HIGH-TEMPERATURE PROCESSES OCCURRING DURING HOMOGENIZATION OF AA6082 ALUMINUM ALLOY

Miroslav Cieslar[1], Jan Bajer[1], Michal Hájek[1], Vladivoj Očenášek[2]
[1]Charles University in Prague, Faculty of Mathematics and Physics, Ke Karlovu 5, CZ-121 16 Prague 2, Czech Republic
[2]SVÚM a.s., Podnikatelská 565, CZ 190 11, Prague 9, Czech Republic

Keywords: AA6082, Homogenization annealing, In-situ SEM

Abstract

Homogenization conditions of the 6082-type aluminum alloy were tested by means of electrical resistivity measurements and in-situ heating SEM experiments. It was shown that close to 540 °C a partial local melting of dendrite cell boundaries occurs accompanied by a significant Mn, Mg and Si solid solution enrichment. The process is significantly accelerated and the surface damage due to the local melting is more pronounced when the temperature is risen to 560 °C. At higher temperatures a full surface melting and alloy degradation occurs.

Introduction

Aluminum alloys of the 6XXX series are often used in the form of extrusions or die forging materials. These multiphase alloys belongs to commercial ones based on the Al-Mg-Si alloy system where the main strengthening effect arises from the homogeneous distribution of fine metastable Mg2Si phase [1]. In the technical aluminum alloys besides the intentional additions, other elements, such as Mn and Fe or Cu may be present. During casting a wide variety of ternary and more complex phases are formed [2,3]. Type of these phases and their distribution depends mainly on the cooling rate. The processing of the final product consists of several steps that may significantly influence the ultimate properties. The first important treating step is the homogenization annealing of the as-cast alloy. During this treatment several processes take place resulting in more homogeneous distribution of main strengthening elements, partial dissolution of dendrite structures and phase transformations accompanied often by the spheroidization of undissolved sharp particles [4]. As this annealing requests high temperature not very far from the melting point, the optimization of the homogenization process is a crucial task which may significantly affect both, the final products quality and their price. The segregated regions can melt at lower temperatures causing severe damage therefore correct choice of the homogenization temperature and dwelling time is a crucial task, which has a significant influence on the material final properties. Several indirect methods such as DSC, DTA or resistivity measurements combined with direct light or scanning electron microscopy are often used for the homogenization conditions optimization. Nevertheless, as the thermal analysis methods are integral, they are not able to distinguish directly between several different processes and even if the data are combined with post-mortem SEM or TEM observations they often give ambiguous interpretation. One of the methods which enable direct observation of the microstructure changes during homogenization is the in-situ electron microscopy in the heating stage.

In the present work the processes occurring on the surface of the specimen prepared from the technical AA6082 type alloy were studied by the in-situ heating SEM. The main interest was focused on the evolution of inter-dendrite spaces containing microsegregated particles of second phases.

Experimental

A commercial AA6082 alloy with a composition (in wt. %) Si – 1 .2, Mg – 0.8, Mn – 0.5, Fe – 0.3 was used in the study. 1 mm thick strips for SEM observation were cut from the as-cast ingot, mechanically grind by the SiC papers to the thickness of 0.2 mm and polished by the alumina suspension. 3 mm in diameter discs were punched from the strip and finally electropolished in the 30 % nitric acid solution in methanol and clumped in the low-noise resistance heating stage. Specimens for TEM observations were prepared from the same discs by the final electropolishing in a twin-jet Tenupol 2 device using identical solution as for the SEM ones. The SEM and TEM observations were performed in the electron microscope JEOL JEM 2000FX equipped with the EM-ASID 10/20 scanning image device and EDS Bruker AXS UHV Si(Li) detector with the QUANTAX controller system. For the determination of the alloy integral behavior, the resistivity measurements were performed on 1 mm thick strips using the DC 4 point method in a linear heating regime using a home-made system equipped with the Keithley computer controlled devices.

Results and Discussion

The low solid solubility of alloying elements leads to the formation of secondary phases in the as-cast microstructure. The size and morphology of these phases depends on the chemical composition, the dendrite arm spacing, the grain size and the local solidification time. The typical initial microstructure observed in the alloy is given in Figure 1. Characteristic structures at the cell boundaries of α-solid solution contain a mixture of second phase particles rich in Fe and Mn (generally β-AlFeSi and α-AlFeMnSi phases are reported [1,3,5]), pure Si and equilibrium Mg_2Si phase. The presence of the above mentioned phases in the as-cast state was confirmed also by TEM. Typical dendritic structures are shown in Figure 2. The matrix between dendrites is generally free of any precipitates, only locally small fraction of thin particles of Mg-rich β'-phase [1] were observed (Figure 3).

