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THE EFFECT OF ROOM TEMPERATURE PRE-AGEING ON TENSILE AND ELECTRICAL PROPERTIES OF THERMOMECHANICALLY TREATED Al-Mg-Si ALLOY

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Abstract

A commercial Al - 0.62%Mg – 0.57%Si was thermomecanically treated (TMT). The TMT process included solution treatment, room temperature preageing, drawing (e=95%) and final ageing. The experimental data were proceeded statistically and mathematical models were derived for the alloy properties such as tensile strength, electrical conductivity and elongation of the wires during TMT. The models are used to find out the area of compromise optimal combination of the alloy properties. Higher final ageing temperature and time are required to design a TMT process for production of a long-term pre-aged wires. The influence of the room temperature preageing on the precipitation process during TMT is discused.

Keywords: alloy, thermomecanical treatment, natural ageing, pre-ageing.

1. Introduction

Al-Mg-Si alloys used for electrical application, mainly as conductors are required to possess a proper combination of properties, such as high strength, high electrical conductivity and optimum elongation. Thermomechanical treatment (TMT) T8 is widely used to satisfy these requirements. In view of the well-known interaction belween natural (NA) and artificial (AA) ageing in Al-Mg-Si alloys, the storing time at room temperature is one of the most important variables concerning material properties, obtained after it. For the best results, it is recommended that the plastic strain is introduced immediately after solution treatment and quenching operations followed by a process of artificial ageing [1-2].

The commercial significance of the effects of NA in Al-Mg-Si alloys has motivated several studies on thermomechanical treatments with room-temperature pre-ageing [3-8]. Most of them employed a medium degree of deformation [3-5] and focused on the changes of tensile properties or hardness. Latkowski and Bronicki [4] have found that the plastic deformation (e = 10-25 %) between NA (24 hrs) and artificial ageing (180°C) of extruded bars could prevent the negative influence of natural ageing. However, in naturally aged Al-l%Mg_2Si-0,4°/oSi sheets the 10% cold reduction prior to artificial aging(AA) leads to double reduction in elongation [5].

In an earlier work Benedyk [6] has observed a beneficial effect of TMT with room-temperature pre-ageing for the Al-0,75 and 1,5% Mg_2Si alloys. Ber [7] has reported an improvement of tensile strength and ductility of an A1-1.15% Mg_2Si-0,14%Si (Russian grade AD31) after TMT, when an 90% cold deformation has been performed between NA (24 hrs) and AA (155°C). Ghosh [8] has included 20 days NA in thermomechanical processing of an Al-0,4Mg-l,3 Si alloy, containing Cr and Ti. He found out that wires meet the requirements for high-strength conductor application with 323 [N/mm^2], electrical conductivity 53,2% 1ACS and 6% elongation, when after NA 80% cold work and 175 °C was used.

The main object of this paper is to present a study of the mutual influence of several variables (room temperature pre-ageing time, artificial ageing conditions) on the mechanical and electrical properties of thermomechanically treated Al-0,62%Mg-0.57%Si (6201) alloy and to find out the area of their optimal values.

2. Experimental procedure

Commercial Al-Mg-Si alloy was received in the form of a continuous produced 9,5 [mm] diameter rod. The chemical composition is shown in Table 1.

Table 1. Chemical composition of the alloy (mass %)

Mg	Si	Fe	Cu	B	ΣCr+Ti+V+Mn	Al
0.6175	0.5644	0.2300	0.0517	0,0138	0.019	rest

The specimens cut from a coil were solution treated at 520°C for 4 hrs, followed by water quenching. Fig. 1 schematically shows the applied thermo-mechanical sequence. The quenched materials were stored at room temperature (natural ageing) for different periods ranging from 4 hours to 2000 hours. Wires from the samples were produced by drawing (ε = 95%) on one pass drawing block. To prevent a significant temperature rise in the wire, oil was applied before and after each pass. The artificial ageing was held for 30 min to 8 hours at temperatures 130, 150, 170 °C. Artificial ageing treatments were done in an oil bath for the short holding times and in an electrical air oven for the prolonged ones.

