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Розглянуто особливості розробки економічно-легованого титаном алюмінієвого сплаву, володіючого високою міцністю і термостійкістю, для виготовлення виробів електротехнічного призначення. Представлені методи і засоби, а також прилади та обладнання, які використані при експериментальних дослідженнях. Вивчено вплив кількості титану та температури заливки на структуру і властивості економічно легованого сплаву

Ключові слова: алюмінієві сплави, легування, титан, міцність, термостійкість, електротехнічні вироби

Рассмотрены особенности разработки экономно легированного титаном алюминиевого сплава, обладающего высокой прочностью и термо-стойкостью, для изготовления изделий электротехнического назначения. Представлены методы и средства, а также приборы и оборудования, которые использованы при экспериментальных исследованиях. Изучены влияния количества титана и температуры заливки на структуру и свойства экономно легированного сплава

Ключевые слова: алюминиевые сплавы, легирование, титан, прочность, термостойкость, электротехнические изделия

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DEVELOPMENT OF LEAN TITANIUM- ALLOYED ALUMINIUM ALLOY FOR ELECTRO- TECHNICAL PURPOSES

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1. Introduction

Power engineering has intensively developed in today's world, due to which leading countries export electricity to neighboring developing countries. In this case, power engineers face the task on electricity generation and transmission over long distances. This, in turn, necessitates design of new materials for electro-technical purposes, as well as

priority development of metallurgical technologies and materials science [1, 2].

At present, given their high mechanical, physical-chemical and corrosive properties, as well as high manufacturability, aluminum alloys are widely used in engineering. Another reason, contributing to a wide application of aluminum alloys, is the availability of large amounts of reserves of aluminum ores in the world.

Some countries also have extensive material and technical potential in the field of production of aluminum alloys. For example, in Azerbaijan, there are several major industrial enterprises, which employ the electrolysis of aluminium oxide to obtain aluminium of varying purity, used for manufacturing aluminium alloys and products for electro-technical purposes.

Due to its unique characteristics, among which high conductivity plays the most important role, aluminium is widely used in many industries, including electrical engineering [2, 3].

Aluminium alloys are known to be widely used for manufacturing wires of high-voltage power lines. Due to a decrease in electrical conductivity to a certain extent, addition of alloying elements to aluminium alloys is limited, resulting in the fact that wires are produced from aluminium or from the alloys of the Al-Si-Mg system [3, 4].

Recently, a great interest in development of alloys with high strength and thermal stability has been observed in the power sector. Requirements for wires, produced from such alloys, include high electric conductivity and retention of strength at temperatures of up to 300 °C [4, 5].

Since there is a serious demand for such alloys, the attempts to develop them are being made in the CIS countries. Development of aluminium alloy with rational alloying by one or two transition metals and manufacturing of electro-technical products from this alloy with application of existing technological capabilities is the most suitable option for the conditions of Azerbaijan. That is why, development of lean-alloyed aluminium alloys and implementation of technology of manufacturing products for electro-technical purposes from them with the use of industrial mass-production equipment is a serious scientific and technical challenge of great practical importance.

2. Literature review and problem statement

One of the most important stages in this technology is determining the structure and properties of melted cast and deformed aluminium alloys, as well as rolling slabs, manufactured from such alloys.

It was established [4, 6] that existing aluminium alloys, alloyed with transition metals have thermal resistance of up to 250 °C, however, high costs of production of electric wires from them, based on deformation, limits a wide application of these alloys. Metastable intermetallic compounds, which are separated in the form of primary dendrites during crystallization, are formed in binary alloys of the Al-Me system. As a result, effect of solid-solution strengthening by alloying elements decreases and the possibility of occurrence of fragile phases increases [3, 4]. Due to the formed properties, such systems have a wide scope of application [7], which extends through a rational selection of compositions and purposeful influence on the alloy structuration process [8, 9]. The impact factors, considered in the works, apply only to the alloys, based on the Al-Be system. Paper [10] considers alloying of aluminium alloy A 6063 by titanium carbide, due to an increase in the amount of which during alloying, impact ductility and elongation of composites decreased up to 31 % and 35 % respectively. However, as the authors note, density, hardness and breaking strength of the alloys increased up to 7.8 %, 20 %, and 19.55 % respectively. Results of X-ray

diffraction showed that Al and TiC are major elements in composites and do not contain intermetallics. However, microstructural analysis revealed voids and tears in the fracture of the samples.

