

Crystallographic Texture Formation in Metals being Electrodeposited at the External Force Influence

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Abstract The aim of the work was the experimental verification of the validity of the phenomenon of phase formation through a stage of liquid state in metals being electrodeposited. For that the features of formation of crystallographic texture in metals being electrodeposited under the influence of external (centrifugal) force in the directions opposite and along the texture axis were investigated. As a result the effect of suppression of the process of texture formation in metals being electrodeposited up to the complete disordering of crystal lattices of grains under the influence of external force in the direction opposite to the texture axis was discovered. The effect of intensification of the texture formation process in metals being electrodeposited at the influence of external force directed along the texture axis was also found. The comparative analysis of the degree of texture perfection of electrodeposited metals obtained at different directions of the force action during their phase formation was accomplished. It was found that regardless to the direction of the force influence the most significant change of the degree of texture perfection occurs in metals being electrodeposited at relatively low overloads. It was discovered that the action of external force in the direction opposite to the texture axis results in the more significant influence on the texture formation process in metals being electrodeposited in comparison with the force action along the texture axis. The obtained results prove the validity of the phenomenon of phase formation through a stage of liquid state in metals being electrodeposited

Keywords Crystallographic Texture, Metal being Electrodeposited, Centrifugal Force, External Force Influence

1. Introduction

The phenomenon of phase formation through a stage of liquid state in metals being electrodeposited was experimentally found in 1988 [1]. The essence of the discovered phenomenon is that during the electrochemical deposition of a metal onto a solid cathode in an aqueous medium a undercooled metallic liquid is being formed and superfast solidified at the deposition temperature in the form of a crystalline or/and an amorphous phase.

Large volume of experimental materials proving the validity of the phenomenon of phase formation through a stage of liquid state in metals being electrodeposited has been obtained so far [2–8]. Nevertheless, the obtaining of additional data proving or contradicting the validity of this phenomenon was of interest. Therefore, the aim of this work was the further experimental verification of the validity of the discovered phenomenon.

General idea for the arrangement of new experiments was based on the fact that the degree of perfection of crystallographic texture appearing in metals being solidified

from liquid state at the action of the external force significantly depends on the direction of the force action. A change of the degree of perfection of texture in metals being electrodeposited with the change of the direction of minor force action (in comparison with the value of plastic deformation creating texture in solids) will be a proof of the validity of the discussed phenomenon.

2. Experimental Proof of Validity of the Phenomenon

2.1. Idea One and Its Realization

It is known that texture formation of metal during its solidification occurs by oriented crystallization in the direction perpendicular to the crystallization front [9]. On the other hand, it is known that crystallographic texture of electrodeposited metal is the axial texture of growth with the axis perpendicular to the surface of a deposit [10]. Assuming that crystallographic texture of metal being electrodeposited is formed as a result of oriented crystallization of continuously renewed metallic liquid, the action of centrifugal force directed perpendicular to the crystallization front and opposite to the texture axis will obstruct the formation of texture.

As a result of such action of centrifugal force the metal

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with more dispersed texture will crystallize and the further increase of the value of centrifugal force can even cause random grain orientation of metal being electrodeposited. In this regard the suppression of the texture formation process in metals being electrodeposited at the action of centrifugal force in the direction opposite to the texture axis will be a proof of the validity of the discussed phenomenon.

2.1.1. Materials and Methods

The investigations of features of oriented structure formation in metals being electrodeposited in the field of centrifugal force were performed on copper and nickel samples using the unit described in the work [11]. The unit provided the influence of centrifugal force proportional to normal acceleration kg , where k is overload coefficient, on the process of electrocrystallization of metals. The values of centrifugal force were varied stepwise by the change of the centrifuge rotor speed from 0 to 3000 rotations per minute with the step of 500 rotations per minute, which provided normal acceleration $1g$, $35g$, $140g$, $314g$, $558g$, $872g$ and $1256g$.

The cathode was mounted at the bottom of the cell perpendicular to its axis, therefore the cathode was perpendicular to the rotor radius during cell rotation. As during rotation centrifugal force is directed along the rotor radius from the circle center and the axis of deposit texture is oriented along the normal to its surface in the direction of growth from cathode toward external layers, that at such cathode arrangement the force influence on metal being electrodeposited was provided in the direction opposite to the texture axis.

The composition of copper- and nickel-plating electrolytes as well as parameters and conditions of metals deposition in the field of centrifugal force are presented in the Table 1. As a result of metals current yield being 99.5-100 % and impossibility of electrolyte motion relative to the cathode in the cell during electrodeposition process [11] the influence of hydrogen evolution and electrolyte motion on texture of samples being deposited was completely eliminated.

