Application of Electrical Conductivity Measurements in Material Research and in Solution of Technological Problems

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Electrical conductivity depends significantly on a structural state. It can be influenced by chemical composition, content of alloying elements, presence of different types of phases and dislocation substructure as well. Therefore, the relation between electrical conductivity and mechanical properties is very interesting from a material point of view. In combination with structural analyses (metallography, electron microscopy), important information about materials structures and their changes as a function of various factors (deformation, heat treatment) can be obtained. The present paper describes the role of electrical conductivity measurements during an investigation of the effect of technological parameters on the structure and properties of aluminium alloys.

Keywords: electrical conductivity, aluminium alloys, heat treatment, production technology

1 Introduction

Electrical properties of metals and their alloys are described by specific electrical conductivity κ and its reciprocal value ρ (κ=1/ρ) - specific electrical resistance (resistivity). The value of conductivity (resistivity) is affected also by the interaction of conductive electrons with crystal lattice of the metal. The main contribution arises from lattice periodicity variations (defects, modifications) caused by the presence of foreign atoms (impurities, alloying elements) and thermal oscillations of the crystal lattice. This effect is well described by Mathiessen's rule [1]. Total resistivity of the metal containing a small amount of impurities is given by the sum of the contribution $\boldsymbol{\rho}_p$ that is independent on temperature and characterizes the scatter of conductive electrons on foreign atoms and lattice defects, and the thermally dependent contribution $\rho(T)$ describing their scatter on thermal oscillations of the lattice according to the relation

$$\rho = \rho_p + \rho(T) \qquad (1)$$

The unit of electrical conductivity is m/(Ω.mm^2 (alternatively 10^6/Ω.m), that of resistivity is Ω.mm^2/m (alternatively 10^{-6} Ω·m). However, units S.m/mm^2 and MS/m (1 S = 1 Ω^{-1}) are often used in an industrial practice for electrical conductivity κ. For an easier comparison of materials, a conductivity unit 100 % IACS is taken; this is the conductivity of pure copper at 20 °C the value of which is 58.0·10^6 S/m = 58.0 MS/m, i.e. ρ = 0.0172.10^{-6} Ω·m.

The sensitivity of electrical resistivity to the structural state is utilized for the assessment of structural changes connected with lattice defects and precipitation processes. These measurements are used both in the field of a fundamental research (precise resistometric measurements under laboratory conditions) and in the assessment of the structural state as a function of technological parameters during manufacturing (conductivity measurement by means of the probes based on eddy currents [2, 3]). The later one belongs to the group of non-destructive testing methods utilizing the principle of electromagnetic induction. Because an induction of eddy currents occurs in thin surface layer of the material the information obtained characterizes only a narrow volume of the material near the measuring probe. This fact should be taken in consideration whenever this method is applied.

Electrical conductivity measurements by eddy currents were often successfully proved in a technological practice. In the case of a modern instrument Sigmatest 2.069, the method mentioned above has shown to be reliable and sufficiently precise to perform both, laboratory measurements and time-undemanding preliminary measurements under the operational conditions. The measurements have to be carried out in well defined conditions ensuring not only the determination of the electrical conductivity with a sufficient precision but, in particular, also the reproducible values. However, using the eddy current method measurements various factors e.g. the specimen thickness, the quality of specimen surface (roughness), testing frequency, specimen temperature, instrument temperature and measuring probe warming have to be always taken in a consideration. On the other hand, when the eddy current method is applied, the manufacture of the specimens of precise dimensions is not necessary. However, it is necessary to consider a lower precision that does not necessarily enable the detection of fine changes occurring in the structure.

Electrical conductivity values complete very well the results of structure assessment and evaluation of mechanical properties. In some materials, the electrical conductivity is the parameter that is required quite indispensably (conductive materials, hardenable alloys), in other cases, together with the results of microstructure analysis, it is possible to obtain information characterizing the nature of processes occurring in the course of forming or heat treatment.

