A R C H I V E S O F M E T A L L U R G Y A N D M A T E R I A L S
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STUDIES ON THE PROCESS OF HEAT TREATMENT OF CONDUCTIVE AlZr ALLOYS OBTAINED IN VARIOUS PRODUCTIVE PROCESSES

BADANIA NAD PROCESEM OBRÓBKI CIEPLNEJ PRZEWODOWYCH STOPÓW AlZr UZYSKANYCH W RÓŻNYCH PROCESACH PRODUKCYJNYCH

A novelty in electroenergetics of the recent years are anti-blackout conductors of the HTLS type. These conductors allow for an above average increase of the transmission capacity of electric energy through overhead lines. The high temperature work of such conductor is possible with the use of thermally resistant aluminium alloys with the addition of zirconium. Compared to the traditionally applied material of the EN AW 1370 class, those alloys' permissible work temperature is at the level of 150 to 230°C (aluminium 80°C). The production technology for the alloys of this type includes a line of continuous casting and rolling of the wire rod, and next, its long-term heat treatment. The aim of the process of the heating of the wire rod is obtaining the desired level of the material's properties, which will make it possible to obtain the final properties of the wires assigned for the creation of conductors. The paper presents the results of the studies aiming at the description of the influence of the heat treatment of the material obtained in various production process conditions on its mechanical and electric properties.
Keywords: aluminium alloys, AlZr, conductive materials, heat treatment, electric conductivity

Nowością ostatnich lat w elektroenergetyce są antyblackoutowe przewody typu HTLS. Przewody te umożliwiają ponadprzeciętne zwiększenie zdolności przesyłu energii elektrycznej liniami napowietrznymi. Wysokotemperaturowa praca takiego przewodu, możliwa jest poprzez wykorzystanie odpornych cieplnie stopów aluminium z dodatkiem cyrkonu. Stopy te w porównaniu z tradycyjnie stosowanym materiałem w gatunku EN AW 1370 posiadają dopuszczalną temperaturę pracy na poziomie 150 do 230°C (aluminium 80°C). Technologia produkcji tego typu materiałów obejmuje linię ciągłego odlewania i walcowania walcówki, a następnie jej długoczasową obróbkę cieplną. Celem procesu wygrzewania walcówki jest osiągnięcie wymaganego poziomu własności materiału, który umożliwi uzyskanie końcowych własności drutów, przeznaczonych do budowy przewodów. W pracy przedstawiono wyniki badań zmierzających do określenia wpływu parametrów obróbki cieplnej materiału uzyskanego w rożnych warunkach procesu produkcyjnego na jego własności mechaniczne i elektryczne.

1. Introduction

The aluminium-steel conductors ACSR (Aluminium Conductor Steel Reinforced) traditionally applied in overhead lines are characterized by their permissible work temperature at the level of 80°C, which equals the existence of limits for the transmission capacity of the electroenergetic networks. A long-term excess of this temperature causes a risk of irreversible degradation of the strength properties of the aluminium wires (EN AW 1370), which have both the conductive and carrying function in the lead. That is why the work of the conductor with a impermissible current-carrying capacity results in system turn-offs of the line called the Blackout type break-down. One of the methods of increasing the energetic safety of the industrial system, with a simultaneous increase of the flow capacity of the overhead lines, is the application of high temperature, low-sag conductors of the HTLS type (High Temperature Low Sag Conductors) [1]. These conductors are composed of wires made of thermally resistant aluminium alloys and the carrying core made of low thermal expansion coefficient materials. Such selection of materials, on the one hand, enables a safe thermal work of the conductor, and on the other hand – small increases of its sag in high temperatures. The HTLS constructions characterize in a high permissible working temperature, compared to that of the traditional ACSR conductors, which remains, depending on the type of the used material, within the range of 150 – 230°C. Such high range

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of the working temperatures of the conductive material is obtained by adding zirconium to aluminium – in the quantity up to about 0.3% mass. Depending on the application, four basic thermally resistant conductive alloys have been classified, namely: AT1, AT2, AT3 and AT4 [2]. The selected properties of the wires remaining in accordance with the IEC 62004 requirements, are presented in Table 1. As can be easily seen, wires made of the thermally resistant AlZr alloys, with the exception of AT2, have their strength property level similar to the EN AW 1370 [3]. The resistivities of the AT1 and AT3 wires are higher than those of aluminium wires by only 0,5 nΩm.