The resistivity measurements (see Figure 4) reveal significant changes occurring in the material at temperatures near 250 °C and 450 °C. Nevertheless, these changes are known to be associated with phase transformations inside dendrites (precipitation and dissolution of the Mg_2Si β-phase (below 400 °C) and α-AlFeMnSi phase [1] at higher temperatures) that are not a subject of the present study. Nevertheless, the presence of the β-phase and the α-AlFeMnSi phase were also proved by TEM in the material after the homogenization treatment (Figure 5). As seen on the Figure 4 and on the detail in the inset, above 540 °C a local minimum on the derivative of the resistivity curve was observed, followed by a deep drop of this value above 565 °C.

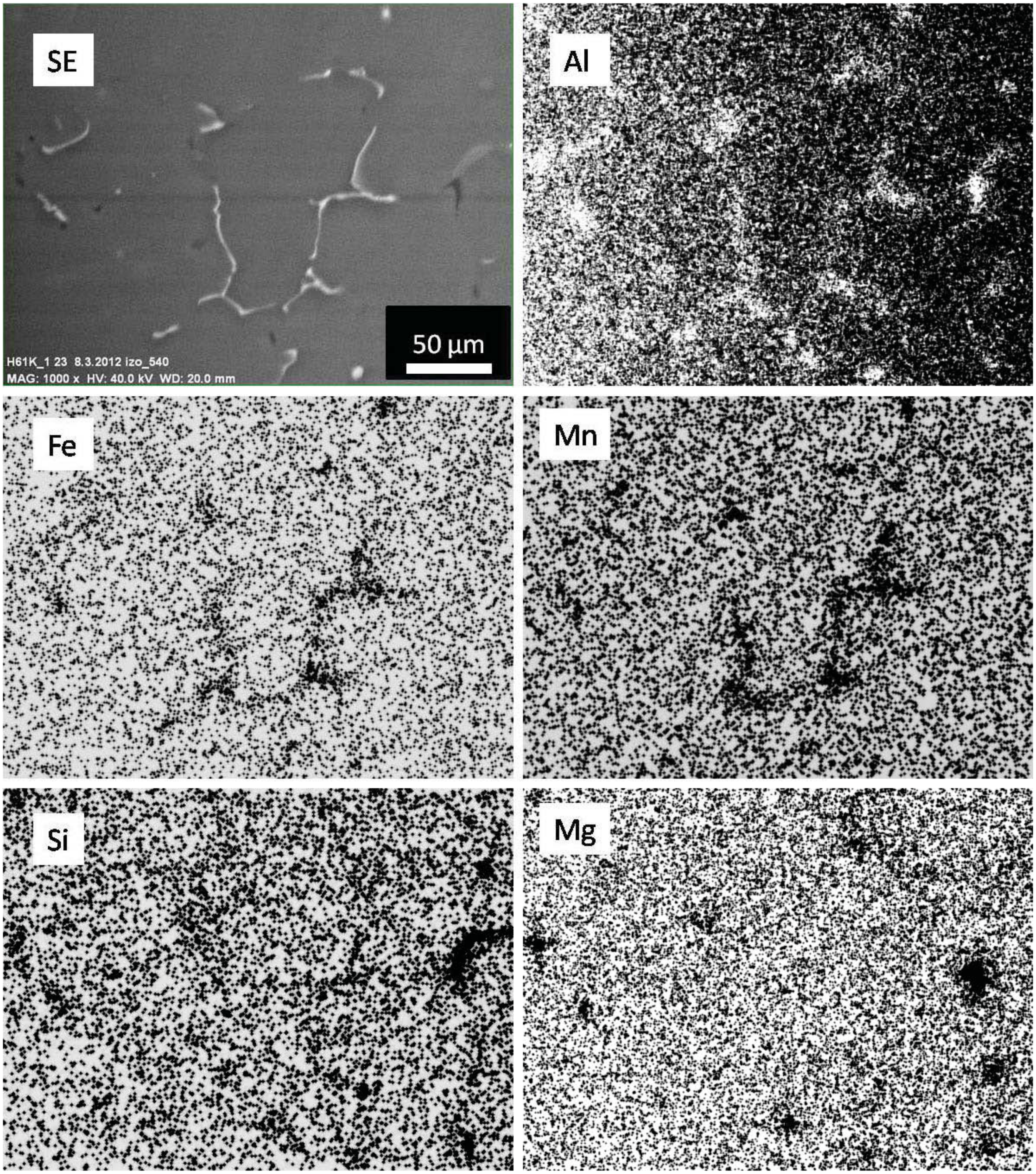

Figure 1. SEM image of the initial as-cast microstructure of the AA 6082 alloy (secondary electrons – SE) and the distribution of main alloying elements (dark spots) determined by EDS.

