposition. Particularly, the pores and defects origin leads to the partial microdistortions relaxation in the coating. The electrolyte temperature increasing from 30 to 40 ^{0}C causes the formation of film with less porosity from 17,2 to 10,4. The matter is, that at the high nickel sulphamate concentrations ~ ~ 500 g/l, the electrolyte temperature decreasing enhances its viscosity, that makes the molecular hydrogen escape from the deposit surface more difficult, and the coating porosity increases.

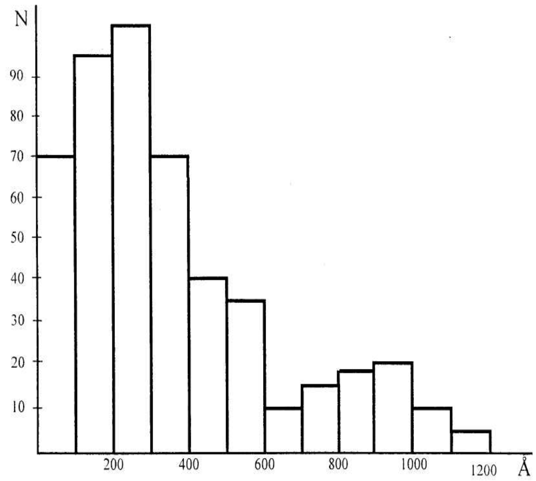

Fig. 2. The histogram of the grains distribution with size for nickel film.
Electrolysis regimes:
t = 30 °C; i_K = 2 A/dm^2; pH = 4,0

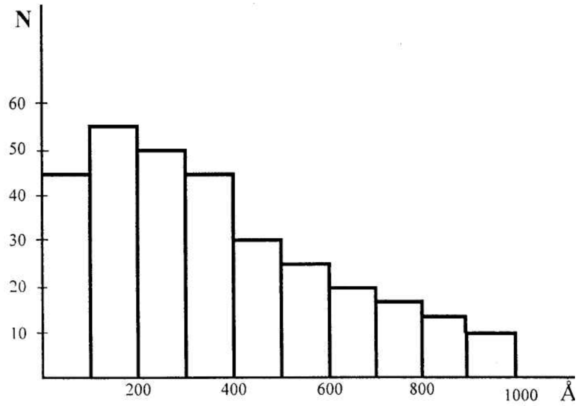

Fig. 3. The histogram of the grains distribution with size for nickel film.
Electrolysis regimes:
t = 50 °C; i_K = 2 A/dm^2; pH = 4,0

It was received, that the largest hydrogen consumption (V_{H2}) at the t = 30^{0}C is 117 cm^3/ 100 g. The hydrogen consumption variation with temperature in the nickel coating is represented in the table

Table.

The electrolyte temperature influence on the nickel films porosity and hydrogen permeation (coating thickness d = 4 мкм).
Electrolysis regimes: pH = 4,0; i_k = 2 A/dm^2

t, ^{0}C	Porosity n, pores/cm^2	Hydrogen permeation V_{H2}, cm^3/100 g
30	17,2	117
40	10,4	104
50	11,1	88,4

Conclusions

The coatings, received at the electrodeposition regime: i_k = 2 A/dm^2, pH = 4,0, t = 40 ^{0}C, are characterized by less defects and pores number, larger mosaic blocks size, that leads to the microdistortions growth and more tense coating formation. The tensions and D_{HKL} of the coatings, deposited at this regime, are maximal.

The temperature growth from 40 to 50 ^{0}C causes the pores number reduction and the hydrogen consumption decreasing in the nickel deposit. At the t = 50 ^{0}C V_{H2} = 88,4 cm^3/ 100 g. The crystal lattice microdistortions are decreasing when t enhances up to 50 ^{0}C. Simultaneously, the lowing of the cathodic evaluating hydrogen quality leads to the less active crystallization centers blocking, that results in the bigger number of such centers, and the deposit structure is becoming more equalgrained. The coating with less tensions is forming.

The electrolyte temperature growth decreases its viscosity. Thus, the hydrogen desorption from the cathodic surface becomes easy, and the coating porosity decreases.

References

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