metals

MDPI

Article

The Effect of Al-Mg-Si Wire Rod Heat Treatment on Its Electrical Conductivity and Strength

Beata Smyrak *, Bartosz Jurkiewicz, Małgorzata Zasadzińska, Marek Gniełczyk and Patryk Jałowy

Department Non-Ferrous Metals, AGH University of Science and Technology, 30-059 Krakow, Poland; bartosz.jurkiewicz@agh.edu.pl (B.J.); malgorzata.zasadzinska@agh.edu.pl (M.Z.); marek.gnielczyk@agh.edu.pl (M.G.); jalowy@agh.edu.pl (P.J.)

* Correspondence: smyrak@agh.edu.pl; Tel.: +0048-661024190

Received: 20 June 2020; Accepted: 28 July 2020; Published: 31 July 2020

check for updates

Abstract: The raw material for the production of Al-Mg-Si wires is wire rods, created in the Continuus Properzi line in temper T1 (cooled after forming at an elevated temperature and after natural aging). The general technologies for shaping the mechanical and electrical properties of Al-Mg-Si wire rods include two kinds: high- and low-temperature heat treatments. High-temperature heat treatment includes a homogenization process and a supersaturation process. Low-temperature heat treatment takes place after supersaturation and includes natural or artificial aging. This study shows how the amount of Mg and Si influences the mechanical and electrical properties of EN-AW 6101 wire rods after different kinds of heat treatments. As the general aim of this study was to determine the effect of the material's temper on its mechanical and electrical properties, the research considered the initial parameters of the starting materials being examined. These parameters can be modified by selecting the chemical composition of the Al-Mg-Si alloy and the value of precipitation hardening obtained with artificial.

Keywords: Al-Mg-Si; EN-AW 6201; EN-AW 6101; overhead conductors; AAAC (all aluminum alloy conductors); artificial aging

1. Introduction

One of the conductor types of overhead transmission lines is homogeneous conductors made of Al-Mg-Si alloy wires. All aluminum alloy conductors (AAACs) do not require a steel core, as is the case with traditional solutions (aluminum conductor steel reinforced (ACSR); see Figure 1). The advantage of using homogeneous conductors is primarily a reduction in the weight of the conductor, and there is also no risk of corrosion from steel–aluminum contact. To ensure adequate strength, 6xxx series aluminum alloys are used in the construction of such cables. Achieving high electrical conductivity and high tensile strength in the wires is possible by using a combination of heat treatment processes (supersaturation, aging) and deformation processes (drawing process).

The traditional homogenization and quenching treatments of Al-Mg-Si alloys occur at a temperature above 500 °C, and artificial aging occurs in the range of 100–200 °C. Different heat treatment technologies are used around the world. Additionally, heat treatment procedures are carried out on the raw material continuously in a continuous casting and rolling line, such as continuously heat-treated alloy. Very often, however, heat treatment procedures are conducted on wires. In the literature, most analysesb refer to heat treatment procedures for Al-Mg-Si wire, as in the series of works by Martinowa [1,2], which consider optimization problems in heat treatment procedures of Al-Mg-Si alloys, grades 6201 and 6101, for the required tensile strength and electrical conductivity of the wires. Using experiments, Martinowa aimed to find a material with the greatest possible strength of 340 MPa and resistivity of less than 32 nΩm.

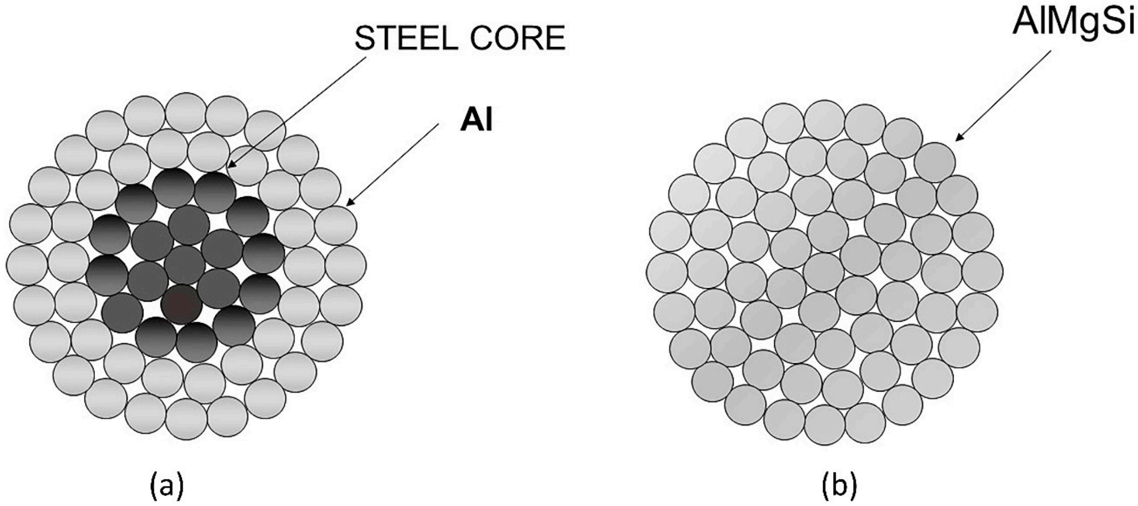

Figure 1. Aluminum conductor steel reinforced (ACSR) (**a**) and all aluminum alloy conductors (AAACs) (**b**).

