Printed as manuscript

Федорова

Olga V. Fedorova

RESEARCH OF INFLUENCE OF THE TYPE AND PROCESSING MODES ON STRUCTURE AND PROPERTIES OF THE CAST AND THE DEFORMED SEMI-FINISHED PRODUCTS FOR ELECTROTECHNICAL PURPOSES FROM ALUMINUM–ZIRCONIUM SYSTEM ALLOYS

Master's Program Metal and Alloys Forming under Pressure

The abstract of the Master's Thesis

Krasnoyarsk 2014

The thesis work is done at the Department of «Metal Forming Under Pressure» Federal State Autonomous Educational Institution of Higher Professional Education «Siberian Federal University»

Scientific supervisor:
Nikolai N. Dovzhenko, Doctor of Technical Sciences, Professor

Peer Reviewer:
Tatyana A. Bogdanova, Head of the metallurgical department LLC «K&K»

Defence takes place on July 2, 2014 at FSAEI HPE "Siberian Federal University":
95, Krasnoyarsky rabochiy ave, 104 lecture room, Krasnoyarsk 660025, Russia

Head of the Master's program:

Doctor of Engineering,
Professor  Sergey. B. Sidelnikov

INTRODUCTION

Actuality of the work.

The growth in electricity consumption leads to increasing consumer demands for electrical, mechanical and performance properties of the wire used in power cables and wires of electric conductor.

The United Company "RUSAL" is a major Russian manufacturer of wire rod for electrotechnical purposes. At factories UC RUSAL wire rod of aluminum (GOST 13843 -78) and alloy ABE (GOST 20967 -75) are manufactured. An important property of technically pure aluminum is its high electric conductivity but along with this it has the comparatively low strength properties. Alloys of AlMg-Si system are characterized as having higher strength properties and increased specific electrical resistance in the thermal strengthening condition. Recently there has been an increased interest to the promising thermostable alloys of Al-Zr system, which retain the high electrical conductivity and strength in the process of operating the heating up to the temperature 310 ° C.

Currently in Russia there are no aluminum alloys produced industrially, which would satisfy the requirements of international standards ASTM B 941-05 and IEC 62004-09. Therefore for manufacturers of wire rod and wire in conditions of increasing requirements to the electroconductive materials the most acute problem is the creation of new alloys and technology for producing electrical semis of them on the working industrial equipment.

Technology optimization for producing wire rod for electrotechnical purposes with given set of properties in the international standards determines the need of studying patterns of influence of the alloy composition and technological modes of casting, deformation and heat treatment on the structure and properties. The Master’s thesis is devoted to the establishment of such objective laws.

This Master's thesis was performed while realizing the state program support of cooperation development and the use of subsidies FSAEE HPE " Siberian Federal University " and LLC " RUSAL ITC ", jointly participating in the framework of the contract № 13.G25.31.0083 with the Ministry of Education and Science of Russia in the implementation of a comprehensive project to establish high-tech production on the following theme: " Development of technology for production of aluminum alloys with rare earth , transition metals and high-performance equipment for the production of electrical wire rod ."

Purpose of the work.

The investigation of the influence of kinds and processing modes of cast and deformed semi finished products in order to obtain higher complex of properties in accordance with the international standards of ASTM B 941-05 and IEC 62004-09 is the purpose of the work.

To achieve this goal the following tasks were necessary to be solved:

1. To investigate the influence of cutting-edge technologies of producing wire rods for electrotechnical purposes on structure and properties, to choose the resource-saving technology.

2. To investigate the influence of the alloying elements concentration and casting parameters on the properties of the wire rod. To recommend a promising alloy composition and the mode of casting.

3. To calculate the route of cold wire drawing, form changing and power parameters.

4. To conduct an experimental-industrial testing of recommended modes for wire rods and wires obtaining, satisfying the requirements of international standards ASTM B 941-05 and IEC 62004-09.

Object of research.

The object of research is the production technology of wire rods and wires from experienced alloys Al-(0,12-0,36)%Zr-(0,20-0,28)%Fe.

Subject of study.

The subject of study is as follows: methods and ways to increase the properties of the deformed semi finished products using energy efficient deformational and thermal treatment; achievement the properties in accordance with the requirements of international standards of ASTM B 941-05 and IEC 62004-07.

Scientific novelty of the work.

