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THE EFFECT OF HOMOGENIZATION CONDITIONS ON THE STRUCTURE AND PROPERTIES OF 6082 ALLOY BILLETS

WPŁYW WARUNKÓW HOMOGENIZACJI NA STRUKTURĘ I WŁASNOŚCI WLEWKÓW ZE STOPU 6082

The paper presents the results of laboratory homogenization investigations of the 6082 grade alloys, differing in Mg and Si content. At the first stage, the microstructure of alloys was analysed after homogenization finished with water quenching. SEM/EDS investigations and DSC tests were applied to evaluate the dissolution of the Mg_2Si particles and concentration of the main alloying additions in the grains interiors, depending on soaking conditions. In the case of alloy with lower Mg and Si content, homogenization the temperature of 535°C for 8h is sufficient for significant Mg_2Si particles dissolution. For the alloy with higher Mg and Si content, after homogenization the temperature of 550°C for 8h, the amount of undissolved Mg_2Si particles decreases visibly, compared to homogenization at 535°C for 8h. However, an unfavourable tendency of dispersoids growth is observed and these soaking conditions are not found to be recommended.

At the second research stage, the influence of homogenization cooling rate on the size and distribution of the Mg_2Si particles observed in the alloys microstructure was analysed. The ability of the Mg_2Si particles, precipitated during various homogenization coolings, to rapid dissolution was estimated. For this purpose, the hardness after solution heat treatment with short annealing and ageing was determined and the DSC tests were performed. It was found, that cooling after homogenization at 315°C/h is sufficient for precipitation of fine Mg_2Si particles, which dissolve during subsequent rapid heating. Cooling at 40°C/h, causes precipitation of Mg_2Si phase in the form of large particles, unable for further fast dissolution.

Keywords: 6082 alloy, homogenization temperature, cooling rate after homogenization

W artykule przedstawiono wyniki laboratoryjnych badań homogenizacji dwóch stopów w gatunku 6082, różniących się zawartością Mg i Si. W pierwszym etapie analizowana była struktura stopów po homogenizacji z chłodzeniem w wodzie. Szczególną uwagę zwrócono na rozpuszczanie cząstek fazy Mg_2Si podczas wygrzewania oraz zawartość głównych składników stopowych we wnętrzach ziarn. Oceny parametrów wygrzewania dokonano w oparciu o badania SEM/EDS oraz testy DSC. W przypadku stopu z niższą zawartością Mg i Si, homogenizacja w temperaturze 535°C przez 8 h jest wystarczająca dla znaczącego rozpuszczenia cząstek fazy Mg_2Si. W przypadku stopu z wyższą zawartością Mg i Si, po homogenizacji w temperaturze 550°C przez 8h, udział nierozpuszczonych cząstek Mg_2Si spada wyraźnie, w porównaniu z homogenizacją w temperaturze 535°C przez 8h. Jednakże, stwierdzono niekorzystną tendencję do rozrostu dyspersoidów, zatem tych warunków wyżarzania nie uznano za rekomendowane.

W drugim etapie analizowano wpływ szybkości chłodzenia z temperatury homogenizacji na wielkość i dystrybucję cząstek Mg_2Si obserwowanych w strukturze stopów. Wykonano badania SEM oraz dokonano oceny możliwości szybkiego rozpuszczania cząstek Mg_2Si wydzielonych podczas chłodzenia z temperatury homogenizacji z różnymi szybkościami. W tym celu wykonano badania twardości stopów po przesycaniu z krótkim wygrzewaniem oraz starzeniu, jak również badania DSC. Stwierdzono, że studzenie z temperatury homogenizacji z szybkością 315°C/h jest wystarczające dla wydzielenia drobnych cząstek Mg_2Si, które ulegają rozpuszczeniu podczas późniejszego gwałtownego nagrzewania. Chłodzenie z temperatury homogenizacji z szybkością 40°C/h powoduje wydzielanie fazy Mg_2Si w postaci dużych cząstek, które nie ulegają pełnemu rozpuszczeniu podczas późniejszego szybkiego nagrzewania.