The presented results refer to the use of Sigmatest 2.069 probe aimed at the effect of technological parameters on the relation between electrical conductivity, structure and properties of products from aluminium and its alloys. Several examples of application of electrical conductivity measurements are given: an improving of conductivity of aluminium for electrical engineering use, assessment of changes occurring during a homogenization annealing, investigation of precipitation processes in hardenable alloys, and solution of technological problems.

2 Improvement of electrical conductivity of aluminium used for electrical engineering

Aluminium products of 99.5 – 99.7 purity (sheets, strips, wires) used in electrical engineering, must ensure a certain minimal electrical conductivity together with required mechanical properties. Measuring of electrical conductivity should be sufficiently precise and reliable. For this purpose, the application of devices using the eddy currents method is quite satisfactory. When stable and well-defined measuring conditions are guaranteed it is possible to monitor and control continuously the effect of technological parameters of the manufacturing process on electrical conductivity of the final product.

Electrical conductivity of aluminium of technical purity is affected by impurities. If the elements are distributed randomly in a solid solution, the dependence of electrical conductivity on the concentration of the element in the solid solution can be described in the first approximation by the relation

$$\rho = \rho_0 (1 + \alpha C) \quad (2)$$

Where:
ρ_0…resistivity of pure aluminium,
C…amount of the dissolved element in wt. %,
α...change of resistivity corresponding to the mass unit of the dissolved element.

The effect of elements in the solid solution on the electrical conductivity of pure aluminium can be divided approximately into three groups. The first group with the strongest effect comprises Li, Mn, Cr, Ti, Mo, Zr and V, the second group with a medium effect consists of Mg, Si, Cu, Fe, Ag and Ni; Sn, Sb, Ga and other elements belong to the third group and their effect on electrical conductivity of Al is relatively small. The most interesting elements for technological purposes influencing electrical conductivity – are above all the transition elements Cr, Zr, Ti and V and the most important impurities Fe and Si. In aluminium materials used for electrical engineering differing only by their purity (i.e. Al 99.5 – 99.7) and containing at most 0.5% of foreign atoms, the electrical conductivity reaches the values of about 34 - 36 MS/m in dependence on the state (wrought, soft) and purity (Al 99.9 – 99.7) of the raw material. Thus, the possibilities for modification of the electrical conductivity are rather limited. The higher is the purity of the material, the more limited are possibilities for the increase of its electrical conductivity.

The increase of electrical conductivity can be achieved with the help of various technological measures, e.g. an alloying of the master alloy by boron or by an annealing at 460 – 470 °C. The alloying of the master alloy results in the formation of borides of the transition elements CrB_2, VB_2, TiB_2 and ZrB_2 which are heavier than the melted aluminium and create the sediments at the bottom of the furnace. On the other hand the annealing aims at the depletion of the solid solution by the transition of alloying elements to the phases, i.e. it leads to a heterogenization of the alloy. Electrical conductivity of the cast aluminium of Al 99.5 to Al 99.7 purity increases at these annealing temperatures by up to 3% [4, 5]. The improvement of the electrical conductivity is firstly due to elements Fe and Mn which are incorporated into the phases. The temperature should not exceed 480 °C; above this temperature, some phases already dissolve and the solid solution becomes enriched with alloying elements, in particular with Mn.

The alloying of master alloys by AlTiB, which is used for grain refinement and cold working, has an unfavourable effect on maximal possible electrical conductivity. Therefore, simultaneous alloying of master alloys by AlB (for the increase of conductivity) and AlTiB (for the grain refinement) requires an optimum dosage of both additions. The effect of cold working on the decrease of electrical conductivity becomes greater with decreasing purity of aluminium and the deformation of about 50% could lead to the decrease of electrical conductivity by 0.08 – 0.36%. After the reduction above 90% this decrease is between 0.3 and 2.5%. Therefore, in order to obtain the requested conductivity values hardening state demanded by the customer should be also taken into the consideration.