Alloying with insignificant amount of Zr (0.1 %) and Ti (up to 0.2 %) was conducted for development of aluminium alloy which combines high mechanical and casting properties [11]. Obtained results led to the conclusion about certain merits of such alloys as compared to corresponding properties in the commercial alloy A 356. Specifically, it was observed that when the content of Ti increased, there was a concave character of change in fluidity in medium-thick walls of the mould and a monotonous increase in thin-walled fluidity. In this case, addition of Ti resulted in a decrease in tendency to hot tear shaping (HTS), which eventually reached zero. Moreover, a decrease in a grain size and a change in morphology due to a combined addition of 0.2 % Ti and 0.1 % Zr resulted in improvement of casting properties of an alloy. However, these results apply to cast alloys and problems of application of other technological solutions were not considered. In particular, the problem of deformed aluminium alloys and the influence of the structure and properties of additives in the Al-Me systems on formations remained unresolved. The gap in findings in this part of research is compensated in paper [12]. It shows that the amount of alloying elements should be limited due to the negative influence of primary crystals, which occur in the Al-Me system, on the structure and properties of cast and deformed aluminum alloys. Addition of a small amount of Cr, Mn, Zr, Sc and Ti as alloying elements to the Al-Si-Fe system can be considered a promising direction, although not fully refined. The proof to this can be also found in the above-mentioned articles [5, 6], as well as in [13]. It shows, for example, that alloying metal alloy Al-Cu-Mn leads to formation of nanocrystalline supersaturated Al-based solid solution with zirconium content of up to 20 %. Microhardness of the alloy increases up to 440 HV with an increase in zirconium concentration. Subsequent deposition of metastable phase of Al₃Zr (L12) with a maximum content of 28 % by volume leads to an increase in microhardness up to 520 HV. However, this study was conducted on the powder Al-Cu-Mn alloy and the use of other experimental materials – cast or deformed alloys – can lead to a difference in results. It should also be noted that it would be useful to consider technological aspects of obtaining products from such alloys, regarding, for example, thermal treatment of alloyed Al-Me systems. In this regard, studies, described in [14–16], can be useful.

Thus, paper [14] explored semi-solid slab form aluminum alloy 7005, manufactured with the use of recrystallization and partial remelting (RAP), then tixofomed at different isothermal temperatures, preheating temperatures and at different loading routes. The results showed existence of a spheroidal microstructure resulting from three-stage thermal extrusion with inductive heating. It was found that an increase in isothermal temperatures resulted in a small decrease in mechanical properties of tixoshaped product due to consolidation of solid grains. The highest fluidity level, tensile strength level and elongation of tixoshaped components with thermal treatment make 237 MPa, 361 MPa and 16.8 %, respectively, achieved at isothermal temperature of 605 °C. It was also shown that loading route has a significant influence on mechanical properties and the microstructure of a tixoshaped product.

Paper [15] shows results of the impact of thermal treatment modes at 180, 240 and 320 °C over 1, 30, 50, 70, 90, and 120 min on the quality of alloy AA 7075. They made it possible to conclude that the influence of temperature and duration of the process on impact ductility and hardness are associated with precipitates on the grains' boundary.

A change in the microstructure and properties with a prior deformation of aluminium alloys 2219 when using the ageing mode is described in paper [16]. As it follows from the results, shown in it, prior deformation leads to an increase in dislocation density in a matrix, which, in turn, leads to formation of dense and less deposited θ' -phases with dispersion distribution in α -matrix. This in turn leads to an increase in strength of previously deformed samples.