1. Introduction

The homogenization of aluminium alloys billets intended for extrusion is an essential part of the technological process. It is necessary for achieving high productivity as well as expected mechanical properties and quality of the extruded profiles. In the case of AlMgSi alloys, following microstructural phenomena are taking place during heating to the homogenization temperature and soaking [1-4]:

- Dissolution of the Mg_2Si phase particles, formed during casting and elimination of the microsegregation,
- Precipitation of the dispersoids (in alloys containing transition elements, such as Mn and Cr),
- Transformation of the β-AlFeSi intermetallic phase into more favourable α-Al(FeMn)Si phase.

In order to obtain suitable microstructure of the billets,

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the abovementioned structural changes must be taken into consideration during designing the homogenization parameters. As an example, the problem of soaking temperature selection for high strength 6xxx alloys, such as 6082 or 6061, can be mentioned. Homogenization in a high temperature range (eg. > 565°C) facilitates dissolution of the Mg_2Si particles and increases Mg and Si content in a solid solution obtained at the end of a soaking stage. On the other hand, it causes growth of dispersoids, which results in reducing of recrystallization resistance of the material. In consequence, the risk of peripheral coarse grain layer formation and lowering of mechanical properties (to which the press-effect does not contribute) appears in extruded products [5, 6].

Another important parameter of homogenization process, particularly in the case of extrusion with solution heat treatment at the press output, is the cooling rate of billets. This parameter determines the size and distribution of the Mg_2Si particles observed in the billets microstructure after cooling [7, 8]. The Mg_2Si particles present in the microstructure of very slow cooled billets are too large to dissolve entirely during subsequent preheating and extrusion process.

As the result, when the profile leaves the die orifice and the quenching at the press output begins, the concentration of Mg and Si in solid solution is reduced. Thus, the final strength properties of the extruded products are inadequate [7]. Moreover, presence of Mg_2Si particles in the billets microstructure, causes an incipient melting of the alloy at a relatively low temperature, as the result of the unequilibrium eutectic reaction between the particles and surrounding matrix. This melting reaction initiates formation of the profiles surface defects. In order to avoid defects appearance, the exit temperature of the profile must be kept below the eutectic temperature of the alloy and the permissible extrusion speed is reduced [7, 9]. The size of Mg_2Si particles precipitated during billets cooling, decreases with an increasing billets cooling rate (due to higher undercooling), and dissolution of the particles, during subsequent preheating and extrusion, is facilitated. However, too high cooling rates from homogenization temperature are also unfavourable, because of rising supersaturation of the solid solution with Mg and Si. It increases flow stress of the material and breakthrough-pressure [10], which is adverse, especially in the case of extrusion of thin-walled profiles from high-alloyed 6xxx alloys. It follows from above that the billets should be cooled down from the homogenization temperature in a manner that enables precipitation of high amount of Mg_2Si particles, in form and size fostering their further dissolution. This is achieved by continuous cooling with suitable cooling rate or by application of isothermal step on the cooling curve [10-15]. Cooling conditions are selected with respect to the alloy composition, billets diameter, construction of homogenization furnace, but also important are the parameters of subsequent extrusion process [16].

From the remarks presented above it follows that obtaining good extrudability of 6xxx alloys billets and simultaneously the possibility of achieving high strength properties of the profiles, requires precise determining of the homogenization conditions. The presented paper describes the problem of selecting homogenization temperature and cooling rate for 6082 alloy billets, intended for extrusion of thin-walled hollow profiles with solution heat treatment at the press output.

2. Experimental work

The billets of two alloys, with chemical composition within the range of 6082 grade (TABLE 1), were DC cast in semi-industrial conditions.