TABLE 1
Parameters of thermall resistant AlZr alloy wires [2]

Wire type	AT1	AT2	AT3	AT4
Permissible continuous work temperature, °C	150	150	210	230
Resistivity at 20°C, nΩm	28.735	31.347	28.735	29.726
Conductivity, % IACS	60	55	60	58
Tensile strength R_m, MPa	159-169	225-248	159-176	159-169

2. Analysis of problem

Fig. 1 presents the influence of various alloy additions on the electrical conductivity of aluminium [4]. On the basis of the curves in Fig. 1 we can state that Zr, compared to other elements, causes a significant decrease of the electric conductivity of aluminium. Together with such heavy elements as Cr, Mn, Mo, Ti and Li, it has the most negative influence. From the analysis of the presented characteristics, it can be concluded that the AlZr alloy, with its electric conductivity at the level of 60% IACS (AT1, AT3) should not exceed 0.12% mass of the zirconium content.

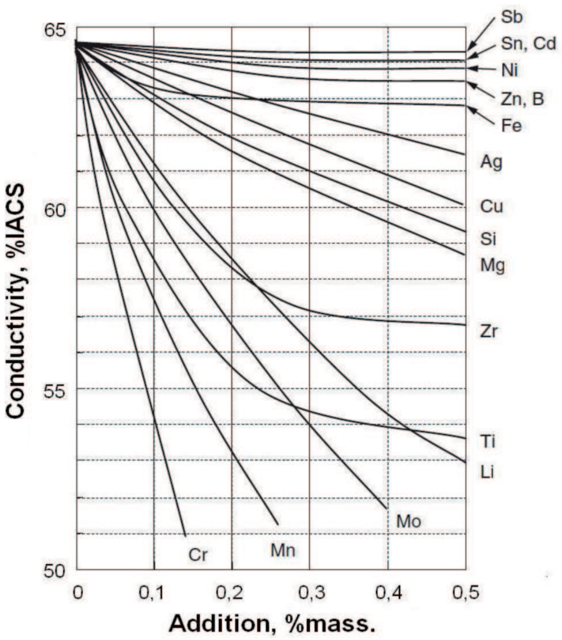

Fig. 1. Influence of the type and content of alloy additions on aluminium's electrical conductivity [4]

A deeper analysis [5, 6] of the effect of zirconium on the electrical properties of aluminium shows that the influence of Zr is dependent on its form and localization in the aluminium's structure. Table 2 includes – defined by different authors – various coefficients of the influence of the selected alloy additions to aluminium on the decrease of its resistivity, which can be applied in Matthiessen's rule [6]. This coefficient – for zirconium – is within the range from 0.4 to as much as over 45 nΩm of % mass of the element. Interesting from the point of view of the technology of the AlZr alloy production are the results of Willey's research, who divided the Zr influence into two groups. The first one is the influence of the presence of zirconium in the solid solution, which causes a significant decrease of aluminium's resistivity, and the other is its presence in the precipitates (intermetallic phase Al_3Zr). According to Willey, the Zr influence in the solid solution equals 17 nΩm/% mass. and in the precipitates – only 0.4 nΩm/% mass. This level is lower than the influence of such elements as Fe and Si [7].

On the basis of the coefficient presented in Table 2, we can describe, with the use of Matthiessen's rule, the approximate Zr quantity which should be added to aluminium in order to obtain the alloy of the desired level of electrical properties. As a result of the performed calculations and analyses, within the frames of the project: "High temperate heat resistant resisting electroenergetic conductors with the aluminium alloy basis" [8], implemented at the NPA plant in Skawina by the scientific team WMN AGH, several hundreds of chemical compositions of thermally resistant AlZr alloys have been developed.