The results, described in papers [14–16], do not refer to the field of studies, linking alloying parameters to thermal treatment modes, which would be of practical value and would increase the amount of primary data for theoretical research. It would make it possible to extend theoretical notions about the Al-Me system. In this regard it is also necessary to mention the need for a substantiated choice of an alloying element that is associated with the alloy application range. The Al-Ti systems are of interest in the case of electrical industry, taking into account analysis of the current state of the problem in the field of alloying and thermal treatment of the Al-Me systems. In this case, theoretical background of titanium-alloying of aluminum alloys is the diagram of state of Al-Me (Fig. 1). According to this diagram, the alloy is characterized by the presence of a broad area of solid solution, based on TiAl compounds [12, 17].

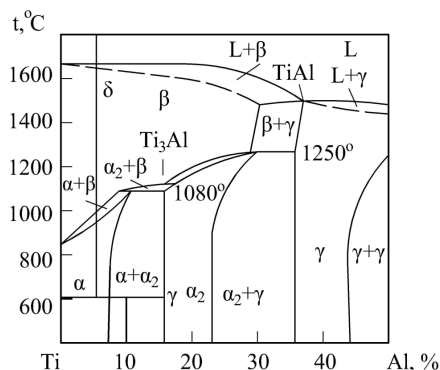


Fig. 1. State diagram of Al–Ti

It should also be said that it is advisable to use titanium as an alloying element, positively influencing the structure and properties of aluminum alloys for products for electro-technical purposes [18–20]. Development of this research can be selection of rational amount of alloying element and optimization of alloy obtaining modes. In fact, speaking of electrical products that require high strength, heat resistance and stable electrical properties, it is necessary to mention especially the need to achieve a set of properties of an alloy. They include specified strength, low electrical resistance and high thermal stability. The solution to this problem is possible with the use of the lean-alloying technology.

3. The aim and objectives of the study

The aim of present research is to develop aluminum alloy and the technology of manufacturing products for

electro-technical purposes from it by formation of a rational composition and the structure of material with specified strength and electrical properties.

To accomplish the set goal, the following problems must be solved:

- to explore the structure and properties of aluminium alloys;
- to assess the effect of various alloying elements and identify relationships between their composition, structure and properties;
- to develop technologies for manufacturing a lean-alloyed alloy and the rolling products made from it.

4. Materials and methods of research

The studies were conducted at two stages: the compositions of the experimental alloys were developed under laboratory conditions, then various physical-mechanical and electrical properties of these alloys were studied.

Experimental alloys are composed by adding various alloying elements in the double Al-Si system.

Ingots, slabs, sheets and wires of different shapes and sizes were manufactured as a result of the cold and hot deformation

Chemical compositions of experimental alloys are given in Table 1.

Aluminium alloys were melted in graphite crucibles in 50-kg electric furnace of resistance. Liquid metal was poured in special steel metal moulds at the temperature of approximately 750 °C. Within crystallization interval, the average rate of cooling metal, poured in the mould, was 10–20 K/s. Rectangular ingots of dimensions of 15×30×200 mm were cast from the liquid alloy.

The ingots, previously heated in electric furnaces CHOL-2.0-M2, were subjected to thermal treatment, enabling cooling at the rate of approximately 10 K/s. Depending on the purposes of thermal treatment, samples were heated up to a certain temperature, kept at this temperature, then cooled both in the oven, and in the air.

Deformed slabs were obtained in the laboratory by preliminary and intermediate heating of ingots for about 10–12 transitions using a laboratory rolling machine. The thickness of the sheet-like slabs had approximate degree of deformation of 90 %.

Using different modes of deformation and thermal treatment, under conditions of the Sumgait Technology Park (STP), the wires from ingots of 300 mm in length and 40 mm in diameter were obtained.

Chemical composition of aluminum alloys was determined by the emission spectrometer ARL 1583 in the technological laboratory of STF. As a result of the use of intermediate annealing, the sheets of the thickness of ~2.0 mm were subsequently obtained from the ingots, subjected to cold deformation.