Heat treatment procedures, in combination with the selection of the optimal chemical composition to achieve the highest electrical conductivity of the material, are one of the latest global trends in the development of Al-Mg-Si alloys. It is well known that transmission losses are proportional to the product of the squares of ampacity and the resistance of cables, and transmission losses depend on the geometric parameters of cables and precisely on the resistivity of the material. This issue has been the subject of extensive research worldwide. Among others, the works of Karabay [3–5] present the possibility of obtaining attractive mechanical and electrical properties in overhead conductors (AAACs), making them competitive products because of their ability to reduce transmission losses. In other studies [6,7], the authors have examined the possibility of obtaining high electrical conductivity and high tensile strength of Al-Mg-Si wires, grade 6201, in which the input material in the process of drawing was manufactured using Castex, an alternative method of continuous casting and extrusion. The authors emphasize the benefits of being able to control the structure and size of the precipitates in the Al-Mg-Si alloy through the appropriate choice of parameters in Castex technology [6,7]. In summary, the modification of the properties of Al-Mg-Si alloy wires can be divided into three areas. The first area is related to the alteration of mechanical and electrical properties with heat treatment carried out on the wires (temper T81). The second area is related to heat treatment procedures conducted on the raw material, and the third area is related to heat treatment procedures performed at the stage of casting and hot working of the raw materials. All these areas of research show the possibility of using different mechanisms of strain and precipitation hardening, depending on the state and form of the material [8–13].

The basic advantage of using Al-Mg-Si alloys is the possibility of conducting precipitation hardening, which is done in combination with deformation strengthening. Physically, the possibility of this heat treatment depends on the following factors:

- The variable limit solubility of the components in the solid-state matrix;
- A suitably low phase separation rate, enabling the supercooling of the solid solution;
- Correspondingly higher matrix hardness of the separated phase.

Depending on the heat treatment conditions (temperature and time of burning), materials have metastable phases with different morphologies and different chemical compositions. The sequence of individual phases during the aging process of Al-Mg-Si alloys has been the subject of many studies, which have proposed the following scheme for the evolution of supersaturated solution breakdown in Al-Mg-Si alloys:

$$supersaturated\ solid\ solution\ \alpha \rightarrow clusters\ (Mg,\ Si) \rightarrow GP\ zone \rightarrow pre\ \beta'' \rightarrow \beta' \rightarrow \beta(Mg_2Si),\ Si$$

All the mentioned phases, except the equilibrium phase β'', corresponding to the compound Mg_2Si, have metastable phases. The sequence of individual phases is a complex and multidimensional process that depends, first of all, on the content of the alloying elements (Mg and Si) and the state of the material, which is shaped by heat treatment (supersaturation, natural and artificial aging). The main strengthening phase in Al-Mg-Si alloys is the β'' phase, which is formed during aging at elevated temperatures [14–16].

It is well known that an increase in the content of Mg and Si in the Al-Mg-Si alloy should lead to better precipitation hardening of the material. In answer to the question on the amount of Mg and Si needed to change the mechanical and electrical properties of the raw material, we can conclude that the content of these two elements ranges from 0.3% to a maximum of 0.9%. The aim of the present research was to determine the exact amounts of Mg and Si in the Al-Mg-Si alloy, which guarantee higher strength and higher electrical conductivity of the raw material because of the drawing process [17–19].

The influence of the chemical composition of the Al-Mg-Si alloy has been the subject of many studies aiming to determine the optimal content of Mg and Si, as well as that of other accompanying additives, in order to obtain the highest level of strengthening and the highest electrical conductivity. Previous research has presented a broad research program aiming to assess the impact of Mg, Si and Fe content on the shaping of the mechanical and electrical properties of the material.

2. Materials and Methods

The raw material for this experimental research was a wire rod with a diameter of 9.5 mm in temper T1 (cooled after hot forming and natural aging), manufactured under industrial conditions in a continuous casting and rolling line (Continuus Properzi). The chemical composition of the material used in this study is shown in Table 1.

Table 1. Chemical composition of the Al-Mg-Si wire rod.

Alloy	Elements' Amount, wt. %									
	Fe	Si	Cu	Zn	Ti	V	Cr	Mn	Mg	Al
A	0.21	0.56	0.001	0.004	0.004	0.009	0.001	0.002	0.56	98.64
B	0.21	0.71	0.002	0.007	0.001	0.006	0.001	0.004	0.72	98.34

The properties of the raw material in temper T1 and after the homogenization temper, supersaturation and natural aging, are shown in Table 2.

Table 2. Properties of the Al-Mg-Si wire rod with different Mg and Si amounts.