TABLE 2
Coefficients of the influence of selected elements on the electrical properties of aluminium [5, 6]

According to author	Influence of 1% mass. on the increase of resistivity, nΩm na % mass.			
	Fe	**Si**	**Ti**	**Zr**
Aluminium Tasschenbuch	32	6,8	31	20
Van Horn	25.6	10.2	28.8	17.4
Kutner and Lang	8.5	5.16		45.4
CRC-handbook		6.7	3.14	13.5
Willey *in solid*	26	10	29	17
Willey *out of solid*	0.6	0.9	1	0.4
Harrongton R.H.	1	–	18	5

An example of the chemical composition of the alloy which is the subject of investigation in this article is presented in Table 3.

TABLE 3
Chemical composition of the thermall resistant alloy [8]

Alloying element content % mass					
Fe	**Si**	**Ti**	**V**	**Zn**	**Zr**
0.1-0.2	0.04-0.08	0.001-0.005	0.001-0.005	0.006-0.012	0.20-0.25

Figure 2 presents the double equilibrium system of AlZr, on which the alloys with the zirconium content between 0.2 and 0.25% mass have been marked.

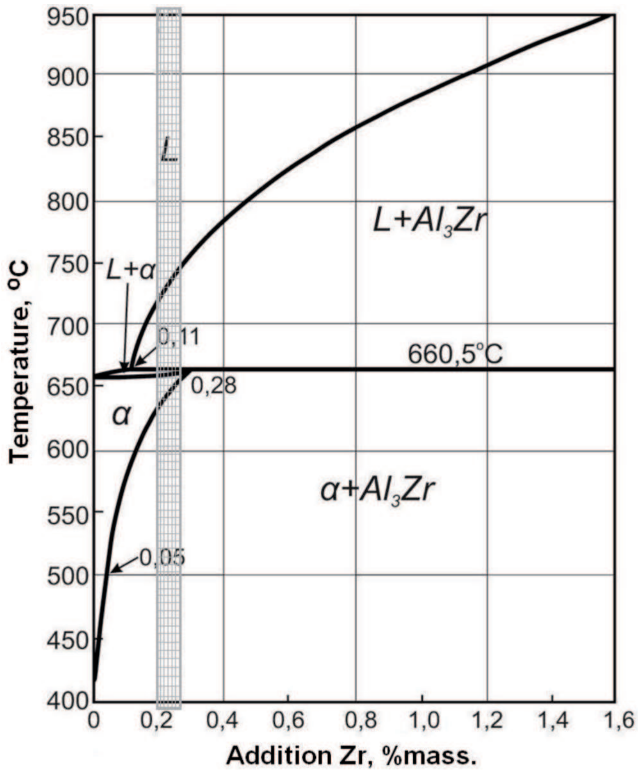

Fig. 2. Part of the equilibrium system of AlZr [11]

The liquidus line in this range of chemical composition is located at the level of about 740°C. This corresponds to the casting temperature, which, due to the presence of the area of heterogeneous liquid ($Al+Al_3Zr$), should equal at least 780°C. After exceeding the liquidus line, the primary phase Al_3Zr is starting to precipitate from the liquid, which, as a result of the perytectic transformation, still at 660.5°C, is subject to decomposition to the solution α, from which, according to the solvus line, will next precipitate the secondary, dispersive Al_3Zr phase.