Table 1. Electrolytes composition and parameters of electrodeposition of metals

Metal	Electrolytes composition ($g\ L^{-1}$)	Electrolyte temperature ($^{\circ}C$)	Current density ($A\ dm^{-2}$)	Current yield (%)
Cu	$CuSO_4 \cdot 5H_2O - 250$ $H_2SO_4 - 50$	25	1	100
Ni	$NiSO_4 \cdot 7H_2O - 330$ $NiCl_2 \cdot 6H_2O - 50$ $H_3BO_3 - 35$ $C_{11}H_{23}COONa - 0.1$	25	1	99.5

X-ray texture analysis of samples of electrodeposited metals was performed according to the method [12] at the automated and modernized X-ray diffractometer DRON-2. The texture of electrodeposits was determined by the method of direct pole figures combination [13]. For the quantitative estimation of the degree of texture perfection the average dispersion angle α of the axial texture component was used.

2.1.2. Results and Discussion

As a result of completed investigations it was found that the texture of nickel electrodeposited at $1g$ can be described by the following components: the main axial component [210], the subsidiary axial component [221] and random component. At that the axes [210] and [221] of the axial texture components of nickel are oriented perpendicular to the surface of electrodeposits in the direction of growth from the cathode to the external layers.

In fact, the presence of the textural maximum of the axial orientation with the axis [210] at 39.2° angle and the textural maximums of the axial orientation with the axis [221] at 15.8 and 54.7° angles on the (111) pole figure of electrodeposited nickel (Fig. 1a) allows making such conclusion. And the presence of the textural maximum of the axial orientation with the axis [210] at 26.6° angle and the textural maximum of the axial orientation with the axis [221] at 48.2 angle on the (100) pole figure (Fig. 1b) confirms this fact.

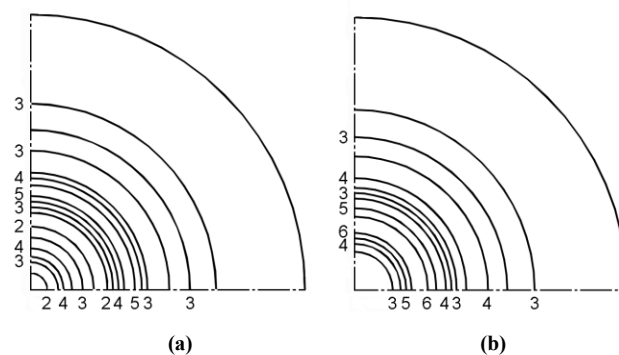


Figure 1. Pole figures (111) (a) and (100) (b) of nickel, electrodeposited at $1g$ (initial condition). 2-6 – levels of equal intensity of diffraction X-rays

The obtained result coordinates with the data of the work [14], where the axial component [210] was determined as the main texture component of nickel electrodeposited at the similar conditions.

From the comparison of the texture curves of nickel electrodeposited under the influence of centrifugal force directed opposite to the texture axis it is found that with the increase of the force action disorientation of texture occurs (Fig. 2) and at the overload of $1256g$ nickel electrodeposits with random grain orientation are being formed. This fact indicates that at the action of centrifugal force in the direction opposite to the texture axis the suppression of texture formation process occurs in nickel being electrodeposited.