The SEM observations during the in-situ heating were performed on specimen annealed in the step-by-step heating regime with the step of 20°C/4min which corresponds to the continuous regime of 5°C/min used during resistivity measurements. The modification of the continuous regime was required due to the longer time which is necessary for SEM image stabilization and acquisition. Nevertheless, the effective heating rate remained identical in both, SEM and resistivity measurements. The Figure 6 shows a

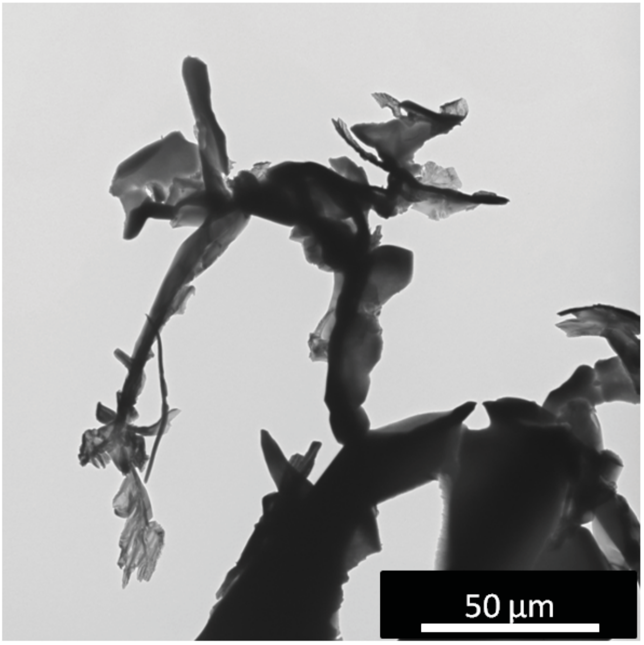

Figure 2. Fe and Mn rich particles at dendrite boundaries in the as-cast material.

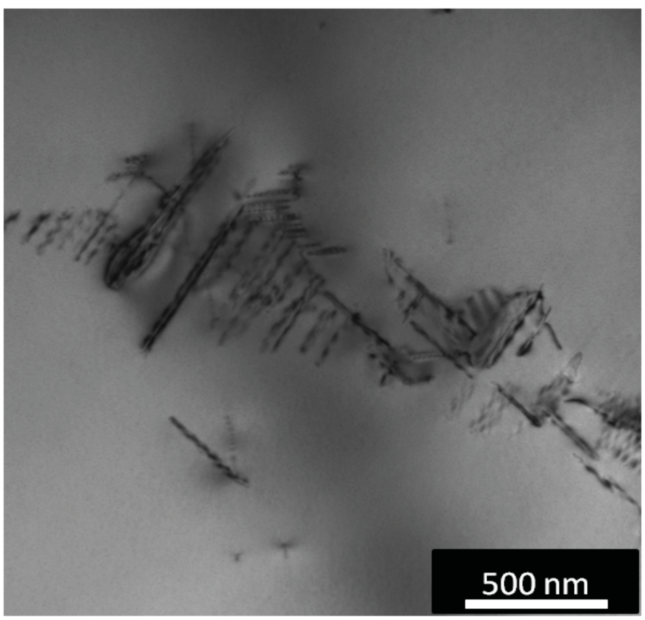

Figure 3. TEM image of rarely distributed particles of the Mg-rich β’-phase in the as-cast material.

sequence of images acquired on specimen isochronally annealed up to 560 °C and then held at this temperature. First significant changes in the microstructure of cell boundaries were observed very shortly after the specimen reached the requested temperature. Within two minutes all the dendritic boundaries were fuzzy and smeared on a distance of about 20 micrometers. The first minutes of the exposure at this temperature result in the partial dissolution of cell boundary particles and long distance diffusion of alloying elements into the dendrite cell interiors. Partial boundary melting could not be excluded. Finally, at longest annealing times, the grain boundaries contain only debris of original or transformed phases rich in Fe, Mn and traces of Si, that were not dissolved during the treatment. At temperatures above 570 °C the melting of the material occurred and after solidification at lower temperatures distinct dendrites were formed on the specimen surface (Figure 7).