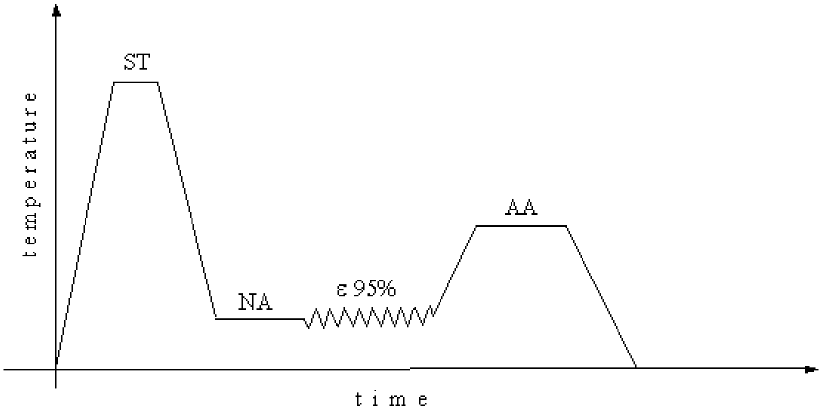

Fig. 1. Scheme of thermomechanical sequence

Specific electrical resistivity measurements were made at 20°C using a standard Double Thomson Bridge with a *one meter* length of the samples. The results are reported here as conductivity. Standard tensile testing instrument was utilised to determine the ultimate tensile strength (Rm) and elongation (A%) on every step of TMT.

3. Results

Properties of preliminary naturally aged alloy indicate the effective work hardening as depicted in Table 2. The samples stored for more than 4 hours after quenching exhibit higher ultimate tensile strength, lower electrical conductivity and elongation. The increment of strengthening due to deformation is higher than the one due to the NA, but it diminishes with the pre-ageing storing (NA) time.

Table 2. Mechanical and electrical properties of as drawn wires ε = 95%

NA pre-agcing time, [hours]	Ultimate tensile strength, [N/mm^2]	Electrical conductivity, [mΩ^{-1}mm^2]
0	327	28.18
4	357	27.99
8	387	27.52
24	385	27.13
72	390	27.02
94	399	27.75
500	387	27.51
700	387	27.39
1000	393	27.19

To establish how the pre-ageing and final ageing conditions of therinomechanical treatment affect properties data obtained by tensile tests and electrical measurements performed on the deformed material were collected and proceeded using a MINITAB statistical software. A mathematical model of general type $Y=b_o+b_1\tau+b_2\tau^2+b_3\tau^3$ was derived for each property, were τ is

AA-time and b_i (i=0,1,2,3) are coefficients. All models were statistically analysed. They were used for graphical predicament of the properties.

The data are also used to work out second order polynomial models of the general type:

$$y(\vec{x}) = b_o + \sum_{i=1}^{3} b_i x_i + \sum_{i=1}^{2} \sum_{j=1+i}^{3} b_{ij} x_i x_j + \sum_{i=1}^{3} b_{ii} x_i^2 \qquad (1)$$

where x_1 is the NA, x_2 is the AA and x_3 is the temperature.

The values of the model coefficients for the different alloy properties investigated in this paper are shown in Table 3.

Table 3. Model coefficients of the alloy properties.

Coefficient	Coefficient's values		
	Rm	γ	A
b_0	-307.00	37.549	-41.17
b_1	-	0.0046138	0.0041363
b_2	29.543	-1.09799	2.6708
b_3	9.4737	-0.14372	0.5802
b_{12}	-	-0.00017797	0.00031701
b_{13}	0.00026505	-0.00003707	-
b_{23}	-0.20463	0.0100372	-0.0141
b_{11}	-0.00003719	0.00000081	-0.00000495
b_{22}	-0.24765	-0.008899	-0.08348
b_{33}	-0.031879	0.0005198	-0.0017936
Determination coefficient R^2	86.9%	94.0%	61.4%
Calculated F value for significance check of R^2	175.05	333.24	35.77

Some coefficients are missing as they are considered insignificant according to the statistical criteria.

The calculated values of the Determination coefficient R^2 and F value for significance test of R^2 gives reason to conclude that the models are good enough. They are used for the graphical interpretation of the alloy properties and conclusion about the factor space area of their compromise optimal values.