Alloy	Amount, wt. %		Mechanical Properties			Resistivity
	Mg	Si	Yield Point, MPa	Ultimate Tensile Strength, [MPa]	Elongation A_{100}, (%)	nΩm
A	0.56	0.56	140	186	20	34.54
B	0.72	0.71	180	207	19	35.62

The experimental protocol consisted of the following three stages (see Figure 2):

1. The homogenization and supersaturation process of Al-Mg-Si wire rods with different amounts of Mg and Si;

2. The artificial aging of Al-Mg-Si wire rods with different amounts of Mg and Si;
3. Testing of the mechanical and electrical properties of the wire rods.

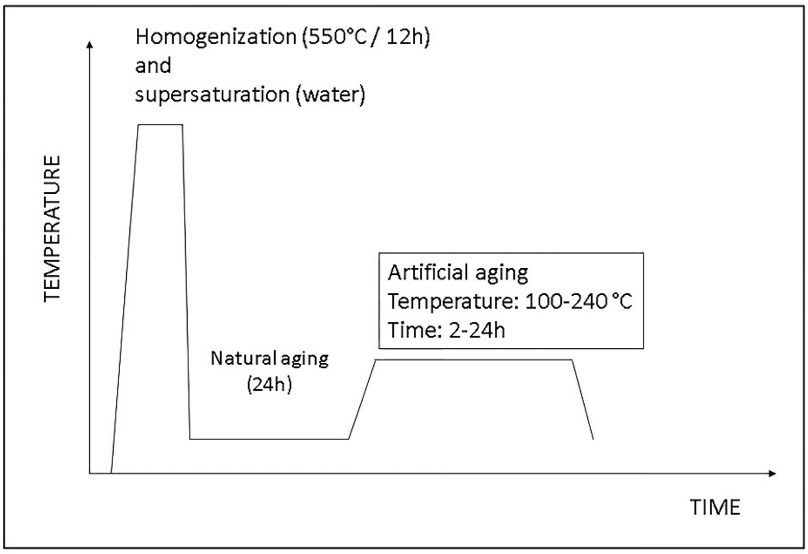

Figure 2. Scheme of the heat treatment of the wire rods.

In Table 3, the detailed parameters (time and temperature) of the homogenization and artificial aging of the materials are shown.

Table 3. Parameters of homogenization, supersaturation and artificial aging.

Process	Temperature/Time
Homogenization, °C/h	550/12
Supersaturation, °C/h	550/12–water
Natural aging, °C/h	20/24
Artificial aging temperature, °C	100, 120, 140, 150, 160, 170, 180, 190, 200, 220, 240
Artificial aging time, h	2, 4, 8, 12, 16, 20, 24

After various heat treatment variants, the wire rods were subjected to tests of their mechanical properties (tensile test) and electrical conductivity (bridge method). The tests of mechanical properties were carried out using the uniaxial tensile test. The mechanical properties of the samples were measured on a Zwick/Roell testing machine (ZwickRoell Ulm, Headquarter, Germany), with a maximum force range of 20 kN. The tests of electric properties were performed using the Thomson bridge. The maximum measuring resolution was 1 nΩ, with measuring currents from 100 μ to 10 A. The accuracy class was 0.01%. The detailed test parameters are presented in Table 4.

Table 4. Parameters of the mechanical and electrical property tests.

Test	Parameters
Tensile test	Measuring base: 100 mm, speed: 50 m/min
Electrical conductivity test	Measuring base: 1000 mm, temperature coefficient: 4000 1/K

Microstructure tests were done using a scanning electron microscope, model Hitachi S-3500N (Tokyo, Japan). This device allows studying the surface morphology of solids at the micro and nanoscales and has a resolution of 2.5 nm at 25 kV and a maximum magnification of ×300,000. It was additionally equipped with an EDS (Energy-dispersive X-ray spectroscopy) x-ray microanalyzer, model Noran 986B-1SPS (Shenzhen, China).

3. Results

Figure 3 presents the selected true stress–true strain characteristics of the Al-Mg-Si wire rod with different contents of Mg and Si after different kinds of heat treatments. Figure 3a: after homogenization (heating at 550 °C and equilibrium cooling), Figure 3b: after supersaturation (heating at 550 °C and water cooling), Figure 3c: natural aging and Figure 3d: after artificial aging.