The industrial production of AlZr wire rods in NPA Skawina, is implemented in the line of continuous casting and rolling (CCR) Continuus Properzi. In general, the production process consists in melting the aluminium of the appropriate purity in melting stoves, and next, after pouring the liquid aluminium into sedimentation stoves – alloying it by means of zirconium master – to the moment of obtaining the assumed chemical composition. The casting process is performed on a casting wheel of the Properzi system. The rotary crystallizer is made of a copper mould in the shape of "U" and a non-ending steel strip placed around the mould. The liquid metal is placed on the casting wheel and after crystallization is directed to the 13 stand rolling mill calibrated into the triangle-circle system. The rolling process with the complete deformation of 95% is performed within the temperature range between 550°C and about 350°C, in which the processes of deformation and dynamic recovery of the material take place. After finishing the process, the wire rod is cooled by means of spraying with water to about 80°C and made into 2 tone coils. By selection of such parameters as the casting speed, the distribution of the intensity of cooling of the casting wheel and particular stands of the rolling mill, the obtained wire rod characterizes in high strength properties, equaling from 140 to 160 MPa, and its resistivity is about 32 nΩm. Such level of properties proves, on the one hand, the occurrence of supersaturation of the alloy on the casting wheel, and on the other hand, the recovery of the material during the rolling process. In order to reduce the electrical properties of the wire rod, the latter is subjected to heating at a temperature from the range 360-420°C in the time up to 120h. An example of the changes of properties of the wire rod during the heat treatment at 400 and 450°C is presented in Table 4 and Figure 3 [9].

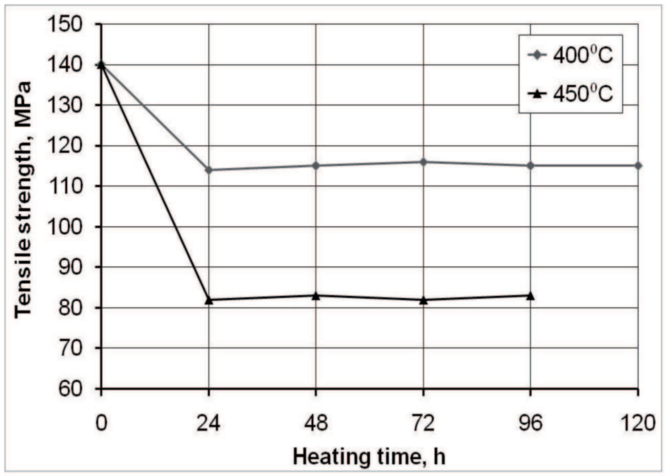

Fig. 3. Dependence of the tensile strength of the wire rod in the function of the heating time (heating temperature 400°C and 450°C; time 120h) [9]

TABLE 4
Change in properties of the wire rod after chemical composition treatment [9]

Heating time, h; temperature 400°C						
		24	**48**	**72**	**96**	**120**
R_m MPa	140	114	115	116	115	115
Heating time, h; temperature 450°C						
R_m MPa	140	82	83	82	83	–

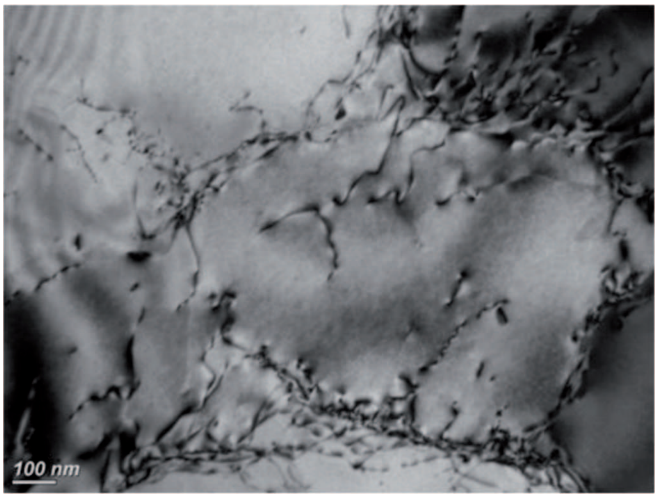

Fig. 4. View of the structural elements in the material (temper F) [10]