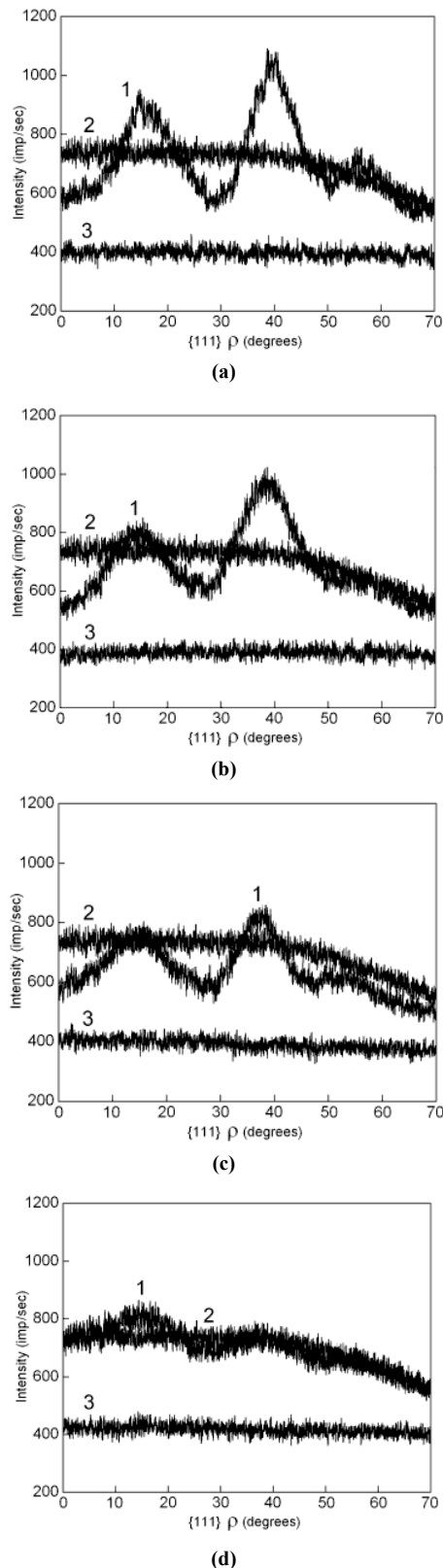


Figure 2. Texture curves of nickel electrodeposits, demonstrating the suppression of the process of texture formation of nickel being electrodeposited under the influence centrifugal force in the direction opposite to the texture axis. 1 and 2 – texture curves of interference {111} of the sample and the reference respectively, 3 – background curve; (a) – force is proportional to acceleration of 1g (initial condition), (b) – 35g, (c) – 314g and (d) – 872g

The comparison of the values of the average dispersion angle of the axial orientations of grains of nickel electrodeposited at different overloads (Fig. 3) confirms the made conclusion. Thus, at Fig. 3 it is seen that the increase of the overload coefficient at the action of centrifugal force in the direction opposite to the texture axis causes growth of the degree of dispersion of the axial orientations with the axes [210] and [221]. And the character of influence of overload on the axial components of nickel texture is the same: significant initial growth of the α angle (approximately 2 times at the increase of the k coefficient from 1 to 314) and then its gradual several percent growth (at the further increase of the k values to 872).

Particularly intensive dispersion of texture of electrodeposited nickel is found at minor overloads (up to $k = 140$). At that even minor growth of the k values (from 1 to 35) results in significant increase of the α angle values (approximately 1.5 degrees) (Fig. 3).

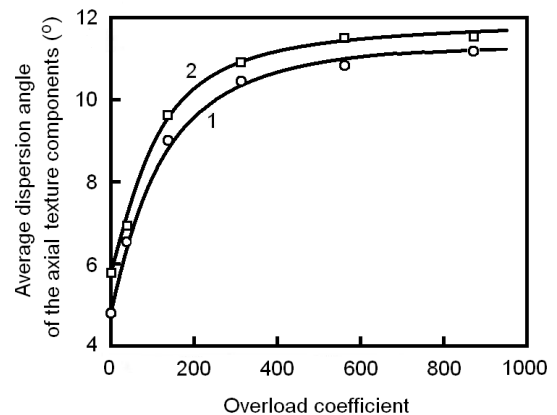


Figure 3. Change of the average dispersion angle of axial components [210] (curve 1) and [221] (curve 2) of texture of nickel electrodeposits depending on the overload coefficient at the influence of centrifugal force in the direction opposite to the texture axis

The discovered effect of the suppression of texture formation process in metals being electrodeposited under the influence of centrifugal force in the direction opposite to the texture axis is clearly confirmed by the experimental data of the investigations of texture formation in copper being electrodeposited in the force field. Thus, at normal conditions of electrodeposition (i.e. at 1g) the axial texture with the axis [110] perpendicular to its surface (Fig. 4a) is being formed and the average dispersion angle of this texture is 9.8°.

If centrifugal force influences the copper deposit being formed that even minor overload (35g) in the direction opposite to the texture axis causes almost complete suppression of preferred orientation of crystal lattices of grains in the direction [110]. As it is seen at Fig. 4b, as a result of such influence low-textured coating very similar to the reference sample with random grain orientation is formed. The further increase of overload in the direction opposite to the texture axis results in electrodeposition of copper samples with random grains orientations.