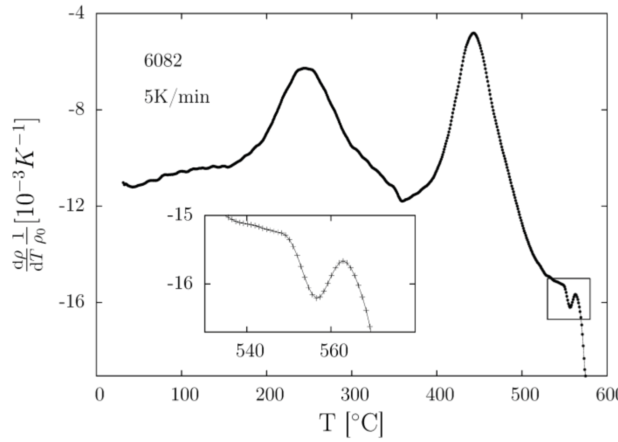

Figure 4. Derivative of the resistivity annealing curve. Processes occurring on cell boundaries are responsible for the changes shown in the inset. ρ_0 is the value of resistivity ρ in the as-cast state.

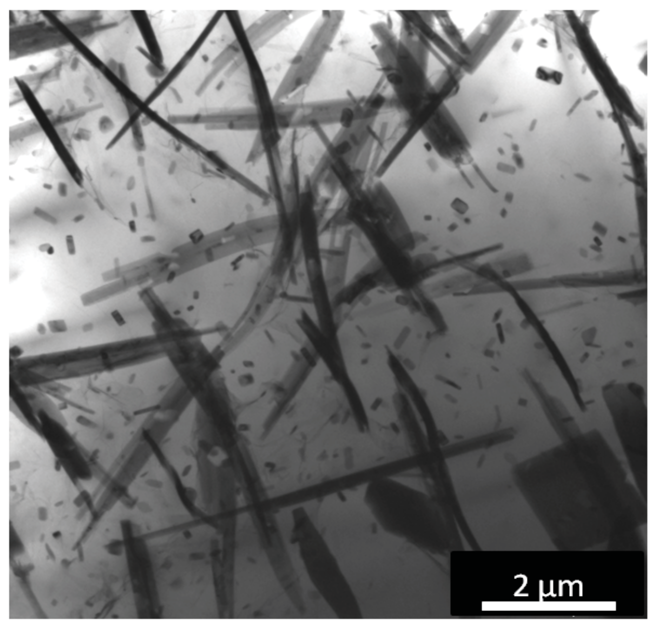

Figure 5. TEM image of Mg_2Si precipitates (long particles) and fine particles of α-AlMnSi phase in the material after the homogenization annealing and slow cooling down to room temperature. Coarser particles in the bottom right corner are the residual partially dissolved particles of primary phases.

The SEM experiments clearly show that the processes responsible for the resistivity changes at temperatures around 560 °C are mainly the ones observed at the boundaries of the dendrite cells. The undulation which is present on the resistivity derivative curve in this temperature range is most probably associated with two competing processes - the local cell boundary melting and solid solution enrichment due to the cell boundary particles dissolution.