Beside the necessity of lowering the specific strength of the wire rod, an equally important issue is a guarantee of the appropriate level of its mechanical properties after the heating process (see Table 1 – wire properties). As can be concluded from the experimental research on the strengthening of aluminium, the minimal level of the wire rod's tensile strength after heating should not be lower than 110 MPa. This arises from the analysis of the characteristics presented in Table 4, according to which exceeding the temperature or time of heating of the wire rod causes the risk of the decrease of its tensile strength to the non-permissible level of 80 MPa, that is the soft state. From such material it is impossible to obtain wires with the required strength properties. As for the long-term heating of the wire rod at temperatures as high as 400°C, during that process we can observe a very slow precipitating of zirconium from the solid state (α) in the form of the dispersive Al_3Zr phase, which blocks the processes of recovery and recrystallization of the alloy, both during the heat treatment and the exploitation in high temperatures. Fig. 4 shows the microstructure of the wire rod on the line CCR (temper F), which, as can be easily seen, does not have visible precipitates of the Al_3Zr phase. Figure 5 presents the microstructure of the wire rod after a long-term heat treatment at a temperature at the level of 390°C, on which we can obviously notice a large number of minor Al_3Zr phase precipitates.

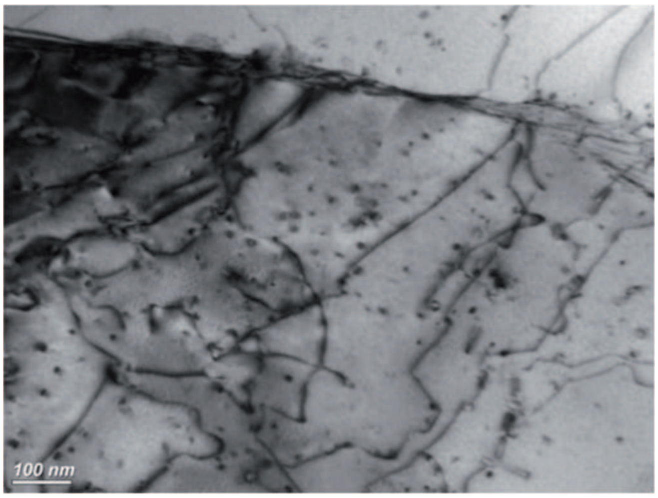

Fig. 5. View of the structural elements and precipitates in the material after heat treatment [10]

Summarizing the presented analysis of the production process of the thermally resistant AlZr wire alloys, it should be noted that the developed technology, with the basis of the CCR line Continuus Properzi, requires the restoring of many technological parameters of the casting and rolling processes, and also, it includes a long and costly thermal treatment of the material. That is why this paper aims at performing preliminary studies on the development of an alternative technology which uses the material produced in the technology of continuous casting of the ingot (CC) [12]. Such material will be subjected to heat treatment, whose only aim will be to obtain the required electrical properties of the alloy. As regards the strength properties, the basis is provided by the natural thermal resistance of the cast's structure. The objective defined in such way assumes the omission of the hot rolling segment and the raising of the cast's heat treatment temperature in such a way as to obtain only the required level of the electrical properties of the material. Due to the initial low level of the cast's mechanical properties (about 60 MPa), there is no risk of a degradation of its strength properties during the heating. This makes it possible to higher the cast's heat treatment temperature to the level of 450-550°C, which causes a significant shortening of the time of obtaining the required electrical properties of the cast. A guarantee the required level of the strength properties of the wire (Table 1) will be achieved only by selecting the appropriate level of deformation strengthening of the material in the drawing process, which corresponds to the selection of the appropriate diameter of the cast material.

3. Experimental

The results presented in the paper include the studies on the process of the selection of the heating temperature and time of the material made of the thermally resistant AlZr alloy, obtained in two different technological processes, namely the process of continuous casting and hot rolling (wire rod), and the process of continuous casting only. The material in the form of a cast was taken from the section between the casting wheel Properzi and the rolling mill. The material in the form of a wire rod was made according to the standard technology, which includes the process of hot rolling of the cast material. The chemical compositions of the examined materials are presented in Table 5.