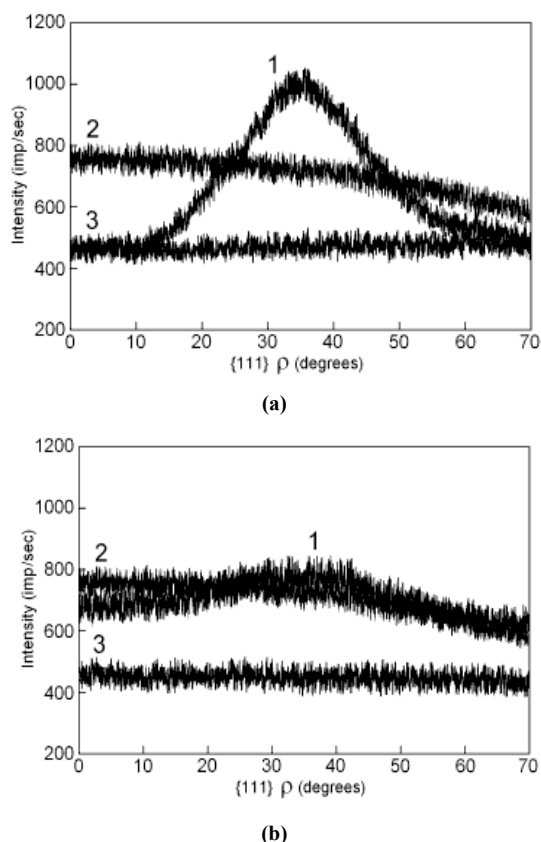


Figure 4. Texture curves of copper electrodeposits, illustrating the suppression of the process of texture formation of copper being electrodeposited under the influence centrifugal force in the direction opposite to the texture axis. 1 and 2 – texture curves of interference {111} of the sample and the reference respectively, 3 – background curve; (a) – force is proportional to acceleration of 1g (initial condition) and (b) – 35g

It is known that for disorientation of grains of textured materials significant deformations are required to provide plastic slide in crystallographic planes of different orientations [15]. Assuming that phase formation of metal being electrodeposited occurs without a stage of liquid state, the influence of centrifugal force must provide the formation of compression stress sufficient for occurrence of plastic deformation. The stress required for the beginning of plastic deformation of material is estimated as offset yield strength [16].

To determine the value of compression stress σ appearing in copper deposit during electrodeposition under the influence of centrifugal force causing disorientation of the lattices of grains, the following expression obtained by simple conversions can be used:

$$\sigma = \gamma \cdot h \cdot k \cdot g, \quad (1)$$

where γ – density of copper-plating electrolyte, kg/m^3 ; h – height of the electrolyte column above the deposit, m; k – overload coefficient, g – free fall acceleration, 9.81 m/sec^2 .

The values of γ , h and k are $1.165 \cdot 10^3 \text{ kg/m}^3$, 0.06 m and 35 respectively. Using the found values in the expression (1), $\sigma = 0.02 \text{ MPa}$ is obtained. This stress value is incommensurable with the value of the yield strength of copper, which is 62 MPa [17]. Even using the maximum

value of the overload coefficient achieved in the experiments ($k = 1256$), the stress caused by centrifugal force in copper electrodeposit will be only 0.86 MPa , which is clearly not enough for beginning of plastic deformation of solid copper electrodeposit.

The value of the compression stress in nickel being electrodeposited, which is caused by centrifugal force of maximum value (at $k = 1256$), is also not exceeding 1 MPa (for comparison, the yield strength of nickel is 83 MPa [17]). Therefore, disorientation of crystal lattices of grains of metals being electrodeposited under the influence of centrifugal force directed opposite to the texture axis cannot be explained by plastic deformation of metals being in solid state.

To make sure about the validity of the proposed concept of passing through a stage of liquid state by metals being electrodeposited the following model experiments were conducted. The samples of copper and nickel were electrodeposited at normal conditions (i.e. at the overload of $1g$). Then these samples were placed into the cell with the electrolyte and influenced by centrifugal force of the maximum value (at the $1256g$ overload) without deposition current. In these cases centrifugal force influenced solid copper or nickel deposit after the process of electrodeposition. The comparison of initial samples and those influenced by the force action shows no differences.

The discovered effect of suppression of the process of texture formation in metals being electrodeposited under the influence of centrifugal force in the direction opposite to the texture axis can be explained on the basis of the found phenomenon of phase formation through a stage of liquid state in metals being electrodeposited [1–8]. Such liquid state of metal being electrodeposited is caused by very quick (explosive) character of its precipitation as a result of a chain reaction of electrochemical formation of atoms [3]. At that during the one act of explosive growth 40–60 atoms are being formed on average.

With regard to very quick act of explosive growth (approximately 10^{-7} sec) there is not enough time for atoms to form a structure with long-distance order in their arrangement [3]. Multitude of such atom clusters appearing in an avalanche-like manner in different places on the surface of the cathode or growing deposit and subsequently combining into larger formations is a liquid phase of metal being electrodeposited.

As a liquid phase possesses very low shear strength [18], solidification of liquid clusters of atoms deformed by centrifugal force will cause disorientation of crystal lattices of grains of electrodeposit being formed. Formation of the preferred orientation of crystal lattices of grains in metal being electrodeposited will be suppressed more with the increase of centrifugal force acting in the direction opposite to the texture axis. Therefore, centrifugal force influencing the electrodeposit being formed opposite to the texture axis will obstruct the process of texture formation, which was experimentally observed.

Perhaps, for metals being deposited at minor

undercoolings the influence of centrifugal force, besides the suppression of texture, which is formed at normal conditions, can also facilitate the formation of new texture. Thus, the discovered effect of suppression of the texture formation process in metals being electrodeposited up to the complete disordering of crystal lattices of grains under the influence of centrifugal force in the direction opposite to the texture axis proves the validity of the discussed phenomenon.