1. It's been found that the achievement of the maximum values of strength and electrical conductivity of wire rods from Al-(0,12-0,36)%Zr-(0,20-0,28)%Fe alloys provides a method for the combined rolling-pressing.

2. The technological parameters of aluminum wire rods production from Al-0,27%Zr-0,20%Fe alloy by the combined rolling-pressing and drawing method were grounded by means of using the calculated and experimental methods.

3. On the basis of the rods properties regularities changing in the concentration of zirconium studying the following dependencies were obtained:

Changing of specific electric resistance (hereinafter on the text SER) is described by the equation $\rho_{20}=0,03 +0,0179 \cdot c_{Zr}$; changing of tensile strength is described by equations $\sigma_v=101,7 +118,3 \cdot c_{Zr}$ and $\sigma_v=145,8-12,3 \cdot c_{Zr}$ (for alloys with 0,20% and 0,28% Fe, respectively).

4. It was revealed that at the temperature of melt 900 ° C and the temperature of casting 800 ° C zirconium is completely dissolved in the solid solution, forming the solid solution of maximum supersaturation. Reducing the melt temperature to 800 ° C leads to undesirable segregation primary crystal phase of Al_3Zr.

Practical importance of the work.

1. An alloy Al-0,27%Zr-0,20%Fe has been recommended. It allows to reach the mechanical properties and SER regulated in the international standards ASTM B 941-05 and IEC 62004-09 in the deformed semi finished products.

2. Technological recommendations for the production of aluminum wire rods and wires from Al-0,27%Zr-0,20%Fe alloy by the combined rolling-pressing and drawing method have been developed.

3. Experimental-industrial testing of recommended wire rod and wire processing conditions allowed to reach thermal stability requirements AT1 and AT3 in accordance with standard IEC 62004-07.

Place of the thesis implementation.

The thesis was performed at the Department of Metal Forming Under Pressure in the Institute of Nonferrous Metals and Materials of the Federal State Autonomous Educational Establishment of Higher Professional Education "Siberian Federal University" together with the departments of metallurgy and heat treatment, the foundry production and Limited Liability Company «RUSAL ITC."

Place of international internship

The International Academy of Management and Technology (INTAMT) Dusseldorf, Germany. Advanced methods of sample preparation studying the structure and properties of materials were studied at the University of Applied Sciences (Fachhochschule Düsseldorf), on the equipment of Carl Zeiss under the direction of Andreas Schmidt.

Volume and structure of thesis

The thesis consists of an introduction, six chapters and a conclusion. It contains 144 pages of typewritten text, 40 figures, 32 tables, 26 formulas, bibliography of 63 positions.

THE MAIN CONTENTS OF WORK

1. Literature Review

This section describes the main methods of getting deformed semi finished from aluminum alloys, including modern energy efficient methods of combined pressure treatment.

Alloys for electrotechnical purposes manufactured in Russia and abroad have been analized. Zirconium was chosen for alloying the conductive aluminum alloys. The choice is based on the scientific research of Russian and foreign scientists.

2. Research methodology.

Low-alloyed aluminum alloys with additions of zirconium were smelted in a high-melter. For the alloys production zirconium in the form of briquettes from the HOESH firm were used. Ligature was introduced under a mirror melt at the temperature of 790 ° C.

The following technological schemes to produce the wire rod of 9 mm diameter were used for the research: hot high-quality rollings (HHQR) was accomplished on the mill "Ambifilo Veloce Rosen " (Mario Di Maio, Italy); combined rolling pressing (CRP) cast billets was carried out on the experimental setup of combined processing, assembled on the base of the mill duo 200; combined casting and rolling, pressing (CMRP) pressing was conducted in the experimental plant using the apparatus for continuous casting, rolling and pressing metal. Wires with diameters of 2-4,5 mm were obtained by method of cold drawing on the chain mill single action.

Specific electric resistance (hereinafter SER) of wire rod and wire was measured with an ohmmeter "VITOK" in accordance with GOST 7229-76. The tensile test of wire rod and wire was conducted on the machine Walter+Bai AG (Switzerland) in accordance with GOST 1497-84. Heat treatment of wire rod was carried out in electric furnaces RK10-1000/12 LAC and LAC RP20-540.