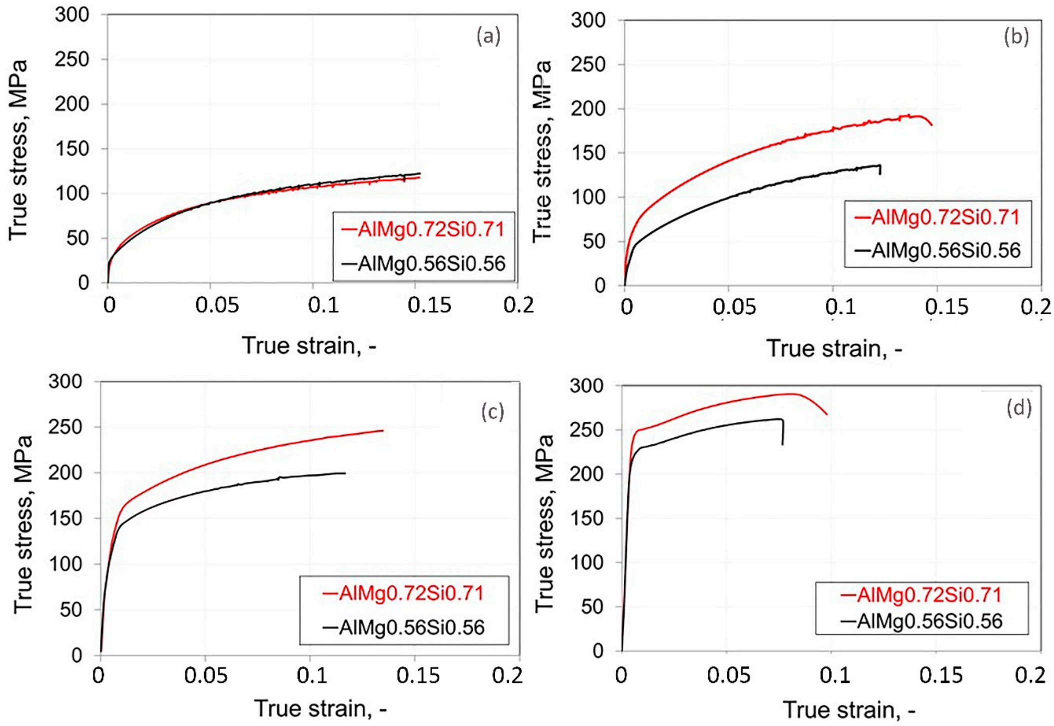

Figure 3. True stress and true strain curves of Al-Mg-Si wire rods with different amounts of Mg and Si after homogenization (**a**), supersaturation (**b**), natural aging (**c**) and artificial aging (**d**).

Figures 4 and 5 present the microstructure results of the wire rods after homogenization.

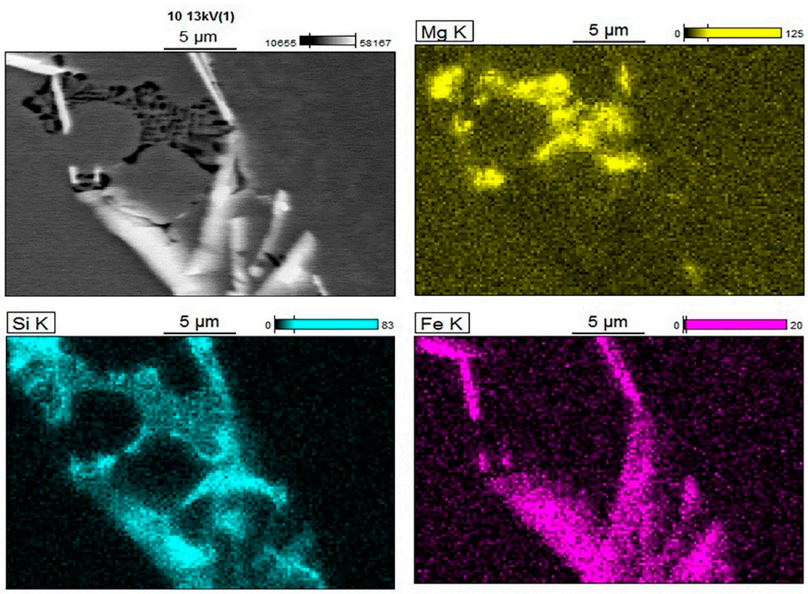

Figure 4. EDS analysis of the AlMg0.72Si0.71 wire rods after homogenization.