2.2. Idea Two and Its Realization

It is known, that texture formation during crystallization of metal occurs in the direction of the external force influencing the metal [9]. At that the degree of perfection of texture of metal being solidified increases with the growth of values of the external force. Therefore, if metal being electrodeposited really passes through a stage of liquid state, then at the influence of centrifugal force on metallic liquid being solidified perpendicular to the crystallization front and along the texture axis the processes of formation and development of texture will intensify.

As a result of such action of centrifugal force phase formation of metal being electrodeposited will be accompanied by its intense texture formation. Formation of more perfect texture in metals being electrodeposited in the field of centrifugal force acting along the texture axis will be the fact proving the validity of the discussed phenomenon.

2.2.1. Materials and Methods

Features of texture formation in metals being electrodeposited in the field of centrifugal force acting along the texture axis were investigated on copper and nickel samples. Electrodeposition of metals at the force action conditions was performed using the equipment described in the work [11]. The anode was built into the bottom of the cell and the cathode was mounted at the upper side of the cell perpendicular to its axis at the distance of $4 \cdot 10^{-2}$ m from the anode. Therefore, during rotation of the cell the electrodes were placed perpendicular to the rotor radius. As centrifugal force during rotation is directed along the rotor radius from the center of the circle and the texture axis of the deposit is oriented along the normal to its surface in the direction of growth from the cathode to the external layers, than at such position of the cathode the force action on metal being electrodeposited along the texture axis was provided.

Obtaining metal deposits under the influence of centrifugal force opposite to the texture axis was performed at the force action values proportional to the normal acceleration of 1g, 35g, 140g, 314g, 558g, 872g and 1256g (see part 2.1). In order to the value of centrifugal force acting on the deposits being formed at the change of the electrodes position in the electrochemical cell to be constant, the centrifuge rotor speed was increased to 606, 1213, 1819, 2425, 3032 and 3638 rotations per minute respectively. The copper- and nickel-plating electrolytes compositions as well as method and parameters of X-ray texture analysis were as previous ones.

2.2.2. Results and Discussion

As a result of the completed investigations it was found that texture of copper deposits electrodeposited at the normal conditions (i.e. at 1g) is characterized by the axial component [110]. At that the [110] axis of the axial texture component of copper deposits is oriented perpendicular to their surface in the direction of growth from the cathode to the external layers.

In fact, the presence of the texture maximum of the axial orientation with the [110] axis at the 35.3° angle on the (111) pole figure of electrodeposited copper (Fig. 5a) allowed making this conclusion. And the presence of the texture maximums of the axial orientation with the [110] axis at the 0 and 60° angles on the (110) pole figure (Fig. 5b) confirmed it.

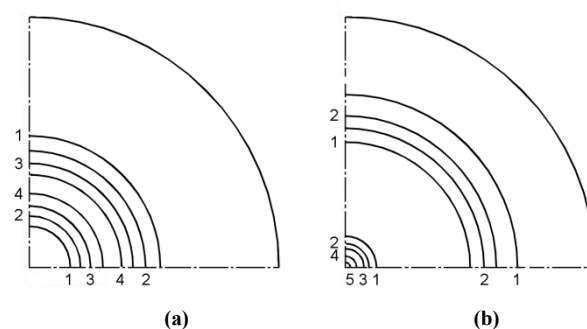
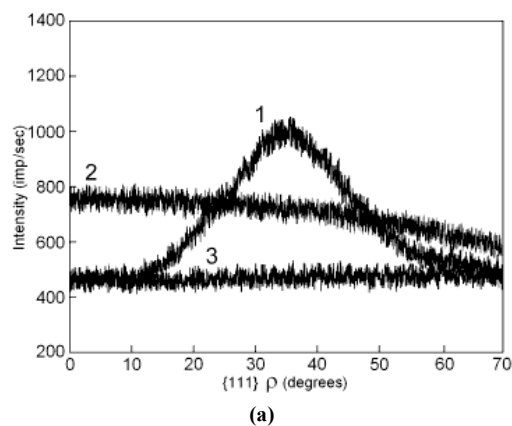


Figure 5. Pole figures (111) (a) and (110) (b) of copper electrodeposited at 1g (initial condition). 1-5 – levels of equal intensities of diffraction X-rays

The obtained result corresponds with the data of the work [19], according to which the axial component [110] is the main texture component of copper electrodeposited in the same conditions.

On the basis of the analysis of the experimental data (Fig. 6) the conclusion about intensification of the process of formation of crystallographic texture with the axis [110] in copper being electrodeposited at the force action along the texture axis was made. In fact, comparing the texture curves of the copper electrodeposits obtained under the influence of centrifugal force of different values directed along the texture axis, it can be seen the increase of the degree of texture perfection of copper samples with the growth of the force action.