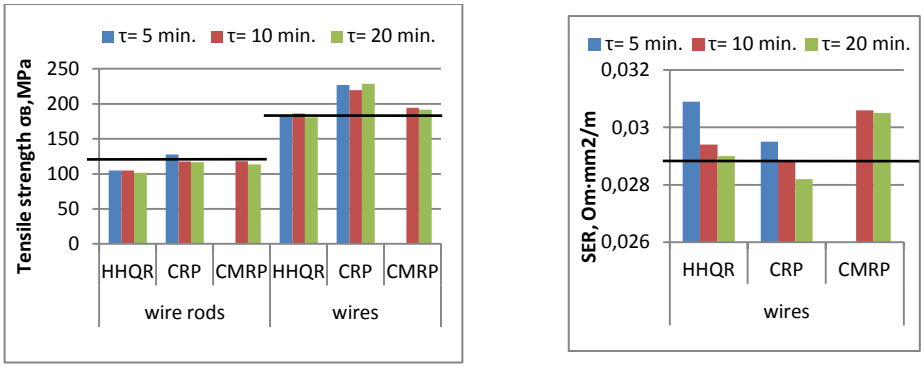
Investigation of the microstructure of alloys was performed on a microscope Carl Zeiss Axio Observer A1m.

Thermal stability test was performed in accordance with IEC 62004-09. Heat resistance of wire from studied alloys was evaluated by changes in tensile strength at room temperature after heating to temperatures of 230, 280 and 400 °C and holding for one hour.

3. Choice of resource-saving technology for wire rod and wire for electrical purposes manufacturing.

In the third chapter of master's thesis was the impact of casting parameters and methods of metal forming at the properties of deformed semis was evaluated. Studies were carried out on the alloy Al-0,15%Zr, which has the minimum SER and maximum tensile strength.

Analysis of tensile strength changes and specific electric resistance of deformed semi finished allowed to reveal that wire rods, received by HHQR and CMRP method does not satisfy the requirements of ASTM B 941-05 and subsequently does not provide the properties of the wire required by IEC 62004-09. It was found that high complex of mechanical and electrical properties of the deformed semi finished is achieved while manufacturing wire rod by CRP, Figure 1.



a b

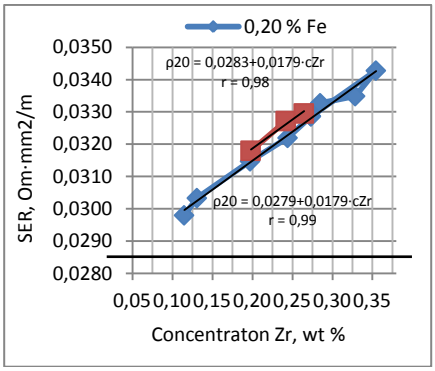
Figure 1 – The dependence of tensile strength (a) and SER (b) of wire rod and wire from the manufacturing technology , the exposure time of the melt and the casting temperature

Based on the above it is recommended to use the method of CRP for the manufacture of the deformed semi finished products satisfying the requirements of international standards, which allows you to reach in the wire from Al-0,15% Zr system alloy maximum values of strength and electrical conductivity. However, this alloy does not provide the necessary heat resistance, which is regulated by the standard IEC 62004-09. Therefore, further research has been focused on testing the composition of alloy and mode of preparation.

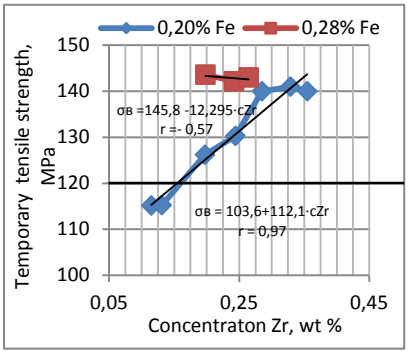
4. Investigation of the influence of alloying elements and processing modes on the wire rods structures and properties from Al-Zr-Fe system alloys.

In this chapter the analysis of the zirconium and iron concentration effect on the mechanical properties and wire rods SER was carried out, Figure 2. Zirconium was introduced to improve the thermal stability of alloys, and iron to increase the strength properties of deformed semis.