It has been shown that the boron alloying by AlB4 (2.8 kg/ ton of Al 99.6) resulted in the increase of the electrical conductivity by 1,8 % and the heterogenization annealing at 475 °C/12 h increased it by further 0.8 % [6]. Electrical conductivity **κ** = 36.01 MS/m (**ρ** = $27.77.10^{-3}$ $\Omega mm^2/m$) was reached. After a simultaneous alloying and heterogenization of Al 99.8, however, the impact on conductivity was considerably lower (about one third of the value mentioned above). It showed to be disadvantageous in economic terms.

3 Homogenization annealing

Homogenization annealing of cast billets, blocks and continuously cast strips belongs to essential technological procedures in the manufacture of aluminium products. This high temperature annealing influences favourably the formability of the as-cast structure. This annealing removes or minimizes dendritic segregation and decreases the macro-heterogeneity of the chemical composition. Because the homogenization modifies the amount of alloying elements in the solid solution and phases distribution at grain boundaries and inside the grains, these changes are well detected by electrical conductivity measurements. Typical example is the effect of the homogenization annealing on the electrical conductivity of round billets for hot extrusion [7]. Homogenization annealing is carried out in chamber furnaces or in continuous ones.

The two ways of annealing differ essentially in the heating rate, dwell on a temperature and by the cooling rate (Fig. 1). Therefore, there is a difference in structural states after annealing proved by the evolution of electrical conductivity across the cross-section of the cast billet. Both ways of homogenization in comparison with the as-cast state of EN AW 6082 alloy exhibit a different shift of curves (Fig. 2). The decrease of the conductivity at the outside rim of round billets is connected with a segregation resulting from a rapid cooling of the surface layer. The longer total dwell on elevated temperature during the annealing in the chamber furnace as well as a lower cooling rate from the annealing temperature lead to a higher electrical conductivity when compared with the continuous annealing, The effect of homogenization on structure of phases is well evident in Fig. 3 and 4, where structures of the as-cast state (Fig. 3) and the state after the homogenization in chamber furnace (Fig. 4) are shown. Homogenization annealing removed the dendritic segregation and led to considerable changes of the distribution and chemical composition of observed particles of other phases.

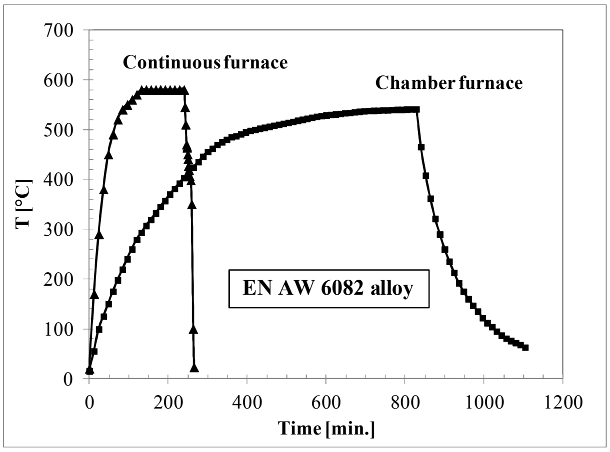

Fig. 1 *Course of temperature during homogenization annealing of cast billets in continuous and chamber furnace*

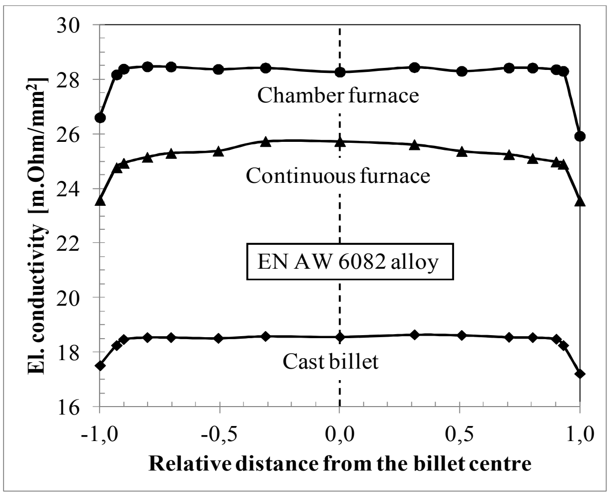

Fig. 2 *Effect of parameters of homogenization annealing in continuous and chamber furnace on electrical conductivity of the cast billets from EN AW 6082 alloy*