A great number of graphs have been made based on the models. Fig. 2 and Fig. 3 illustrate the artificial ageing curves at the temperatures 130, 150 and 170°C for the samples, held at room temperature before deformation – 24h (short term NA) and 500 hours (long term NA). It can be seen that for both of the shown pre-ageing times the conductivity increases during final ageing, which indicates that a precipitation reaction occurres. At the given room temperature pre-ageing, cold reduction ratio and final ageing time, the wires

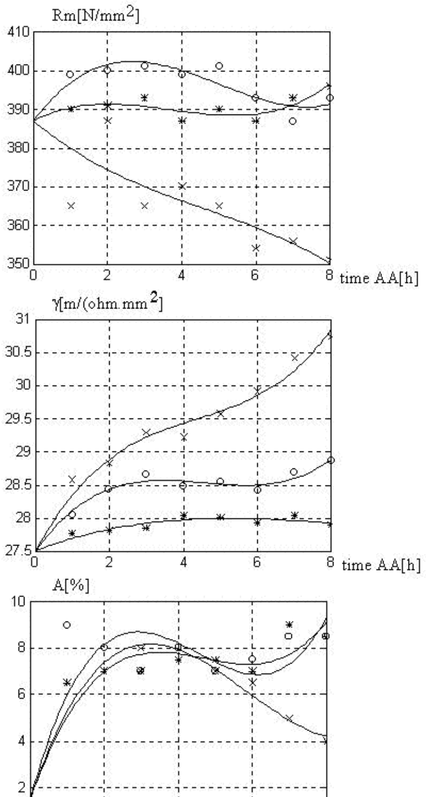

Fig. 3. Arteficial ageing curves at the temperatures 130,150 and 170°C (500h NA, ε = 95%)

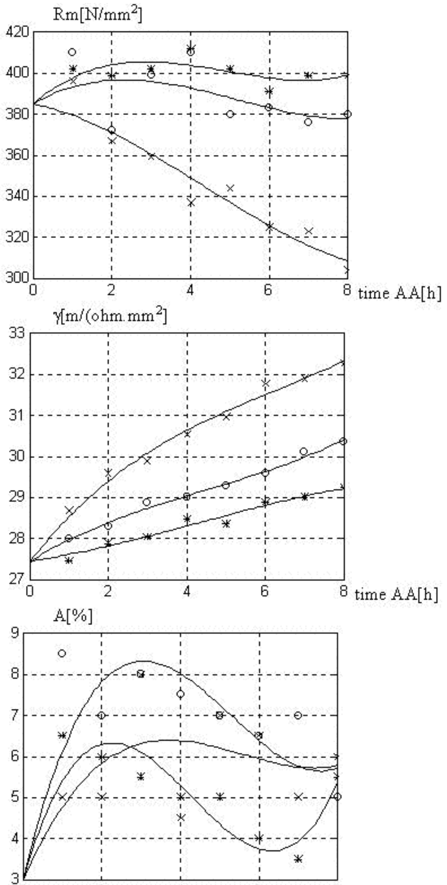

Fig. 2. Arteficial ageing curves at the temperatures 130,150 and 170°C (24h NA, ε = 95%)

give higher conductivity when a higher ageing temperature is employed. The similar ageing kinetics was found for each of the investigated thermomechanical regimes. The ageing response of the deformed alloys leads to an increment in tensile strength at 130-150°C; generally the ageing kinetics curves show that the ageing is inverse proportional to of NA time and final ageing temperatures. No increment is observed by ageing at 170°C where softening of the deformed matrix predominates the strengthening by precipitation. Sharp increase in elongation is observed in the first hour of artificial ageing; it reaches maximum and decreases as the ageing proceeds. The same was reported by Ber [7].