It was established that the investigated wire rods which are made from Al-(0,12-0,36)%Zr-(0,20-0,28)%Fe alloys do not meet the requirements of standard ASTM B 941-05 by SER, Figure 2, a. Furthermore, wire rods with low zirconium content do not meet by the temporary tensile strength, figure 2, b.



a



b

Figure 2 – The dependence of SER (a) and temporary tensile strength (б) of wire rods by concentration zirconium and iron in the alloy, where the c_{Zr}– concentration zirconium in the alloy; r– correlation coefficient

Dependency analysis revealed that with increasing concentration of zirconium SER increases, rectilinear dependence is observed, Figure 2, a. Such a logical SER raise with increasing concentration of zirconium confirms that it is dissolved in the aluminum. In alloys with 0,2% Fe the increasing of zirconium from 0,12 to 0,30 wt.% leads to an increase in temporary tensile strength, a further increase in the concentration of zirconium to 0,36 wt.% does not lead to a change in strength properties. Increase in iron concentration to 0,28% in alloys Al-(0,20-0,27)%Zr eliminates the influence of zirconium on the strength properties of the wire rods (Figure 2b). Equations describing the dependence of temporary tensile strength and SER from the concentration of zirconium in the alloy were derived. SER change is characterized by the equation $\rho_{20}=0,03+0,0179\cdot c_{Zr}$, Figure 2, a. Dependence of the change temporary tensile strength is described by equations $\sigma_v=103,6+112,1\cdot c_{Zr}$ and $\sigma_v=145,8-12,3\cdot c_{Zr}$ (for alloys with 0,20% and 0,28% Fe, respectively), Figure 2, b. Based on studies of the mechanical and electrical properties of wire rods of alloy Al-(0,12-0,36)%Zr-(0,20-0,28)%Fe is alloy Al-(0,27-0,3)%Zr-(0,2-0,28)%Fe providing high complex properties is selected.

Zirconium is capable to form the supersaturated solid solution during the casting, but temperature for preparation of the melt should be substantially higher compared to the conventional technology. Therefore the effect of melt temperature and pouring melt on structure and properties of wire rods from Al-0,30%Zr-(0,15-0,20)%Fe alloy was investigated. The melt preparation temperature was 800 and 900 °C and the casting temperature was 800 and 900 °C. The wire rods obtained by the CRP was investigated.

It was found that the temporary tensile strength of the wire rods from Al-0,30%Zr-(0,15-0,20)%Fe alloys is 135-148 MPa, the relative elongation of the wire rods is 15-25%, which is significantly higher than the requirements of standard

ASTM B 941-05. It was revealed that the temperature of the melt and pouring in the investigated interval has no significant effect on the mechanical properties of the wire rod. SER of the wire rod is 0,0314-0,0334 Om·mm^2/m, which does not comply with ASTM B 941-05. With an increase in the melt temperature from 800 to 900 °C, the specific electric resistance increases.

At the melt and casting temperature of 800 °C primary crystals of the Al_3Zr in the microstructure of the wire rods are found. Isolation of primary crystals of Al3Zr indicates that injected zirconium is not completely dissolved in the solid solution. The solid solution is depleted of by zirconium, because of the release of the primary crystals, this provides reduces the level of SER. At a melt temperature of 900 °C and a casting temperature of 800-900 °C zirconium is completely dissolved in the solid solution, forming the maximum a supersaturated solid solution, which leads to high values of SER wire rods.

Thus, to obtain the high quality cast billet alloys Al-0,27%Zr-0,20%Fe were chosen, as far as they have an increased content of zirconium, which subsequently will allow the preservation of required strength and heat resistance of the wire. The melt temperature of 900 °C and the casting temperature of at least 800 °C was recommended.

5. Crimping mode calculation in the process of drawing wires for Al - Zr system alloys.

In the fifth chapter calculations of cold drawing, form changing and energy-power parameters using V. I. Nepomnyastchy and V. N. Istomin's methods were performed. According to the above given methods the recommended reserve coefficients are in the range of 1,4-2,0.

The reserve coefficients have turned out quite large from 1,96 to 4,69 by the method of V. I. Nepomnyastchy, reserve coefficients are in the range 1,6-2,4 by the method H. V. Istomin. Drawing on the calculated route has not caused any difficulties in practice, so it was decided to shorten the route in order to bring the safety factor to the recommended interval. The obtained values of the factor of safety in all transitions are in the range of 1,4-2,13. Drawing on the 10 transitions flowed steadily without breaks.

According to the results of tensile tests a graph of tensile strength on the degree of deformation for alloys of Al-Zr was constructed, Figure 3.