TABLE 5
Chemical compositions of the examined materials

Examined material	**Element content, % mass.**			
	Al	**Fe**	**Si**	**Zr**
Wire rod	99.29	0.225	0.055	0.260
Cast	99.50	0.176	0.048	0.225

The material samples for the examination were prepared by means of cutting the cast and the wire rod into app. 10 mm thick slices. Thus prepared samples were subjected to heating at temperatures 385°C and 500°C. The materials were heated in a laboratory furnace in the time 24, 48, 72, 96, 120 and 144h. The thermally treated material was next subjected to examinations of hardness and electrical conductivity. The hardness measurements were performed by the Brinell method, with the use of a hardness tester with a 2.5 mm diameter steel indenter. The applied loading was 306.5 N. The electrical conductivity measurements were performed with the use of the Sigmatest device, trademark Foerster, and they were next calculated into the values of the material's resistivity.

4. Results and discussion

The obtained results of the measurements of hardness and resistivity of the examined materials after heating in the selected temperatures are presented in Table 6. Figure 6 shows the graphical forms of the resistivity changes in the function of heating time of AlZr0.2 alloy materials, at 385°C, and Figure 7 presents the hardness changes. Analogous examination results for 500°C are shown in Fig. 8 and 9.

The analysis of the initial properties of the AlZr0.2 alloys made according to different variants shows that their resistivity is at the level of about 32 nΩm, which proves that during the industrial casting process on the Properzi wheel, the solid solution was supersaturated by zirconium. In such a case, we confront the coefficient of the Zr influence on the resistivity of aluminium, which equals – according to Willey – 17 nΩm/% mass. of zirconium. Additionally, it can be noticed that the resistivity of the wire rod is about 0.9 nΩm higher than that of the cast, which proves the influence of the hot rolling process on the electrical properties of the alloy. The value of the initial hardness of the AlZr0.2 alloy after hot rolling (wire rod) equals 41 HB, and the hardness of the cast material reaches the level of 23 HB. This points to the fact that the material in the state after casting possesses at the start the properties of a material in the soft state, and the alloy on the CCR line has the properties of a strengthened material.

TABLE 6
Results of resistivity and hardness for the examined materials for the heating temperatures 385 and 500°C

Heating Time, h	**Resistivity, nΩm**				**Brinell Hardness, HB**			
	385°C		**500°C**		**385°C**		**500°C**	
	cast	wire rod	cast	wire rod	cast	wire rod	cast	wire rod
0	31.91	32.78	31.91	32.78	23.3	41.0	23.3	41.0
24	31.20	30.11	29.62	28.92	23.4	36.1	22.4	26.2
48	30.99	29.56	29.17	28.73	24.2	37.3	23.5	23.7
72	30.75	29.19	29.08	28.71	25.0	38.2	23.4	22.2
96	30.57	28.94	28.98	28.60	25.2	37.5	23.4	21.9
120	30.43	28.79	28.95	28.58	26.2	37.6	23.3	22.1
144	30.17	28.56	28.84	28.50	28.4	37.5	23.0	21.2

The heat treatment performed at 385°C in the time of 144h, in the case of the wire rod, causes a rapid drop of the material's resistivity – from 32.78 to 30.11 nΩm – which occurs already as after 24h of heating. The following changes of resistivity are relatively small, and within the range up to 144h of heating the latter reaches the level of 28.6 nΩm. As for the cast's resistivity during heating, it changes almost linearly, starting from the initial level to the value of 30.17 nΩm after the time of 144h. Also in this case, the speed of the resistivity change process is relatively slow.

The analysis of the hardness changes for materials heated at 385°C is presented in Fig. 7.