Intermediate annealing was carried out in the stage-by-stage mode, at temperature below 500 °C. Final annealing was performed within 1, 10 and 100 hours at the temperature of 300 °C.

Composition of the experimental aluminum alloy was formed by the following procedure: the minimum titanium amount in the alloys of series 1 was 0.15 %, and it was 0.45 % in the alloys of series 3.0.10 – 0.14 % Fe and up to 0.10 % of Si was included in the composition of all alloys as a constant

additive. Liquidus temperature of the experimental alloys is in the range of 730–800 °C.

Table 1

Composition of experimental aluminum alloys

| No. | Amount, mass % | | | | $T_L, ^\circ\text{C}$ | Ingot | Deformed state |
|----------|----------------|-------|-------|------|-----------------------|-------|----------------|
| | Ti | Fe | Si | Al | | | |
| Series 1 | | | | | | | |
| 1 | 0.15 | 0.12 | 0.04 | base | 724 | ingot | wire |
| Series 2 | | | | | | | |
| 2 | 0.28 | <0.01 | <0.01 | base | 730 | slab | rod |
| 3 | 0.30 | <0.01 | <0.01 | base | 760 | rod | wire |
| 4 | 0.26 | <0.01 | <0.01 | base | 750 | ingot | rod |
| Series 3 | | | | | | | |
| 5 | 0.42 | 0.10 | 0.06 | base | 780 | ingot | wire |
| 6 | 0.45 | 0.12 | 0.05 | base | 800 | slab | rod |
| 7 | 0.40 | 0.14 | 0.07 | base | 770 | rod | slab |

Specific electric resistance of flat samples with standard dimensions was measured by the digital millimeter OM-2 in order to assess the effect of alloying elements on electrical properties of aluminum alloys.

The studies of metallographic structures were carried out with the use of optical, scanning and electronic microscopes. Sophisticated studies of metallographic structures were carried out with the help of the scanning probe microscope Integra Prima.

The samples, cut out from the central part of the ingots, obtained from thermally treated aluminium alloys, poured in metal moulds, were taken as research objects. Microsections from such samples were prepared on a set-up for electrolytic polishing and etching.

5. Results of the study of influence of technological modes of obtaining aluminium alloy on electrical and strength properties

The influence of the amount of alloying elements and technological parameters of thermal and deformation treatment on the structure and properties of mass-produced and experimental aluminum alloys was explored. At the first stage of the study, we compared the structure and properties of ingots, as well as of wires, made from mass-produced alloys A5E, A7E, and lean-titanium-alloyed alloys.

It was found that the slabs, produced by deformation of the mass-produced aluminum alloys, possess the ability to work at relatively low temperatures and significantly lose their strength properties at the temperature of 300 °C. A decrease in mechanical properties in some cases is more than twice, and softening of wires due to the temperature effect is observed.

It was found that a decrease in mechanical properties and an increase in plasticity of slabs, obtained from the mass-produced aluminum alloys at high temperatures, are explained by the recrystallization process that occurs during annealing. In this case, intermetallic compounds, formed in the aluminium-based solid solution, cannot prevent softening in the structure of rolling products.

It was found that addition of alloying elements to aluminum alloys significantly decreases softening, occurring in the structure due to temperature influence. As a result,

dependence of mechanical properties of slabs on the temperature of thermal and deformation treatment decreases.

Improvement of mechanical properties of slabs can be explained by formation of phase TiAl_3 and disperse crystals of the Al_3Fe type in a aluminium-based solid solution (Fig. 2).

As we can see, particles of dispersion of the order of 200 μm prevail in the microstructure of ingots form mass-produced aluminium alloy, whereas a more fine-grained structure is observed in the titanium-alloyed alloy.