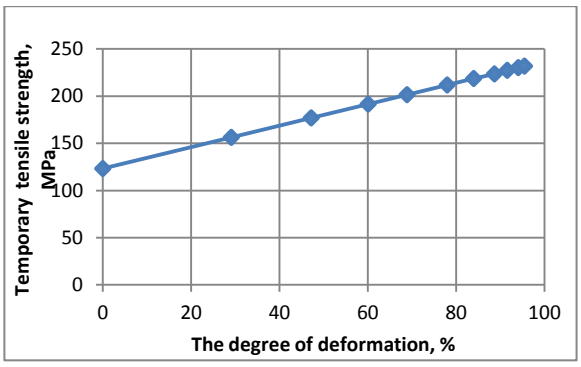


Figure 3 - The dependence of tensile strength on the degree of deformation alloys system Al-Zr

The obtained route of drawing in 10 transitions was used repeatedly and confirmed in practice.

6. Experimental-industrial testing of wire rods technology to obtain heat-resistant wires

In the final chapter methods to achieve the heat resistance of the wires were examined. For this wire rods according to the recommended regime on the Irkutsk Aluminium Smelter from the recommended alloy Al-0, 27% Zr-0, 20% Fe with the following properties: σ_v=147 MPa, ρ_{20}=0,03287 Om·mm^2/m, δ=6 % were manufactured.

For reducing the electrical resistivity the wire rods before drawing were subjected to a two stepannealing (geterogeniziruyuschemu) on the following regime: the first stage is heating to 350 °C, holding for 48 hours + the second stage, at which the concentration of alloying elements in solid solution due to the decomposition of the supersaturated by zirconium solid solution with the release of nano-sized particles of a metastable phase Al_3Zr decreased. Annealing allowed to reduce the wire rods SER up to 0,02824 Om·mm^2/m, at the same time the tensile strength decreased to 127 MPa and elongation increased to 16%. Wire rods property values after annealing satisfied the requirements of standard ASTM B 941-05.

At the final stage the experimental-industrial wires testing for the thermal stability was conducted, wires were manufactured from annealed wire rods from Al-0,27%Zr-0,20%Fe alloy. The tests were carried out on the cable factory JSC "Irkutskkabel" for compliance of the thermal stability types of AT1, AT3 and AT4. For this the wire rods were drawing to diameters of wire 4,5, 3,8 and 2,1 mm. Requirements for the testing temperature as well as for the mechanical properties of the wires are shown in Table 2.

Table 2 - Requirements the values of the thermal stability of wires from heat-resistant aluminum alloy

Type	T_h, ºC, during 1 h.	$T_{all.op.}$,ºC, during 400 h.	T_{op}, °C, during 40 years	ρ_{20}, nOm·m, no more	σ_v, MPa, not less than	δ, %, not less than
AT1	230	180	150	28,735	159–169	1,5–2,0
AT2	230	180	150	31,347	225–248	1,5–2,0
AT3	280	240	210	28,735	159–176	1,5–2,0
AT4	400	310	230	29,726	159–169	1,5–2,0

T_h – heating temperature when tested during 1 hour, ºC; $T_{all.\ op.}$ – allowable temperature of operation during 400 hour (ºC), not less than; $T_{op.}$ – allowable continuous working temperature of operation 40 years (°C), not less than.

Residual stress coefficient of the tensile, measured at room temperature after the warming wires to the temperature indicated in Table 2 should not be less than 90% compared with the original measured value. Test results are shown in Table 3.

Table 3 - Results of tests on thermal stability of the alloy Al-0,27%Zr-0,20%Fe

Ø wires, mm	Original state	The heating temperature is 230 °C (AT1)		The heating temperature is 280 °C (AT3)		The heating temperature is 400 °C (AT4)	
	σ_v, MPa	σ_v, MPa	α, %	σ_v, MPa	α, %	σ_v, MPa	α, %
4,5	167	164	98,2	167	100	151	90,4
3,8	172	171	99,4	166	96,5	154	89,5
2,1	199	199	100	192	96,5	167	83,9

α– residual voltage factor after heating, %.

Studies of the thermal stability showed that the wires from Al-0,27%Zr-0,20%Fe alloy correspond to the AT1 and AT3 types and are located on the border of the requirements type AT4.

CONCLUSIONS OF THE WORK

1. The study of modern technology of metal forming under pressure allowed to recommend the resource-saving technology of combined rolling-pressing for the wire rod manufacturing from Al-0, 15% Zr. system alloy, that satisfies the requirements of the standard ASTM B 941-05.

2. On the basis of studies of the alloying elements concentration effect on the wire rod properties the alloy Al-(0,27-0,3)%Zr-(0,20-0,28)%Fe providing high complex of mechanical and electrical properties was selected.