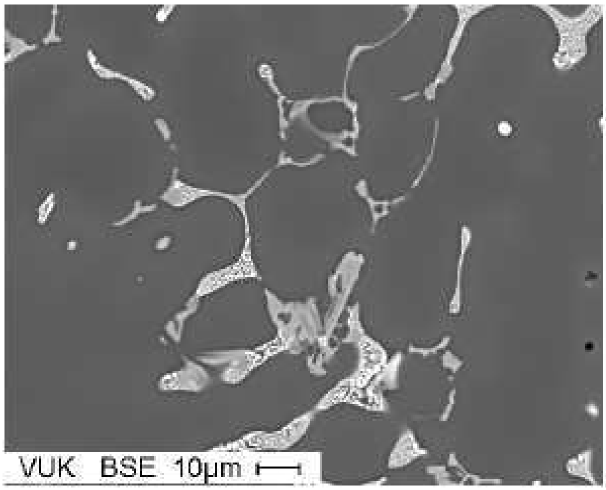

Fig. 3 *Intermetallic phases in cast structure, EN AW 6082 alloy, SEM analysis, [6]*

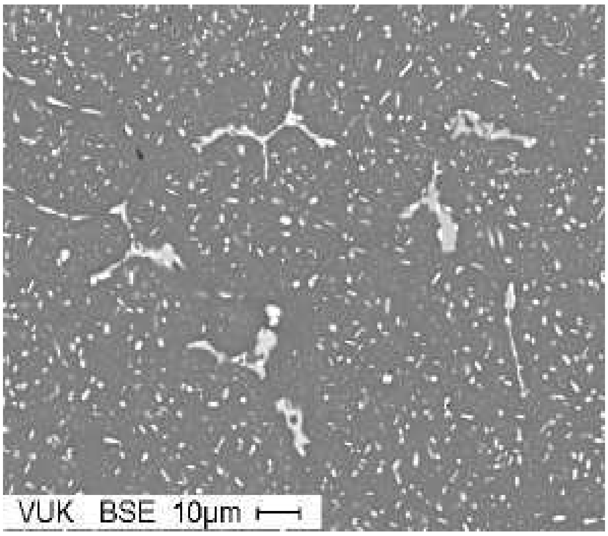

Fig. 4 *Structure after homogenisation in chamber furnace, EN AW 6082 alloy, SEM analysis, [6]*

4 The relation between electrical conductivity and mechanical properties

The relation between electrical conductivity and mechanical properties can be utilized for the investigation of phase transformations. In the field of aluminium alloys, this relation can be used in hardenable as well as in nonhardenable alloys. The most often used mechanical properties are hardness and microhardness. Interpretation of results, particularly in the case of hardenable alloys, however, has to take in consideration that the relation between mechanical properties and electrical conductivity is not linear within the whole range of possible states of the solid solution decomposition. A specific value of electrical conductivity could correspond with different hardness values and, on the contrary, a specific hardness value can be related to different values of electrical conductivity. It is well apparent in Fig. 5, where the relation between hardness and electrical conductivity is shown for a wide range of ageing conditions (natural ageing, artificial ageing, overageing) of EN AW 7010 alloy [8]. In the case of hardenable alloys, this relation is often neither linear nor simple. It could be seen in Fig. 6 and 7 where the changes of hardness and electrical conductivity during the artificial ageing of AlCu3Li2 alloy. Evolution of hardness and conductivity has a similar shape at 130 °C (Fig. 6), while at 160 °C the phase transformations can take place in the way that hardness increases and electrical conductivity remains unchanged (Fig. 7). To understand the structural changes, physical interpretation of the results of measurements of electrical conductivity and hardness is necessary to complete with results of microstructural analysis (SEM, TEM) and a more precise measurements by means of resistometry [9, 10].