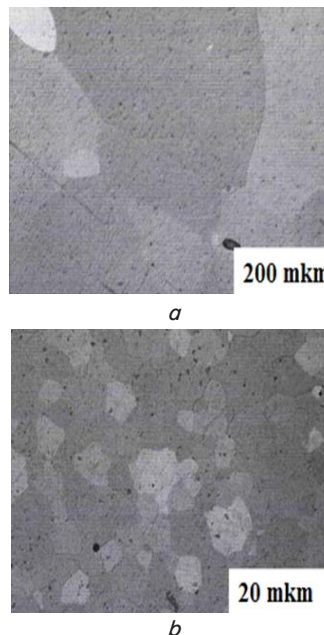


Fig. 2. Microstructure of ingots from mass-produced and lean-alloyed aluminum alloys: a – A7E; b – 0.15 % Ti

It is shown that economical addition of titanium to aluminium alloys changes this picture. Strength properties of alloyed alloys change in a positive direction and load-lifting capacity of sheets and wires, made of these alloys at temperatures of 280–350 °C, considerably increases (Tables 2, 3).

This synergistic effect is explained by formation of heat-resistant dispersoids in solid solution as a result of annealing in titanium-alloyed aluminium alloys. Disperse particles prevents formation of larger particles in the matrix during crystallization.

Table 2

Influence of annealing on mechanical properties of sheets

| Alloy | Annealing mode | σ_b, MPa | $\sigma_{0.2}, \text{MPa}$ | $\delta, \%$ |
|-----------|----------------|------------------------|----------------------------|--------------|
| A7E | no | 160 | 148 | 5.0 |
| | 280 °C | 102 | 100 | 9.0 |
| 0.45 % Ti | no | 180 | 170 | 5.0 |
| | 280 °C | 182 | 172 | 8.0 |

It was found that an increase in titanium amount in the composition by more than 0.5–0.6 % has a negative influence on electrical properties of aluminium-based alloy. Specific electric resistance has a minimal value during annealing of ingots, cold-treated sheets and wires in the range 280–480 °C (Fig. 3, Table 4, 5).

Table 3

Influence of annealing on mechanical properties of wires

| Kind of alloy | Wire diameter | Annealing mode | σ_b , MPa | $\sigma_{0.2}$, MPa | δ , % |
|---------------|---------------|----------------|------------------|----------------------|--------------|
| A7E | 1.4 mm | no | 180 | 160 | 3.2 |
| | | 280 °C | 78 | 40 | 4.2 |
| 0.45 Ti | 1.4 mm | no | 270 | 242 | 2.6 |
| | | 280 °C | 192 | 180 | 7.2 |
| | | F1+350 °C, 1h | 180 | 160 | 8.2 |
| | | F1+400 °C, 1h. | 166 | 140 | 9.7 |
| | 3.8 mm | no | 240 | 210 | 3.0 |
| | | 280 °C | 190 | 170 | 7.4 |

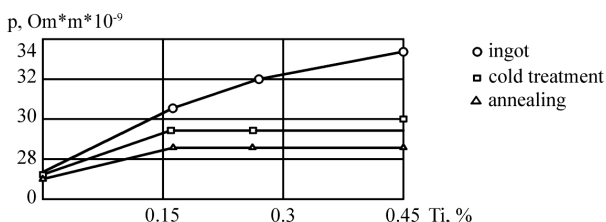


Fig. 3. Influence of titanium content on specific electric resistance of sheets

Table 4

Dependence of specific electric resistance of annealing time

| No. of entry | Alloy | Annealing time at 280 °C, h | | | | | |
|--------------|---------|-----------------------------|-----|-----|------|-------|-------|
| | | 0 | 1 h | 8 h | 80 h | 160 h | 480 h |
| 1 | A7E | 28 | 28 | 28 | 28 | 26 | 26 |
| 2 | 0.15 Ti | 30 | 28 | 26 | 24 | 22 | 20 |
| 3 | 0.30 Ti | 32 | 30 | 28 | 26 | 24 | 22 |
| 4 | 0.45 Ti | 34 | 32 | 31 | 30 | 29 | 28 |

Thus, it was determined that in order to achieve the optimum combination of the physical-mechanical and electrical properties, chemical composition, structure and modes of deformation and thermal treatment, it is necessary to establish their reciprocal influence (Table 5).