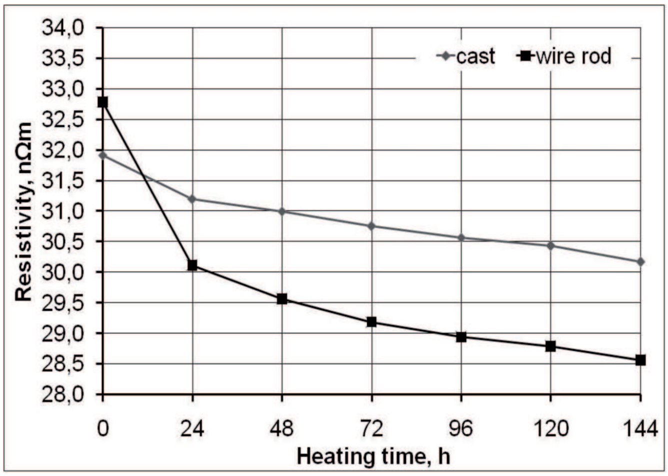

Fig. 6. Dependence of resistivity on the heating time at 385°C

The characteristics of the changes in the hardness of the wire rod is comparable to the course of tensile strength of the wire rod treated in industrial conditions (Fig. 4). Similarly to the case of resistivity changes, in the first 24h of heating, a rapid drop of hardness occurs, and next a stabilization of the material's hardness can be observed at the level of 38 HB. Such behaviour of the material should be explained by a partial elimination of the effects of the deformation strengthening, which took place during the rolling process. In the case of the cast material, a gradual increase of its hardness from 23 to 28 HB can be observed, which in combination with the resistivity change proves the occurrence of the ageing process. That is why we can expect that in a longer period of time (over 144h) an even larger increase of the material's hardness can take place.

As regards the heat treatment at 500°C, the alloy's resistivity, in the cases of both technologies for obtaining materials, drops rapidly in the first 24h of heating, and next, the decrease loses its intensity. The resistivities obtained by both materials – at the level of 28.5 nΩm - are the proof for the fact that we are observing a change of the coefficient of influence of Zr on the resistivity of aluminium to the level of 0.4 nΩm/% mass. (Willey). Thus zirconium was separated from the solution in the form of the Al_3Zr phase. From the analysis of the characteristics presented in Fig. 6 and 8 it can be concluded that after taking into account the resistivity differences of the materials, resulting from the technological conditions of their production, the resistivity level corresponds to the contents of the alloy elements.

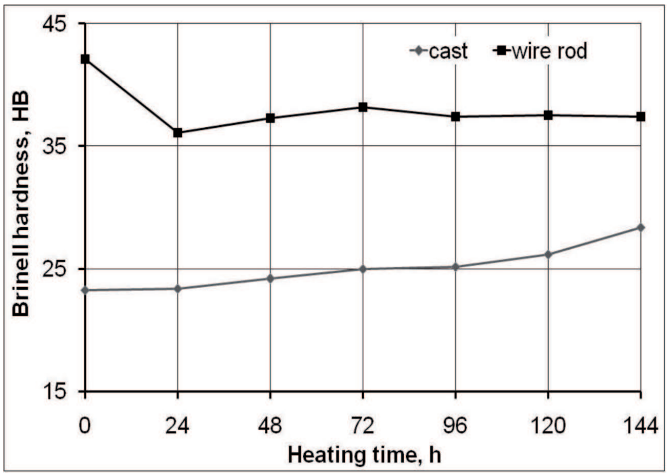

Fig. 7. Dependence of hardness on the heating time at 385°C

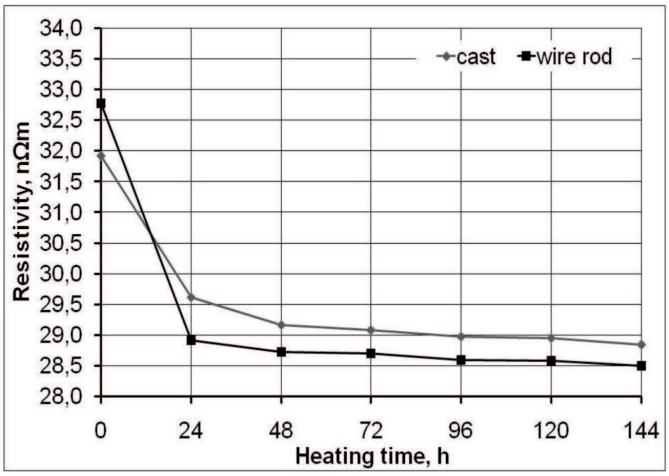

Fig. 8. Dependence of resistivity on the heating time at 500°C

The changes of the hardness of the alloy cast and rolled and the one only rolled as a result of heating at 500°C are presented in Fig. 9. We can notice that the initial hardness of the wire rod was completely eliminated during the heating process. The largest change of hardness for the wire rod took place, similarly as in the case of resistivity, in the time up to 24h of heating (from 41 to 26 HB), and next, the hardness was stabillized at the level of 21 HB. The cast material did not change the level of its hardness, retaining the value of 23 HB in the whole period of heating.