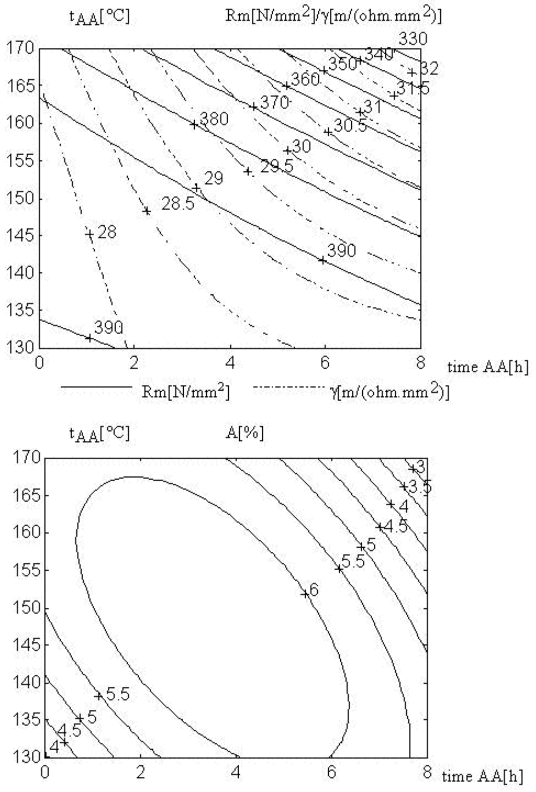

Fig. 4. Isolines of Rm, γ and A for ST-NT 4h - ε 95% - AA

The effect of final ageing conditions on the tensile and electrical properties obtained by TMT are more obviously revealed in the diagrams, depicted in Fig. 4-6. Here, based on the models, isolines for each property are depicted as function of time and temperature of AA. The figures were chosen to demonstrate three representative intervals of natural ageing (delay time between quenching and deformation): 4, 94 and 500 hours. It can be seen that property levels are considerably affected by the final ageing and almost in the

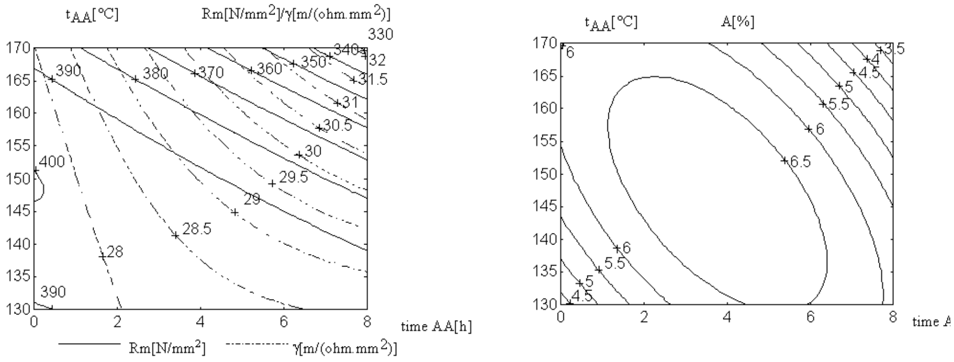

Fig. 5. Isolines of Rm, γ and A for ST-NT 94h - ε 95% - AA

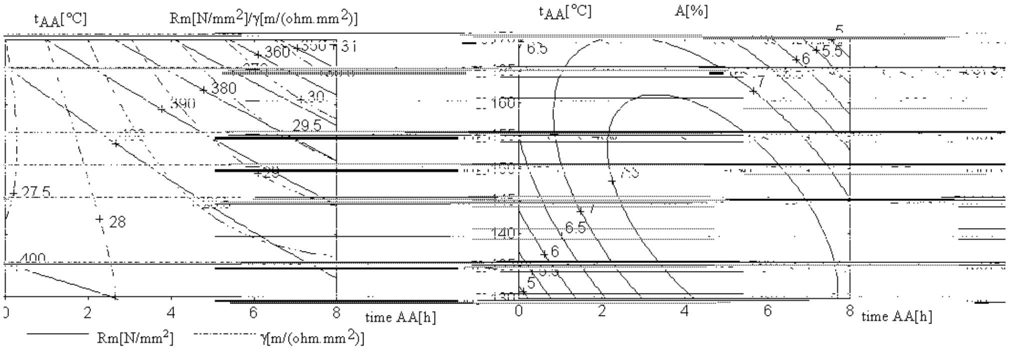

Fig. 6. Isolines of Rm, γ and A for ST-NT 500h - ε 95% - AA

same manner, independent of the delay time. At a given time of AA higher electrical conductivity and lower strength are obtained at higher ageing temperatures, maximum in elongation appears after 3.5-5 hours. Similar to the most precipitation hardened alloys maximae in electrical conductivity, tensile strength and elongation are obtained at different time-temperature regimes. It is not unexpected in the experiments, considering the different structural parameters the properties depend on. The required combination for EC 6201 alloy of Rm ^{3} 320 N/mm^2 and electrical conductivity ^{3} 30.45 [mΩ^{-1} mm^{-2}] (≥52,5%IACS) apparently could be obtained by heat treatment in a compromised time/temperature area. The data for the samples, submitted to a