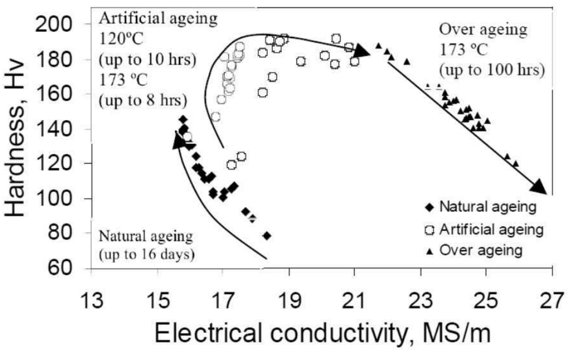

Fig. 5 *Hardness and electrical conductivity profile of AA7010 aluminium alloy at different stages of ageing [8]*

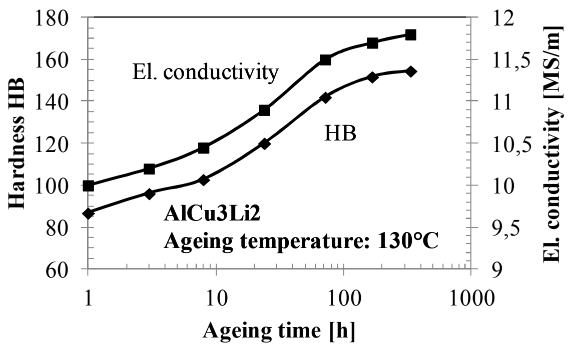

Fig. 6 *Electrical conductivity and hardness HB during artificial ageing of AlCu3Li2 at 130 °C*

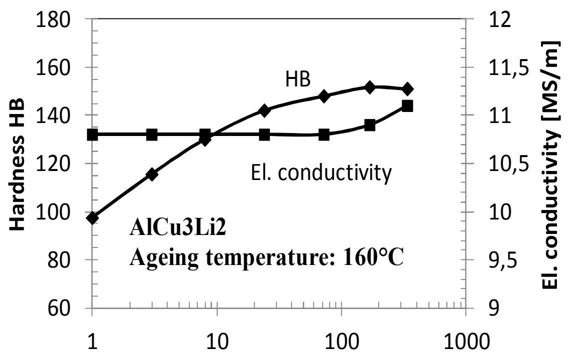

Fig. 7 *Electrical conductivity and hardness HB during artificial ageing of AlCu3Li2 at 160 °C*

5 Inhomogeneities of structure and properties

Measurement of electrical conductivity with the help of eddy currents enables to detect the course of electrical conductivity on both large and relatively small areas and, thus, to assess indirectly an inhomogeneity of the structure and properties. It can be used even for the solution of such technological problems that could be solved by standard procedures with difficulties or not at all. An example is an inhomogeneous crystallization of cast billets of 370 mm diameter from EN AW 6082 alloy. When some anomalies during crystallization occur in the billet, they can influence the structure and properties in the course of following technological operations. They can appear in extruded rods and also afterwards in forgings. Some defects caused by improper crystallization cannot be detected by common methods (macrostructure, microstructure, tensile test) because they are of local type. The following case concerns an anomaly (places with lower hardness) which occurred in extruded rods and forgings. The anomaly was detected after the heat treatment of the product and only in the case of the surface sand blasting. Plastic deformation which takes place during the blasting makes softer places visible. The origin of such anomaly could not be determined unambiguously because of the fact that technological operations (heat treatment, hot deformation and homogenization annealing) that could influence the formation of this defect were carried out before its identification. The place of the defect shows markedly lower hardness HBW=85 than its surroundings (HBW=115) after the heat treatment. In addition to the lower, unsatisfactory hardness, a substantially higher electrical conductivity was found at this place (27 MS/m) than in its surroundings (25 MS/m). It is clear that at the place of the defect, a structure different from that of the surroundings (chemical composition, state of decomposition of the solid solution) must be present. Series of tests proved that there was not the problem connected with the heat treatment and extrusion process. There were the values of electrical conductivity which contributed to revealing the cause of the origin of the soft places and their localization. Proper control of thermally treated butt (end of non deformed extruded billet with cast structure) revealed the places showing a higher electrical conductivity and lower hardness. An example of the heat treated and machined butt with a local structural anomaly is given in Fig. 8. The course of electrical conductivity across the cross-section is depicted in Fig. 9. Structural investigations (metallography, TEM), showed that after etching the area of the structural defect had a different color from the surroundings (Fig. 10). The state of decomposition of the solid solution and the phase composition at the place of the defect were also different. At the place of the defect, coarser, equilibrium Mg_2Si particles remain undissolved and they often appear together with larger Si particles; metastable Mg_2Si particles of the hardening phase in a different state of decomposition are also present here. Overageid β´´ phase or β´ phase (Fig. 11) are the predominant phases at the place of the defect. While a required dense network of hardening coherent β´´ phase corresponding to the heat treated T6 temper appear in its surroundings (Fig. 12).