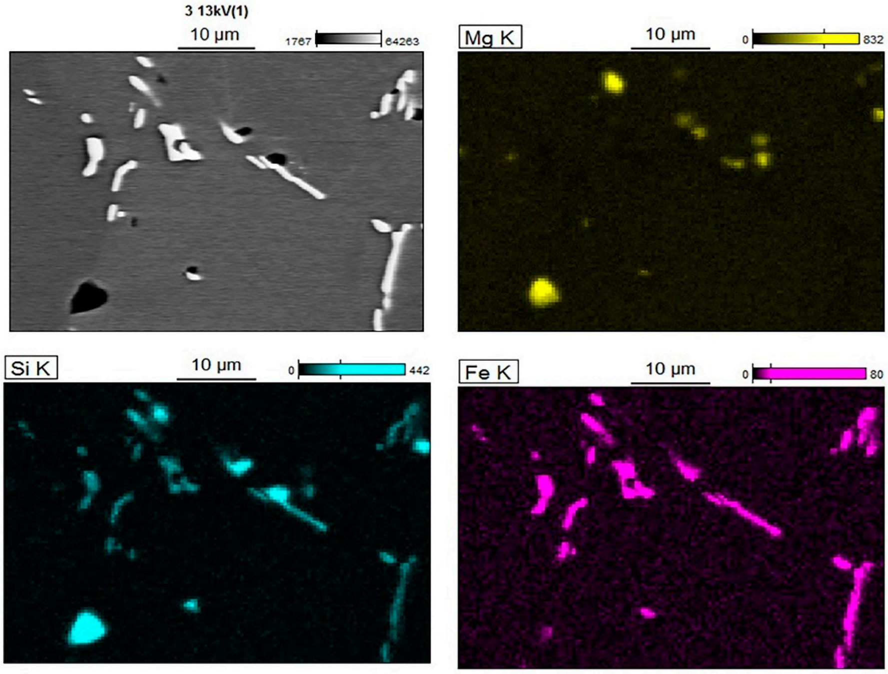

Figure 5. EDS analysis of the AlMg0.56Si0.56 wire rods after homogenization.

The analysis of the samples after the homogenization process shows that in the state closest to the equilibrium state, two types of phases dominate. In the case of AlMg0.72Si0.71, these are the phases Mg-Si and Fe-Si, whereas, in the case of AlMg0.56Si0.56, these are Mg-Si-Fe (dark precipitations) and Fe-Si (white precipitations). This difference is due to the presence of approximately 0.2 wt. % of iron in both materials.

The influence of the time and temperature of artificial aging on the change in the mechanical and electrical properties of the wire rods produced from the alloys for AlMg0.56Si0.56 and AlMg0.72Si0.71 is shown in Figures 6 and 7.

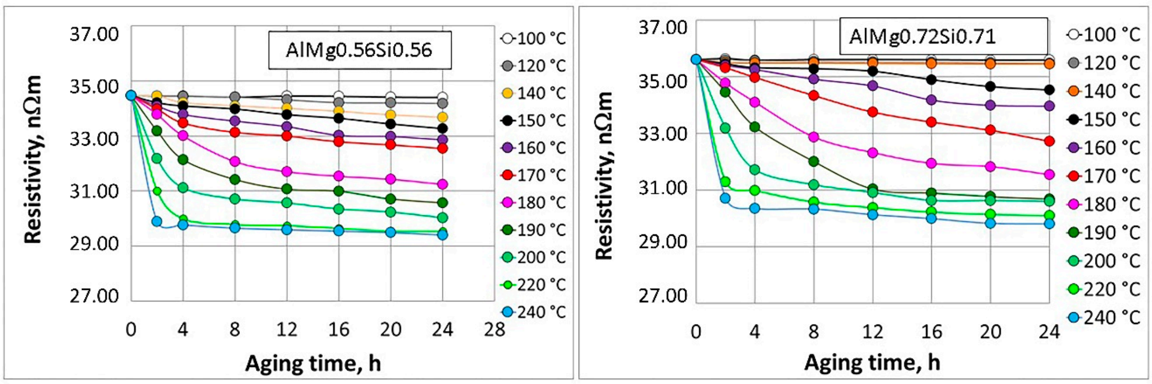

Figure 6. Relationship between resistivity and artificial aging time at different temperatures for the AlMg0.56Si0.56 and AlMg0.72Si0.71 wire rods.