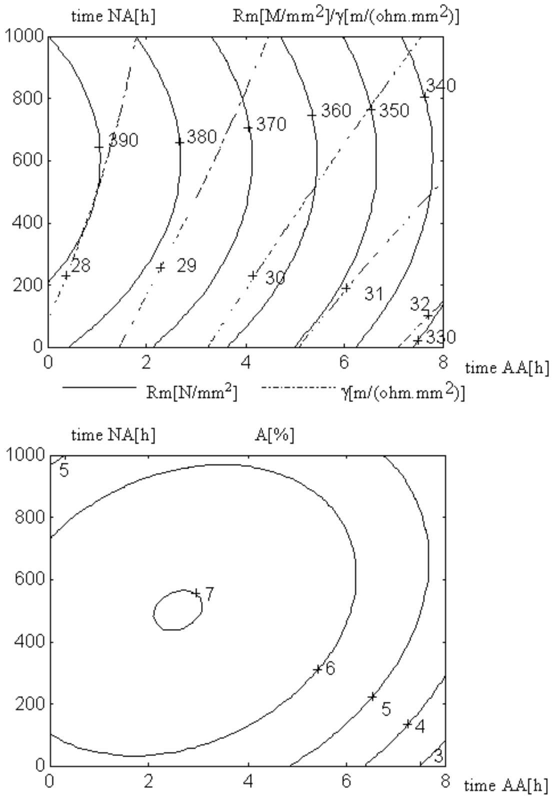

Fig. 7. Isolines of Rm, γ and A for ST-NT 0 - 1000h - ε 95% - AA

short preliminary natural ageing (4 hours) show that final ageing temperature range of 160-170°C enables to achieve Rm 350-360 [N/mm^2] and electrical conductivity γ>30.5-31 [mΩ^{-1} mm^{-2}] for 4.5-6 hours (Fig. 4). The correspon-ding elongation is ~5.5-6% (beyond the max area). When the longer (24-94 hours) NA is given before deformation to obtain the same combination of Rm/γ the final ageing temperatures should be raised up to 165-170°C and the holding time to 5-6.5 hours (Fig. 5). Under ageing at these final conditions, much of the work hardening is preserved, but the electrical conductivity does not reach the the minimum value $\geq$ 30.45 [mΩ^{-1} mm^{-2}] when long term (500-1000hrs) NA is included in termomecanical schedule (Fig.6). In short, the compromised area shifts to higher temperature and longer duration of final ageing if the delay time (NA) between quenching and deformation is too long. At a given time and temperature, for example 170°C, as it is demonstrated in Fig. 7, the extended natural ageing results in higher strength and ductility, but in lower electrical conductivity after TMT compared to the values obtained with shortly stored or immediately-after-quenching deformed wires.

From the slower increment pace of electrical conductivity during final ageing together with a smaller increment of strengths found in a long room temperature pre-aged and then deformed samples it is reasonable to conclude that the natural ageing decelerates the decomposition of solid solution.

3. Disscusion

The summary of information from the latest investigations on precipitation sequence [9,11] of Al-Mg-Si alloy shows inconsistency about the initial stages of decomposition of the solid solution. According to [9] natural ageing more than 30 min leads to formation of GP-I zones (spherical, fully coherent), while Edwards & co-authors [11] proposed that Mg/Si co-clusters form during ageing at room temperature. However, undoubtedly any low-temperature ageing treatment influences the final aged strength. Though the natural ageing between quenching and artificial ageing is generally considered detrimental, there are a number of variables, determining the beneficial or detrimental effect - such as %Mg_2Si in the alloy, duration of delay, etc. As the distribution of hardening phases GP zones-I GP zones II (β”) strongly depends upon the distribution of co-clusters Edwards & co-authors proposed that for any low temperature pre-ageing to be beneficial, sufficient time must be allowed to develop a large number of co-clusters [11].