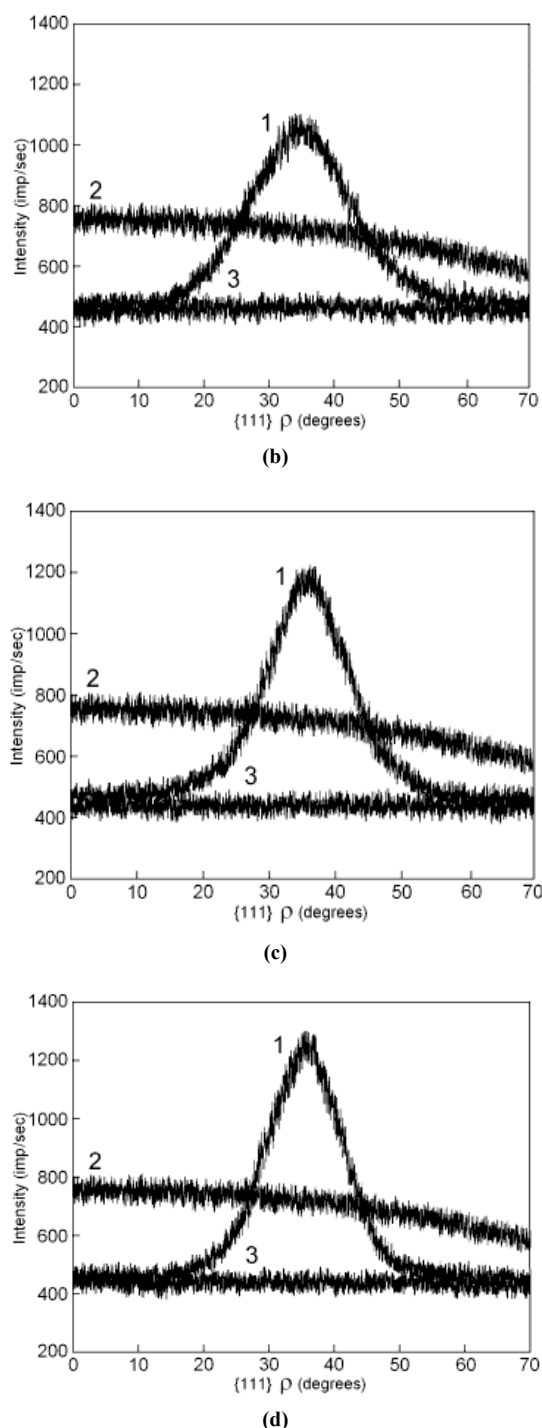


Figure 6. Texture curves of copper deposits showing the intensification of the process of texture formation of copper being electrodeposited under the influence of centrifugal force along the texture axis. 1 and 2 – texture curves of interference $\{111\}$ of the sample and the reference respectively, 3 – background curve; (a) – the force is proportional to the acceleration 1g (initial condition), (b) – 140g, (c) – 872g and (d) – 1256g

Quantitative estimation of the degree of texture perfection of the copper deposits electrodeposited at the action of centrifugal force of different values directed along the texture axis indicates formation of more perfect texture with

the $[110]$ axis with the growth of overload. Thus, at Fig. 7 it is seen that the increase of the overload coefficient from 1 to 1256 at the action of centrifugal force along the texture axis $[110]$ of copper being electrodeposited causes significant decrease of the average dispersion angle of the axial component $[110]$ of texture (by 3.35°).

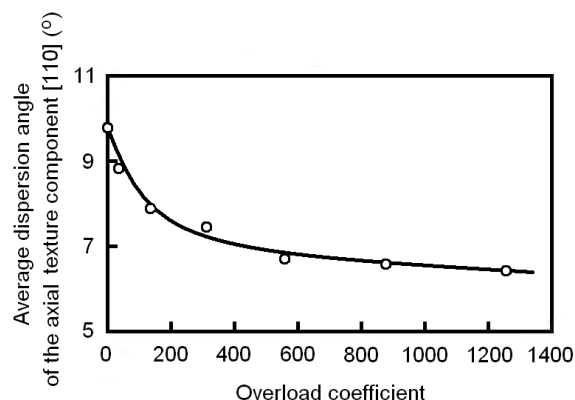


Figure 7. Dependence of the average dispersion angle of the axial component $[110]$ of texture of copper deposits on the overload coefficient at the influence of centrifugal force along the texture axis during electrodeposition

At that the most significant influence on the degree of texture perfection of copper deposits occurs at relatively low overloads (up to $k = 314$). Thus, in the interval of the k values from 1 to 314 the average dispersion angle of texture with the $[110]$ axis decreases by 25% (from 9.8 to 7.4°), while the further increase of the k coefficient to 1256 is accompanied by insignificant intensification of the texture formation process (Fig. 7).