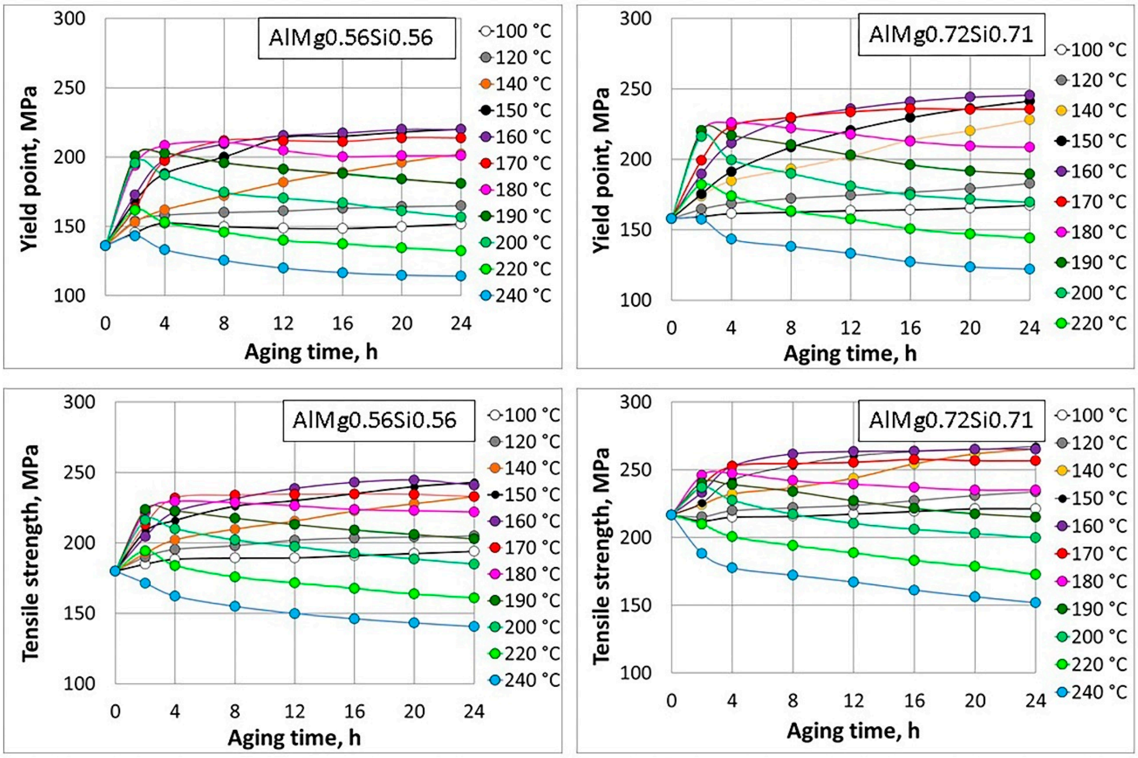

Figure 7. Relationship between strength and artificial aging time at different temperatures for the AlMg0.56Si0.56 and AlMg0.72Si0.71 wire rods.

Based on the above characteristics of the changes in tensile and yield strength, as well as the changes in the resistivity of the wire rods in T1 state, the relationship of resistivity and mechanical properties was developed. The charts were based on the spatial data presented in Figures 6 and 7. The points on the graph shown in Figure 8 represent the values of resistivity and yield strength for each specimen after artificial aging and the values of resistivity and tensile strength for each specimen after artificial aging. Points located in the area of high resistivity represent the effects of artificial aging at a lower temperature range up to approximately 150 °C. Conversely, points located in the area of low resistivity represent the effects of artificial aging at higher temperatures (above 150 °C).

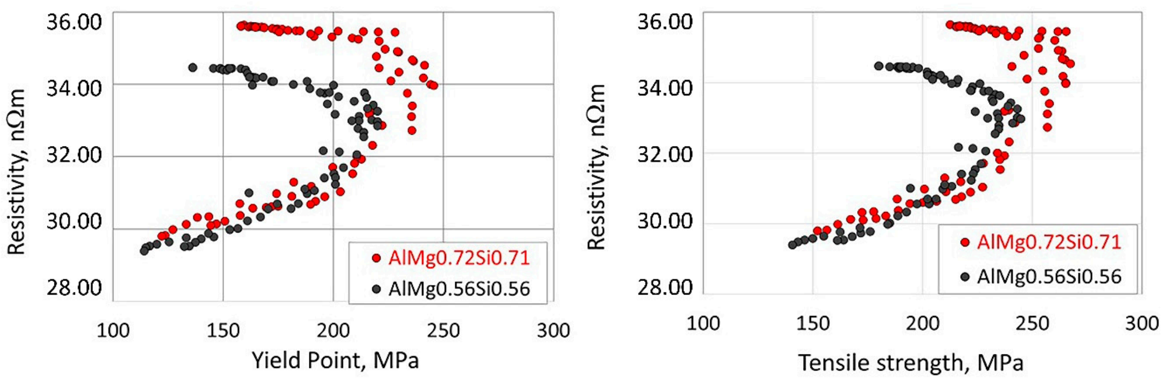

Figure 8. Relationship between the yield point and resistivity and between the tensile strength and resistivity of Al-Mg-Si wire rods after artificial aging (black points: AlMg0.56Si0.56, red dots: AlMg0.72Si0.71).

Figure 8 presents a comparative analysis of the influence of chemical composition on the effects of artificial aging. It should be noted that the essential difference between the effects of artificial aging of raw materials containing different amounts of Mg and Si is reflected in the values of resistivity.

Compared with the red dots (AlMg0.72Si0.71), the black dots on the graph (AlMg0.56Si0.56) are located lower towards lower values of resistivity.

4. Discussion

The graphs shown in Figure 3 indicate that in the case of the post-homogenization material, regardless of the content of Mg and Si, the mechanical properties were similar in both types. The conventional yield point was approximately 25 MPa, the tensile strength was approximately 100 MPa and the total relative elongation was 25% (true total elongation: −0.15). The increase in Mg and Si from 0.56% to 0.72% in the case of the post-homogenization material was practically unnoticeable. Thus, differences in the content of Mg and Si at the level of approximately 0.3% in the material, in which all the alloying elements (Mg, Si and Fe) were dissolved and brought to equilibrium separation by slow cooling, were not noticeable in the tension test. On the other hand, if the material was subject to supersaturation after homogenization, differences in the content of Mg and Si were reflected in the mechanical properties (see Figure 3).

The tensile characteristics of the material after supersaturation, as shown in Figure 3b, indicate that an increase in Mg and Si to approximately 0.72% led to a significant increase in the strengthening of the material. If, at a later stage, the material in the state after supersaturation is subject to natural aging, then the effect of the varying contents of Mg and Si on the mechanical properties is evidentially demonstrated. The increase in Mg and Si in the material after natural and artificial aging clearly leads to a significant increase in the yield and tensile strength (see Figure 3c,d).