As our alloy contains ~1 %Mg_2Si and 0,15% excessive Si, the process of Mg/Si clusters (GP-I zones) homogeneous formation is believed to be facilitated. With extension of natural ageing time the density, size [13], Mg/Si ratio [12] and therefore the stability of co-clusters (GP-I zones [14] increase. It appears likely, that they change their internal structure, as it was proposed in some publications [14,15]. The zone formation process causes decrease of solute solution supersaturating since the concentration of solutes in solid solution depends on the degree of room-temperature pre-ageing [15]. In these conditions, structural changes accompanying the thermomechanical processing with room temperature pre-ageing are even more complicated than after T8 TMT. On one hand, because they involve the interaction between available after NA co-clusters (or GP-I zones) and high density of dislocations, introduced by severe deformation, used in the above demonstrated experiments. On the other hand, the subsequent artificial ageing takes place within a highly deformed (i.e. containing a large number of potential sites for heterogeneous nucleation) but with a lower supersaturating matrix. Higher strength and lower electrical conductivity after drawing were obtained with a longer than 4 hours naturally aged wires. Besides work hardening, zones (co-clusters) themselves and their interaction with dislocations contribute to the strengthening.

It is well known, that strengthening, obtained after TMT is related to the size, shape, distribution of precipitates, acting as the barriers for dislocation movement and also to the role of dislocation substructure and mutual influence of the process of precipitation, recovery and recrystallization. The increase of electrical conductivity during final ageing is due to the depletion of solutes from the matrix by precipitation, loss of coherency between precipitates and matrix, and also to the recovery and recrystallization. Over-ageing favours conductivity, while for maximum strength of most commercial Al-Mg-Si alloys the corresponding structure predominantly should contain b”-needle like fully coherent GP-II zones. The chosen temperature and time on the final step of TMT control the structural parameters and precipitation sequence. The small amount of strengthening in artificial ageing treatment, found in our experiments consists with the results, obtained with 6201 alloy non-slip drawn ε=92% wires [1] and also with AD 31 [7]. This certainly can be attributed to an extent work hardening [1,15] even enhanced by including of natural ageing in the TMT schedule. But as on the elongation/final ageing curves the maximum appears usually after 4-5 hours, it can be presumed that some rearrangement in dislocation substructure also occurs. After such a high degree of deformation the recovery susceptibility of the alloy is appreciably increased. This process is superimposed to precipitation during final ageing especially at higher temperature (170°C) of the final ageing. In a conventional T8 treatment, the plastic deformation, applied immediately after quenching is known to accelerate the heterogeneous precipitation of intermediate b' phase, mainly on dislocations. It provides increase in conductivity and strength. When room temperature pre-ageing is given and co-clusters (GP-zones) are already existing depending on their stability, they will easily transform in b"needle like coherent precipitates (GP-II zones). On the assumption that their number and stability are successfully increased when a longer NA is allowed and the solid solution is impoverished, on final ageing after deformation less β' particles will precipitate. The chosen temperatures 130-150°C are much below the GP-zones solvus temperature. As the zones are stable enough the transition β"-β' is retarded. This explains the higher strength and lower electrical conductivity during final ageing at 130°C. The 170°C final ageing accelerates both the recovery (recrystallization) and the transformation of β" in β' resulting in higher conductivity and lower strength (softening) at a given time of pre-ageing. The extended NA time allows the development of a denser distribution of more stable zones (co-clusters). That is why at a given temperature / time of final ageing, a finer structure, dominated by β" precipitates can be expected in a longer room temperature pre-aged samples. TEM observations will be reported in our next studies.

4. Conclusion

The balance between electrical conductivity and ultimate tensile strength in wires of investigated Al-Mg-Si alloy obtained after TMT with room temperature pre-ageing is strongly influenced by both the final aging conditions and natural aging of quenched rod prior to the deformation. The derived mathematical model for graphical predicament of properties outlines the conditions under which an optimal combination of properties may be obtained. Higher final ageing temperature and time is required to design a long-term-pre-aged wires. With a NA up to 100 hours, final ageing at 170°C allows to meet the requirements for a comparatively short time. The extended NA prior to deformation slows down the ageing kinetics due to the increased stability of zones.

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