Table 5

Influence of annealing temperature on tensile strength (MPa) of cold-rolled sheets

| No. of entry | Alloy | Annealing temperature, °C | | | |
|--------------|--------|---------------------------|-----|-----|-----|
| | | 0 | 280 | 380 | 480 |
| 1 | A7E | 100 | 80 | 60 | 40 |
| 2 | A5E | 80 | 70 | 60 | 50 |
| 3 | 0.15Ti | 140 | 135 | 130 | 120 |
| 4 | 0.30Ti | 160 | 150 | 140 | 120 |
| 5 | 0.45Ti | 180 | 170 | 160 | 140 |

6. Discussion of results of the study of technological possibilities for obtaining high electrical and strength properties of aluminum alloys

Obtained results prove that it is possible to make some positive influence on the structure and mechanical properties of Al-Ti alloys by controlling the casting temperature and cooling rate. Thus, it was found that the more positive impact on the structure can be achieved at significant

overheating of alloyed alloys compared to mass-produced technical aluminium alloys. Obtaining of highly dispersed structure can be ensured only at casting temperature that is higher than melting temperature of the given alloy.

It was determined that cooling rate has a significant influence on the structure and properties of ingots, obtained from titanium-containing alloys. The highest cooling rate leads to formation of a structure with smaller particles.

It was found that an increase in load-lifting capacity of lean titanium-alloyed aluminium alloys is possible through rational alloying of alloys together with other transition elements. However, since such alloying is expensive and is associated with technical difficulties, its use is inappropriate.

The given results of experimental research on the influence of deformation and thermal treatment modes on mechanical and electrical properties of alloys of the Al-Ti system are of great practical importance. The same can be said about the proven fact of the influence of deformation and thermal treatment parameters on mechanical properties and electrical resistance of non-alloyed and lean titanium-alloyed aluminum alloys.

It was found that in mass-produced aluminium alloys, annealing, performed after cold deformation in the range 280–400 °C, considerably decreases their strength properties and improves plasticity. That is why products for electro-technical purposes from such alloys do not meet requirements for their thermal stability and load-lifting capacity.

The influence of Fe and Si amount on the structure and properties of aluminium alloys was studied. It was revealed that four types of phases are possible to be formed in complex-alloyed alloys and these phases generally serve to preserve tensile strength after intermediate annealing.

There is a positive influence of cold deformation and intermediate annealing on formation of rational structure and a good combination of electrical and strength properties. Fig. 4 shows microstructure of the alloy with 0.30 % Ti content after annealing for 80 hours, sub-particles and dispersoids of the TiAl₃ phase

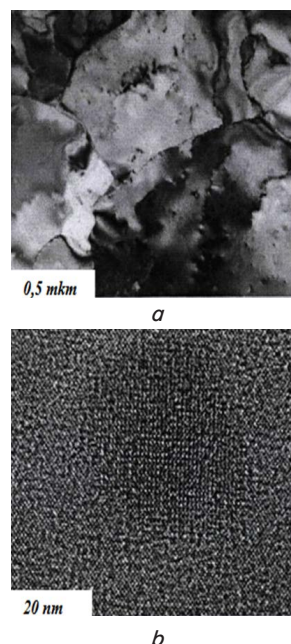


Fig. 4. Microstructure of alloy with 0.15 % Ti content after annealing for 80 hours: a – sub-particle; b – dispersoids of TiAl₃ phase

To verify the obtained results, we carried out practical tests of lean-alloyed aluminum alloys for electro-technical purposes on the technological line of obtaining rods under conditions of STP (Fig. 5). It was found that the optimum alloy for this purpose can be lean-alloyed Al-Ti alloy.