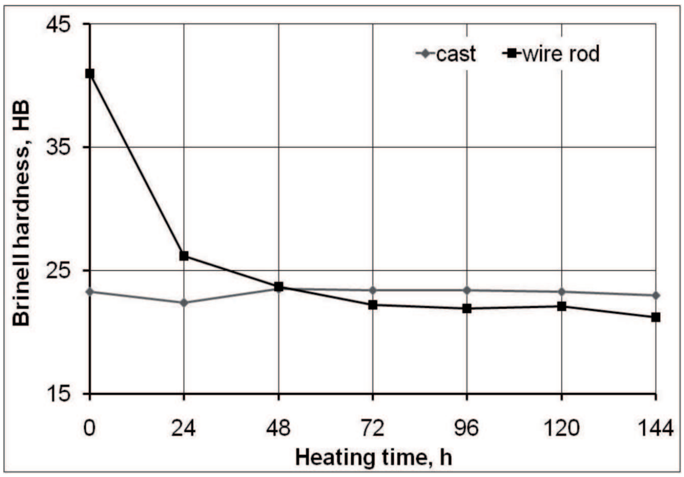

Fig. 9. Dependence of hardness on the heating time at 500°C

Summarizing the performed experiments, we should state that the heat treatment of the materials made of the AlZr0.2 alloy, obtained in various conditions of the technological process (CCR, CC), lowers the level of their resistivity from 32 to about 28.5 nΩm. Raising the temperature of the heat treatment of the alloy causes a degradation of the strength properties of the wire rod, resulting from the recrystallization processes, which makes it impossible to use it in the process of drawing the wires with the required strength properties. This results from the too little deformation strengthening during the process of drawing the 9,5 mm diameter wire rod into e.g. 3 mm diameter wires. In the case of the cast, such situation does not take place, as through the appropriate selection of its initial diameter, we can obtain the required level of deformation strengthening of the wire.

5. Conclusions

On the basis of the performed experiments on the heat treatment of the AlZr0.2 alloy, in the forms of materials after casting and hot rolling (wire rod) and after casting only (cast) obtained in the CCR line, trademark Properzi, we can draw the following conclusions:

1. During the process of casting the AlZr0.2 alloy on the casting wheel of the Properzi system, we observe the effect of supersaturation of the alloy by zirconium.
2. The heat treatment of the wire rod at 385°C causes a significant decrease of the alloy's resistivity, with a slight decrease of its hardness. This procedure requires, however, a long-term heating.
3. The heat treatment of the wire rod at 500°C is the cause for a significant decrease of both the resistivity and the hardness of the material, which takes place during the heating in the time up to 24h. However, such material is not suitable for further drawing into conductive wires because of the insufficient level of strengthening.
4. The heat treatment of the cast at 385°C causes the occurrence of the ageing effect, which manifests itself in the decrease of the resistivity and the increase of the hardness; it is, however, a long-term process.
5. The heat treatment of the cast at 500°C enables a quick decrease of the material's resistivity, without the change of the hardness level. The required level of the wires' properties after drawing will be the consequence of the appropriate selection of the cast's diameter.
6. The observed changes in the resistivity of the material during the heating point to the correctness of Willey's conception of dividing the influence of alloy element additions on the electrical properties of aluminium into two groups, that is the influence of the addition present in the solution and of that in the form of precipitates, and the proposed coefficient values correlate well with the results of the experimental studies.

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