The made conclusion about intensification of texture formation process in copper deposits obtained during electrodeposition in the field of centrifugal force directed along the texture axis is confirmed by the experimental data of investigations of the texture $[210]$ and $[221]$ of electrodeposited nickel. Thus, at Fig. 8 the texture curves of the nickel deposits obtained at the action of centrifugal force directed along the texture axes and proportional to acceleration from 1g to 872g during electrochemical phase formation are presented. The texture of these nickel deposits is characterized by two axial orientations with the axes $[210]$ and $[221]$ and random component. At that the axes $[210]$ and $[221]$ of axial texture components of nickel are oriented perpendicular to the surface of electrodeposits in the direction of growth from cathode to the external layers.

The comparative analysis of the texture curves of the nickel deposits (Fig. 8) indicates the perfecting of their texture in the crystallographic directions $[210]$ and $[221]$ with the increase of the force influence along these directions. Thus, comparison of Fig. 8a and 8d clearly indicates the decrease of dispersion of nickel texture at the centrifugal force action along its axes during phase formation at the electrochemical crystallization.

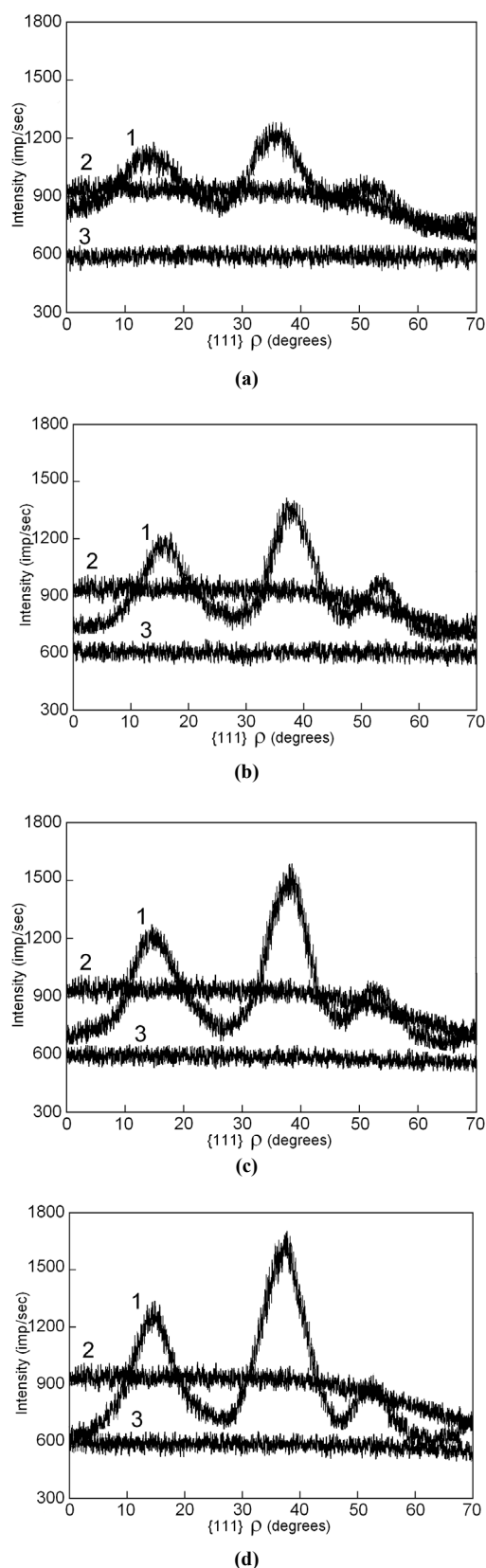


Figure 8. Texture curves of nickel deposits demonstrating the intensification of the process of texture formation of nickel being electrodeposited under the influence of centrifugal force along the texture axis. 1 and 2 – texture curves of interference $\{111\}$ of the sample and the reference respectively, 3 – background curve; (a) – the force is proportional to the acceleration 1g (initial condition), (b) – 35g, (c) – 314g and (d) – 872g

Quantitative estimation of the degree of texture perfection of the obtained deposits shows that the influence of centrifugal force on nickel being electrodeposited along the texture axes $[210]+[221]$ intensifies the process of texture formation, which is proved by the decrease of the average dispersion angle of the axial orientations with the axes $[210]$ and $[221]$ (Table 2).