3.It was found that zirconium is completely dissolved in the solid solution forming maximally supersaturated solid solution, when the melt temperature is 900 ° C and the casting temperature is 800 ° C. The decrease temperature of the melt to 800 ° C leads to an undesirable separation of the primary crystals phase of Al_3Zr.

4. Calculation of cold drawing, form changing and power parameters allowed us to determine the route of drawing with the optimum values of the safety factor 1,4-2,13.

5. Wire rods obtained by the recommended mode on the Irkutsk Aluminium Smelter from the alloy Al-0,27%Zr-0,20%Fe after annealing satisfies the requirements IEC 62004-07.

6. Wires obtained in production conditions of JSC "Irkutskkabel" from the alloy Al-0,27% Zr-0,20%Fe, satisfies the requirements of the international standard IEC 62004-07 by the type of wires AT1 and AT3 with permissible continuously acting temperature of exploitation of 150-210 °C for 40 years, and is located on the border of the type AT4.

MAIN PROVISIONS OF DISSERTATION PUBLISHED IN THE FOLLOWING WORKS

1. **O. V. Fedorova,** V. A. Berngardt «Development of wire rods regimes annealing from Al-Zr system alloys to achieve a given complex of properties» //

Journal of Siberian Federal University, series «Engineering and Technologies». July 2014 (tome 7, Number 4). **(accepted for publication, the edition recommended HAC).**

2. Pat. 139085 The Russian Federation, IPK[7] B 21 C 23/08. Device for continuous rolling and pressing of wire rods of the nonferrous metals and alloys / **Fedorova O. V.,** Sidelnikov S.B., Dovzhenko N. N. et al.; The applicant and the patent-holder is Federal State Autonomous Educational Institution of Higher Professional Education «Siberian Federal University» B. – № 2013152037/02 ; application 21.11.13 ; published10.04.14, bulletin № 10. – 2 p.

APPROBATION OF RESULTS OF WORK

1. **O. V. Fedorova,** V. A. Berngardt, V. M. Bespalov «Investigation of the influence of species and modes of processing on the properties of deformed semifinished products of electrotechnical appointment» // Collection of scientific papers XIII International Science and Engineering of the School-Seminar metallurgists and young scientists of the Ural. 2012. – P. 268–270.

2. **O. V. Fedorova,** V. A. Berngardt, V. M. Bespalov «Influence of modes of heat treatment on structures and properties of the wires from Al-Zr system alloys» // Collection of scientific papers XIII International Science and Engineering of the School-Seminar metallurgists and young scientists of the Ural. 2012. – P. 326 – 328.

3. **O. V. Fedorova,** V. A. Berngardt, V. M. Bespalov «Investigation of manufacturing technology wires of electrotechnical appointment with improved performance properties» // Collection of scientific papers XIV International Science and Engineering of the School-Seminar metallurgists and young scientists of the Ural. 2013. – P. 250 – 252.

4. **O. V. Fedorova,** A. S. Sidelnikov «Investigation of the structure and properties of cast and deformed semifinished of the combined processing alloys of system Al-Zr» // Electronic collection of works of the Conference Youth and Science IX. 2013. Access mode: http://conf.sfu-kras.ru/sites/mn2013/thesis/s007/s007-024.pdf

5. **O. V. Fedorova,** V. A. Berngardt, V. M. Bespalov «Research of influence of the type and processing modes on properties of the deformed semi-finished products of electrotechnical appointment from Al-Zr system alloys» // Electronic collection of works of the Conference Youth and Science IX. 2013. Access mode: http://conf.sfu-kras.ru/sites/mn2013/thesis/s082/s082-011.pdf

6. **O. V. Fedorova,** V. A. Berngardt «Investigations of the influence of heat treatment on structure and properties of the wire rods from Al-Zr system alloys» // Electronic collection of works of the Conference Youth and Science IX. 2013. Access mode: http://conf.sfu-kras.ru/sites/mn2013/thesis/s007/s007-006.pdf

7. **O. V. Fedorova,** V. A. Berngardt, V. M. Bespalov, T. N. Drozdova. «Investigation of the influence of chemical composition and parameters of casting on the properties of semi-finished products of electrotechnical appointment» // Electronic collection of works of the Conference Youth and Science X. 2014. Access mode: http://conf.sfu-kras.ru/sites/mn2014/pdf/d03/s10/s10_015.pdf.