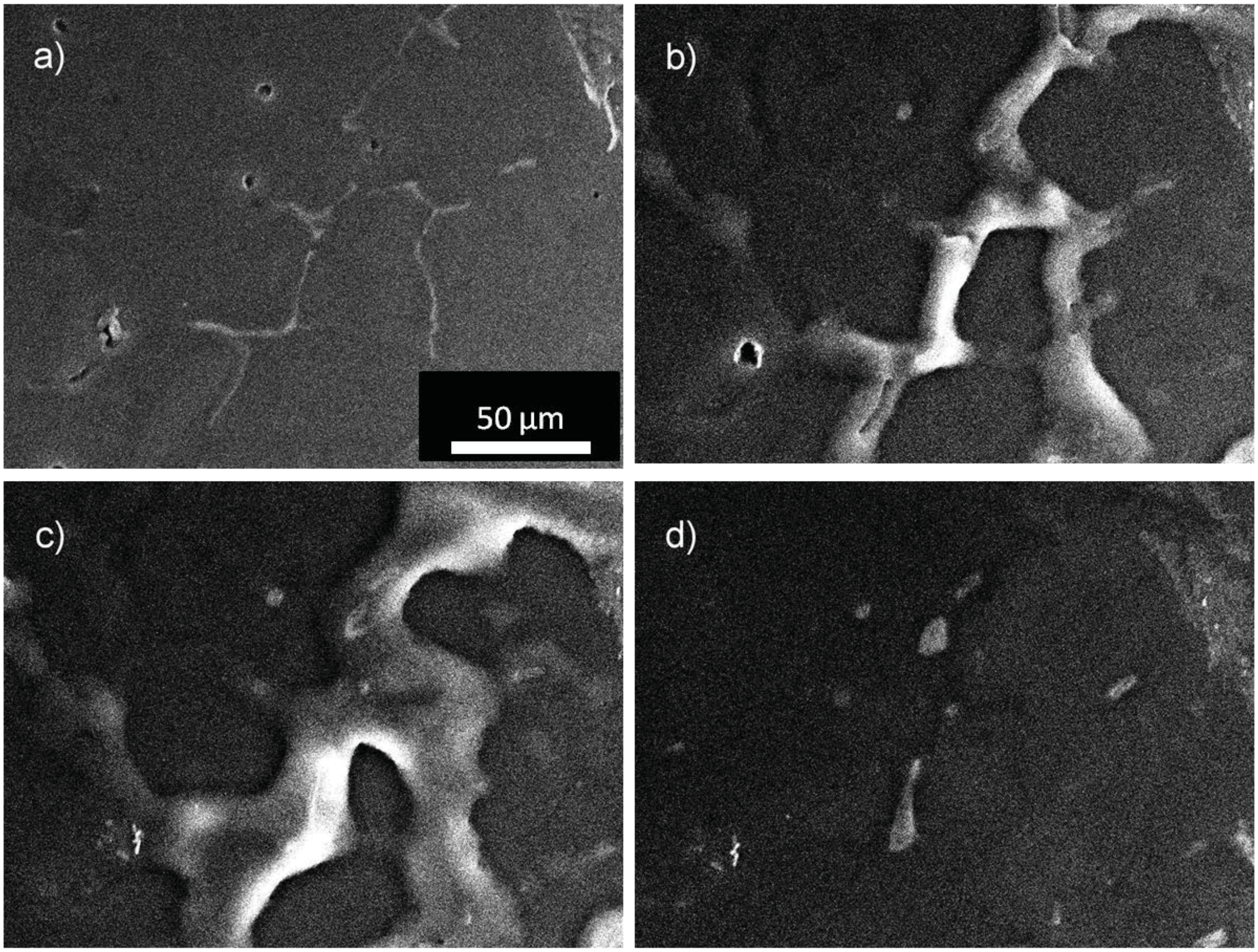

Figure 6. Evolution of the microstructure at 560 °C. SE images in the as-cast specimen (a) and specimens isothermally annealed for 120 s (b), 240 s (c) and 600 s (d).

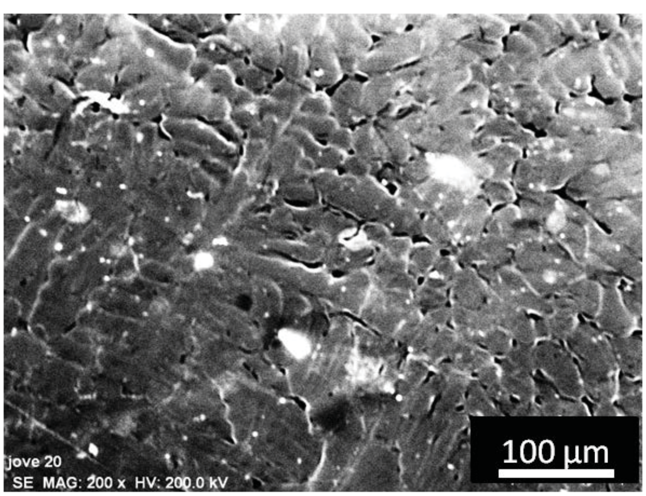

Figure 7. SEM image of the specimen after annealing at 570 °C and cooling down to room temperature.

The first one causes surface morphology changes and also a drop on the resistivity derivative curves. This drop is, however, strongly retarded by the rising resistivity of the matrix caused by diffusing Mn, Mg and Si atoms. When all the dissoluble cell boundary particles are diluted and there is no more driving force for the matrix enrichment, the curve of the resistivity derivative drops again.

Conclusions

Homogenization conditions of the 6082-type aluminium alloy were tested by means of the electrical resistivity measurements and in-situ heating SEM experiments. It was shown that at temperatures close to 560 °C a partial local melting of dendrite cell boundaries occurs accompanied by a significant Mn, Mg and Si solid solution enrichment. At higher temperatures a full surface melting and alloy degradation occurs.

Acknowledgements

The financial support of the project TA02011137 of the Technology Agency of the Czech Republic is gratefully acknowledged.

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