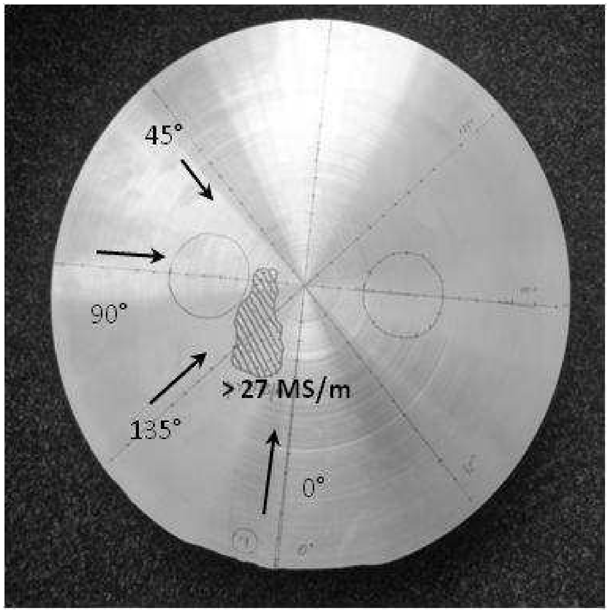

Fig. 8 *Position of zone exhibiting higher electrical conductivity in the place of anomalous crystallization in the butt of two hole die; situation after heat treatment to T6 temper*

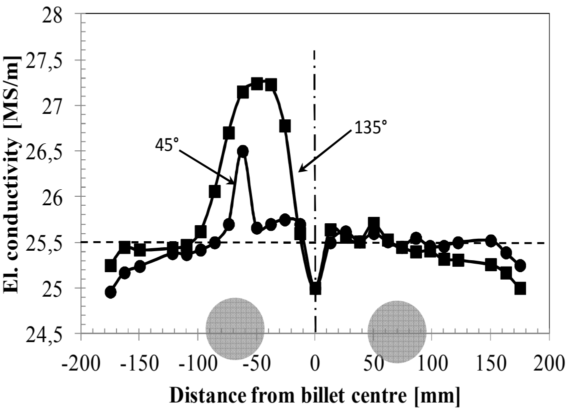

Fig. 9 *Electrical conductivity across the cross-section of butt of two hole die after heat treatment to T6 temper*