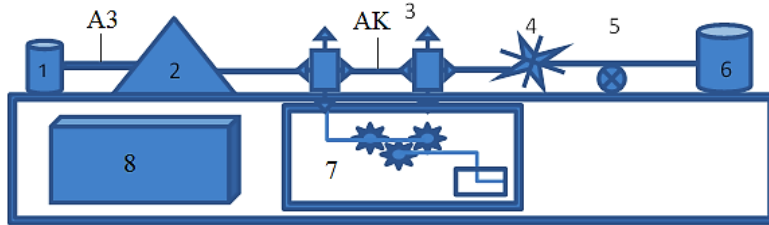


Fig. 5. Technological scheme of production of aluminium rod: 1 – mixer; 2 – copper bandage; 3 – cooling line; 4 – scissors; 5 – guide to coiler; 6 – coiler; 7 – cooling system depot; 8 – oil-emulsion depot; A3 – aluminium alloy; AK – aluminium rod

However, if technological parameters are selected incorrectly, primary dendrite crystals that have a negative impact on electrical and mechanical properties of a solid aluminium-based solution, as well as on the properties of finished products, can be formed in the structure.

7. Conclusions

1. Chemical composition, microstructure and physical-mechanical and technological properties of mass-pro-

duced and experimental aluminum alloys were studied. It was found that mass-produced aluminium alloys do not meet requirements for thermal stability and load-lifting capacity. It was determined that lean titanium-alloyed aluminum alloys have better mechanical and electrical properties, which is explained by formation of heat-resistant dispersoids in solid solution.

2. The influence of various alloying elements on the structure and properties was assessed, and relationships between the composition, structure and properties of the titanium-alloyed aluminum alloy were identified. It was found that an increase in the amount of titanium by more than 0.5–0.6 % has a negative influence on electrical properties of the aluminium-based alloy. It was revealed that formation of four types of phases in complex-alloyed Fe and Si alloys contribute to tensile strength.

3. The technology of production of lean titanium-alloyed aluminium-based alloy and rolling electrical products from it was developed. It was found that aluminum ingots, cold-treated sheets and wires preserve the necessary strength and minimal specific electrical resistance at high enough temperatures. A positive influence of cold deformation and intermediate annealing on formation of a rational structure and a good combination of electrical and strength properties of the products was revealed.

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Запропоновано отримання багатощарових електрохромних плівок на основі $Ni(OH)_2$ і $Co(OH)_2$ катодним темплатним методом. Отримані плівки показали електрохімічну активність і електрохромні властивості. Найкращі електрохромні характеристики показала плівка, яку отримували послідовно в розчинах із додаванням полівінілового спирту, що містять нітрат кобальту і нікелю 2 і 78 хвилин відповідно

Ключові слова: $Ni(OH)_2$, $Co(OH)_2$, електрохромізм, електрохромні матеріали, $CoOOH$, полівініловий спирт, багатощарові покриття

Предложено получение многослойных электрохромных пленок на основе $Ni(OH)_2$ и $Co(OH)_2$ катодным темплатным методом. Полученные пленки показали электрохимическую активность и электрохромные свойства. Наилучшие электрохромные характеристики показала пленка, которую получали последовательно в растворах сдобавлением поливинилового спирта, содержащих нитрат кобальта и никеля 2 и 78 минут соответственно

Ключевые слова: $Ni(OH)_2$, $Co(OH)_2$, электрохромизм, электрохромные материалы, $CoOOH$, поливиниловый спирт, многослойные покрытия

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A STUDY OF MULTILAYERED ELECTROCHROMIC PLATINGS BASED ON NICKEL AND COBALT HYDROXIDES

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1. Introduction

Electrochromism is a phenomenon, which is characterized by substance changing its optical properties under applied electrical current. Electrochromic materials can change different optical characteristics: color, transparency, opacity, reflectivity. Electrochemical systems often find application in electrochromic devices. Materials, in which electrochemical processes occur, are characterized by chang-

es of optical properties which occur in parallel to changes in composition and oxidation state of elements that constitute the compound.

Electrochromic devices allow controlling the amount of light and heat that pass through. The properties can be used for various purposes. Creation of temperature regime and suitable lighting, visual separation of rooms, creation of mirrors with adjustable reflectivity, creation of indicators for slow changing values (environment temperature, pressure,