These results are in accordance with previous data on the influence of the amount of Mg and Si (i.e., amount of the Mg_2Si phase) and with data on Si excess in relation to the amount required to form the compound Mg_2Si. Differentiation of the content of Mg and Si at the level of approximately 0.3% leads to diversity in material strengthening at a level of as much as 50 MPa, even in materials subject to natural aging (cf. Figure 3c). On the other hand, a variation in the resistivity of the material subject to homogenization was observed after analyzing the effect of Mg and Si content on the electrical properties of materials following various heat treatment procedures (Figure 6).

This finding seems obvious, as higher total contents of alloying elements by approximately 0.6% (0.3% Mg and 0.3% Si) must affect the increase in the material's resistivity. Data on the impact of the number of alloying elements on the resistivity of aluminum should also be considered here. Based on the literature, it can be assumed that the addition of 1 wt. % of Mg leads to an increase in resistivity by approximately 5–7 nΩm/wt % In the case of Si, the increase in resistivity is higher and ranges, according to different authors, from 0.8 to as much as 10 nΩm/1 wt. % of the element. These data refer to elements dissolved in the solution and elements in the form of precipitations.

The reason for the higher resistivity of the material may be the presence of excess Si content in the solution. It is well known that an excess of Si, as per the stoichometry-required compound Mg_2Si, is involved in Al-Fe-Si precipitates. On the other hand, the increase in the content of Si and Mg in the alloy, with the Fe content being at the same level, leads to a situation in which excess Si must remain in the solution.

A detailed analysis of the nature of strengthening required developing the parameters of Hollomon's equation based on the characteristics

$$\sigma_T = K\varepsilon_T^n \tag{1}$$

where σ_T is the true stress, ε_T is the true strain, n is the strain-hardening exponent and K is the strength coefficient. In Hollomon's model, the strain-hardening exponent measures the ability of a metal to strain-harden; larger magnitudes indicate larger degrees of strain hardening. For most metals, the strain-hardening exponent ranges from 0.10 to 0.40. The values of the linear factor K and the power factor n for our samples are presented in Table 5.

Table 5. Values of the *K* and *n* coefficients according to Hollomon's equation for Al-Mg-Si wire rods after different types of heat treatments.

Temper	AlMg0.56Si0.56		AlMg0.72.Si0.71	
	K	*n*	*K*	*n*
After homogenization	208	0.291	238	0.338
After supersaturation	303	0.374	372	0.326
After natural aging	279	0.148	358	0.182
After artificial aging: 140 °C/10 h	322	0.121	381	0.128
After artificial aging: 160 °C/10 h	311	0.083	348	0.087
After artificial aging: 240 °C/10 h	190	0.081	205	0.082

We note that the highest values of the *n* factor are observed for the material after and supersaturation. After artificial aging, the values of *n* are almost 10 times lower. In turn, the analysis of the impact of artificial aging parameters clearly showed that the increase in temperature and time of artificial aging led to a decrease in both the *K* and *n* factors from 0.13 to 0.08. A lower value of the coefficient *n* samples after natural and artificial aging is associated with the precipitation curing mechanism, which means that the material has dispersive precipitates. The variation of the coefficient *n* is a monotonic function of time and temperature. In the analysis of materials after aging, the highest value of the coefficient *n* is obtained by wire drawing after natural aging, and the lowest value for the material occurs in its obsolete state.

The content of Mg and Si has a greater effect on the value of the linear factor *K*. In turn, the value of the factor *n* has a greater impact on the type of heat treatment.

5. Conclusions

Based on our findings, the following conclusions are made:

1. The same level of ultimate tensile strength, yield point and resistivity of wire rods can be obtained under different temperatures and aging times.
2. A difference in Mg and Si content at the level of 0.15 wt. % results in an increase in the tensile strength by about 25 MPa and a resistivity differentiation of about 1 nΩm.
3. The increased presence of Mg and Si in the alloy results in a better effect of artificial aging, which means higher strength and higher electrical conductivity.
4. Restricting the amount of Si in the alloy is important because excess Si results in the decreased electrical conductivity of the wire rods. Therefore, the optimum amount of Si depends on the amount of Mg necessary to create Mg_2Si.
5. The optimum parameters in terms of temperature and time for the artificial aging of Al-Mg-Si wire rods are 150 °C and 4–10 h. These parameters result in a tensile strength of 200 MPa and an electrical resistivity of 30 nΩm. These properties are ideal for the drawing process to create wires with high strength and high electrical conductivity.
6. The content of Mg and Si has a greater effect on the value of the linear factor *K*, whereas the value of the hardening factor *n* has a greater impact on the type of heat treatment.

Author Contributions: Writing—original draft preparation: B.S.; editing: M.Z.; writing—review and editing, P.J.; and investigation: B.S., M.Z., M.G., P.J. and B.J. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Conflicts of Interest: The authors declare no conflict of interest.