Table 2. Change of the average dispersion angle of the axial components $[210]$ (α_1) and $[221]$ (α_2) of texture of nickel being electrodeposited with the increase of the overload coefficient (k) at the influence of centrifugal force along the texture axes

k	1	35	140	314	558	872	1256
α_1 (degrees)	4.8	4.3	4.1	4.0	3.8	3.7	3.6
α_2 (degrees)	5.8	5.1	4.85	4.7	4.6	4.4	4.3

Intensification of the process of texture formation during electrocrystallization of metals under the influence of external force along the texture axis can be explained by oriented solidification of undercooled liquid of metal being electrodeposited along the force action. Preferred orientation of crystal lattices of the multitude of clusters of metal atoms being solidified along the action of the external force and epitaxial solidification in the same direction of the multitude of new clusters of atoms of continuously renewed metallic liquid during electrochemical deposition cause intensification of the process of texture formation with the increase of the force action.

For the verification of the validity of the proposed conception of passing through a stage of liquid state by metals being electrodeposited the following model experiments were completed. Copper and nickel samples were obtained by the method of electrodeposition under the normal conditions (i.e. at the 1g overload). Then these samples were placed into cells with the electrolyte and were influenced along the texture axis by centrifugal force of the maximum value (at the 1256g overload) without the deposition current. In such cases centrifugal force influenced solid copper and nickel deposits after the process of electrodeposition. The comparative texture analysis of the initial samples and the ones influenced by the force action shows no differences.

The following features of change of the degree of perfection of texture in metals being electrodeposited at the force action during their phase formation should be mentioned. First, regardless to the direction of the force influence the most significant change of the degree of perfection of texture in metals being electrodeposited occurs at relatively low overloads. Thus, the most significant suppression of the process of texture formation (at the force influence opposite its axis) and the most essential intensification of the process of texture formation (at the force action along the texture axis) occur in metals being electrodeposited at the overload of 314g. The further force influence on metals being electrodeposited during their phase formation (up to 1256g overload) causes only minor change of the degree of perfection of texture of the deposits.

Such influence of the force action on the process of texture

formation is not typical for solid metals, for which the formation of texture, as a rule, is directly proportional to the influence of the external force [15].

Second, the action of centrifugal force in the direction opposite to the texture axis causes much more significant influence on the process of texture formation in comparison with the force influence along the texture axis. Thus, at the force action along the texture axis during copper electrodeposition the overload of just 35g results in the decrease of the α angle approximately by 1°, which is a significant growth of the degree of perfection of texture (Fig. 7), and the force action of the same overload opposite the texture axis almost completely suppresses the process of texture formation of copper being electrodeposited (Fig. 4).

The similar result is obtained from the comparison of quantitative characteristics of texture [210]+[221] of nickel electrodeposited at the force influence opposite and along the texture axes. Thus, at the action of centrifugal force proportional to the overload of 314g in the direction opposite to the texture axes the average dispersion angles of the orientations [210] and [221] increase approximately two times (Fig. 3). At the action of centrifugal force proportional to the overload of 314g along the texture axes the average dispersion angles of the orientations [210] and [221] decrease just 1.2 times (table 2).

The described feature of texture formation is also not typical for metals in solid state, because it is known [15] that texture formation in solid metals and alloys the most intensively occurs at the external force influence along the texture axis. As for the suppression of texture formation of solid metals and alloys (i.e. at the obtaining of texture-free reference samples), this task is hardly accomplishable.

Summarizing the obtained results, it is possible to make a conclusion that the effect of intensification of the process of texture formation in metals being electrodeposited under the influence of external force directed along the texture axis is experimentally found. At that, the action of external force in the direction opposite to the texture axis causes much more significant influence on the process of texture formation in comparison with the force action along the texture axis. And regardless to the direction of the force influence the most significant change of the degree of texture perfection of metals being electrodeposited occurs at relatively low overloads. The obtained results prove the validity of the phenomenon of phase formation through a stage of liquid state in metals being electrodeposited.

3. Conclusions

The investigations of the features of formation of crystallographic texture in metals being electrodeposited under the influence of external (centrifugal) force in the directions opposite and along the texture axis were completed. The effect of suppression of the process of texture formation in metals being electrodeposited up to complete disordering of crystal lattices of grains under the

influence of external force in the direction opposite to the texture axis was discovered. The effect of intensification of the texture formation process in metals being electrodeposited at the influence of external force directed along the texture axis was also found.

The comparative analysis of the degree of texture perfection of electrodeposited metals obtained at different directions of the force action during their phase formation was performed. It was found that regardless to the direction of the force influence the most significant change of the degree of texture perfection in metals being electrodeposited occurs at relatively low overloads. It was discovered that the action of external force in the direction opposite to the texture axis results in the more significant influence on the texture formation process in metals being electrodeposited in comparison with the force action along the texture axis.

The obtained results prove the validity of the phenomenon of phase formation through a stage of liquid state in metals being electrodeposited.

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