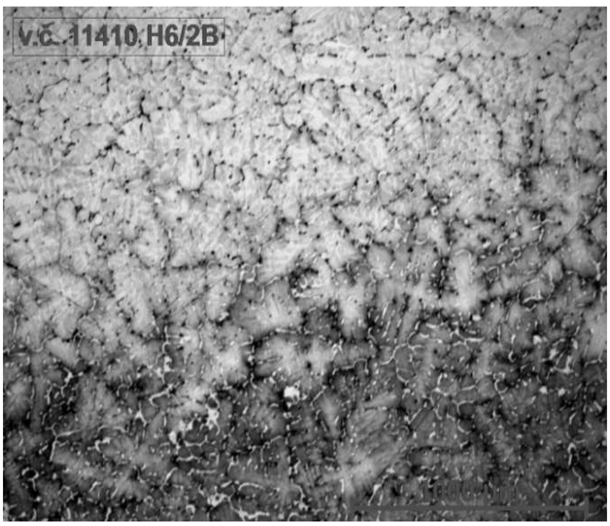

Fig. 10 *Fuzzy boundary between defect and surrounding structure after etching*

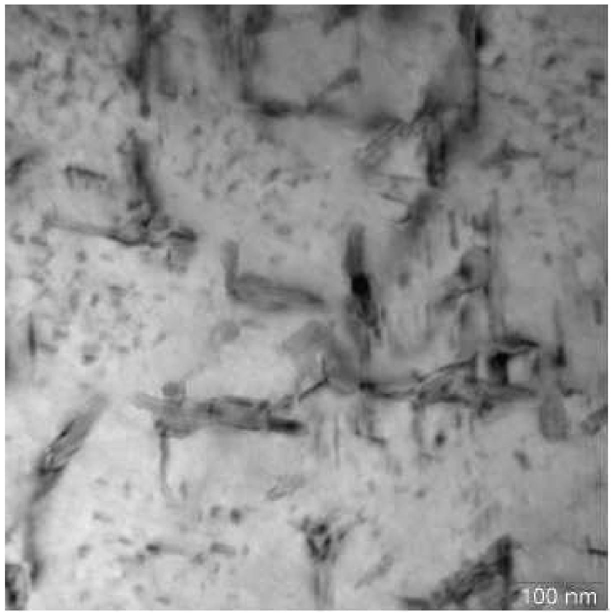

Fig 11 *Structure of phases at the defect place, TEM. Occurrence of overageid phases β´´ and phase β´; fine hardening β´´ phase which is present at structure outside the defect, doesn´t occur here*

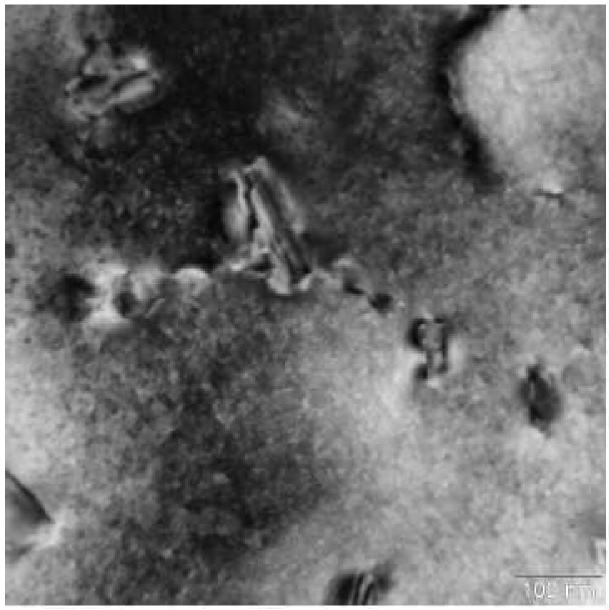

Fig. 12 *Structure of phases outside the defect, TEM. Presence of fine hardening β´´phases*

6 Conclusion

Electrical conductivity measurement by means of the eddy currents shows to be an effective method for indirect evaluation of the structure of aluminium alloys Simultaneous evaluation of mechanical properties and carrying out structural analyses enables to obtain important information on processes taking place in the structure during a series of technological operations. This method is beneficial at observation of thermally activated processes during the casting, homogenization annealing and, in particular, at investigation of phase transformations in the course of decomposition of the solid solution in hardenable alloys. Examples given in the paper, show that its use is useful in both, the industrial practice during the quality check and solution of technological problems, and in the applied research during an optimization of technological parameters.

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