References

1. Martinova, Z.; Zlateva, G. Microstructure development during thermomechanical treatment of AlMgSi alloy. *J. Min. Metall. Sect. B.* **2002**, *38*, 133–162. [CrossRef]
2. Martinova, Z.; Damagaliev, D.; Hirsh, M. The effect of room temperature pre-aging on tensile and electrical properties of thermomechanical treated AlMgSi alloy. *J. Min. Metall. Sect. B.* **2002**, *38*, 61–73. [CrossRef]
3. Karabay, S.; Güven, E.A.; Ertürk, A.T. Enhancement on Al–Mg–Si alloys against failure due to lightning arc occurred in energy transmission lines. *Eng. Fail. Anal.* **2013**, *31*, 153–160. [CrossRef]
4. Karabay, S. Influence of AlB2 compound on elimination of incoherent precipitation in artificial aging of wires drawn from redraw rod extruded from billets cast of alloy AA-6101 by vertical direct chill casting. *Mater. Des.* **2008**, *29*, 1364–1375. [CrossRef]
5. Karabay, S. Modification of AA-6201 alloy for manufacturing of high conductivity and extra high conductivity wires with property of high tensile stress after artificial aging heat treatment for all-aluminium alloy conductors. *Mater. Des.* **2006**, *27*, 821–832. [CrossRef]
6. Zhou, T.; Jiang, Z.; Wen, J.; Tieu, A.K. On-line aging to improve the tensile strength and conductivity of cold drawing AA6201 wires. *Mater. Sci. Forum* **2007**, *561–565*, 349–352. [CrossRef]
7. Zhou, T.; Jiang, Z.; Wen, J.; Li, H.; Tieu, A.K. Semi-solid continuous casting-extrusion of AA6201 feed rods. *Mater. Sci. Eng. A* **2008**, *458*, 108–114. [CrossRef]
8. Zen, L.; Kang, S.B. The effect of pre-aging on microstructure and tensile properties of AlMgSi alloys. *Scr. Mater.* **1997**, *36*, 1089–1094. [CrossRef]
9. Friis, J.; Holmedal, B.; Ryen, Q.; Nes, E.; Myhr, O.R.; Grong, Q.; Furu, T.; Marthinsen, K. Work hardening behavior of heat treatable AlMgSi alloys. *Mater. Sci. Forum* **2006**, *519–521*, 1901–1906. [CrossRef]
10. Raeisinia, B.; Poole, W.J.; Lloyd, D.J. Examination of precipitation in the aluminium alloy AA6111 using electrical resistivity measurments. *Mater. Sci. Eng. A* **2006**, *420*, 245–249. [CrossRef]
11. Jensrud, O.; Pedersen, K. Cold forging of high strength aluminum alloys and the development of new thermomechanical processing. *J. Mater. Process. Technol.* **1998**, *80–81*, 156–160. [CrossRef]
12. Lacom, W.; Spiradek, K. Assessment of thermal-treatment conditions for age-hardenable particulate-reinforced aluminium alloys by calorimetric methods. *Thermochim. Acta* **1998**, *310*, 125–132. [CrossRef]
13. McQueen, H.J.; Xia, X.; Cui, Y.; Li, B.; Meng, Q. Solution and precipitation effects on hot workability of 6201 alloy. *Mater. Sci. Eng. A* **2001**, *319–321*, 420–424. [CrossRef]
14. Dumitraschkewitz, P.; Uggowitzer, P.J.; Gerstl, S.S.A.; Löffler, J.F.; Pogatscher, S. Size-dependent diffusion controls natural aging in aluminium alloys. *Nat. Commun.* **2019**, *10*, 4746. [CrossRef] [PubMed]
15. Cao, L.; Rometsch, P.A.; Zhong, H.; Mulji, Y.; Gao, S.X.; Muddle, C. Effect of natural ageing on the artificial ageing response of an AlMgSiCu alloy. In Proceedings of the 12th International Conference on Aluminium Alloys, Yokohama, Japan, 5–9 September 2010; pp. 395–399.
16. Nakamura, J.; Matsuda, K.; Kawabata, T.; Sato, T.; Nakamura, Y.; Ikeno, S. Effect of silver addition on the b′ phase in AlMgSi Alloy. *Mater. Trans.* **2010**, *51*, 310–316. [CrossRef]
17. Bruggemean, T.; Bollman, C.; Mohles, V.; Gottstein, G.; Hirsh, J.; Myhr, O.R. Yield stress development during age -hardening of AA6xxx: Comparison of two model predictions with experimental findings. In Proceedings of the 12th International Conference on Aluminium Alloys, Yokohama, Japan, 5–9 September 2010; pp. 296–299.
18. Huynh, K. Effects of the heat treatment on the mechanical properties of 6201 aluminium alloy wire. *Vietnam J. Sci. Technol.* **2019**, *57*, 11–20. [CrossRef]
19. Bunte, C.; Glassel, M. Proposed Solution for Random Characteristics of Aluminium Alloy Wire Rods Due to the Natural Aging. *Procedia Mater. Sci.* **2015**, *9*, 97–104. [CrossRef]