TABLE 1

Chemical composition of the investigated 6082 alloys, wt%

	Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	Al
alloy 1	1,04	0,19	0,004	0,52	0,67	0,005	0,009	0,02	bal.
alloy 2	1,12	0,18	0,013	0,52	0,79	0,005	0,009	0,02	bal.

The material in as-cast state was subjected to microstructure observations and DSC analysis.

A Hitachi SU-70 scanning electron microscope equipped with EDS detector was used for microstructure observations. The samples for SEM investigations were prepared using standard metallography methods, including mechanical grinding and polishing with diamond suspensions and colloidal silica. EDS analyses were applied to determine the chemical composition of observed particles and to measure Mg and Si content in the grains interiors after homogenization.

DSC analyses were performed using a Mettler Toledo 821^e heat flux type calorimeter. The disc shaped samples were inserted in ceramic pans into the cell with the temperature of 490°C and heated 20°C/min to the temperature of 700°C. The solidus temperature and heat of the incipient melting reactions were determined.

The samples, mechanically sectioned from billets, were subjected to homogenization in laboratory conditions. At the first stage of work, soaking parameters were evaluated. The alloy 1 was homogenized at the temperature of 535°C for 8h. In the case of alloy 2, with higher content of Mg and Si, soaking was performed at the temperature of 535 and 550°C for 8h. In all the cases the samples were heated up to homogenization temperature during 6 h and quenched in water after soaking.

At the second stage, the influence of cooling rate from the homogenization temperature on the billets microstructure was investigated. Both alloys were heated to the temperature 535°C during 6h, soaked for 8h and cooled to room temperature in two ways. The average cooling rates, estimated on the basis of samples temperature measurements recorded during cooling cycles (Fig. 1.), in the temperature range of 535-200°C, were 315 and 40°C/h. The parameters of all homogenization variants, are summarized in TABLE 2.

The materials after homogenization were subjected to SEM observations and DSC analysis, as described above. The microphotographs of specimens cooled in water after homogenization, were additionally subjected to image analysis procedure. For each specimen, 10 photographs recorded by magnification 500x in randomly selected areas were collected (the total analysed area was 0,451 mm^2). Microstructure images were binarized in order to determine the number and area of undissolved Mg_2Si particles in the microstructure. An example of binarization procedure result is presented in Fig. 2.

TABLE 2

Parameters of the laboratory homogenization experiments

Material	Heating	Soaking	Cooling
alloy1	20-535°C/6h	535°C/8h	water quenching
			315°C/h
			40°C/h
alloy 2	20-535°C/6h	535°C/8h	water quenching
			315°C/h
			40°C/h
	20-550°C/6h	550°C/8h	water quenching

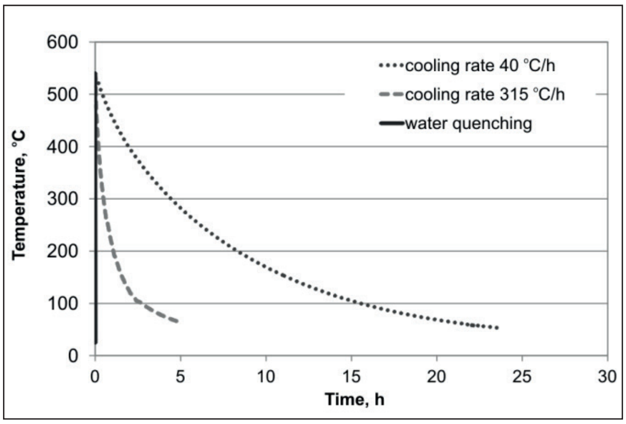

Fig. 1. Cooling curves recorded during homogenization experiments

In order to evaluate the Mg_2Si particles dissolution ability during rapid heating, which occurs in the extrusion process, the following experiment was realized: the homogenized samples were annealed at the temperature of 540°C for 5 min., quenched in water and aged at the temperature of 175°C for 8h. Afterwards, Vickers hardness of the age hardened samples was measured using a Shimadzu MHV hardness tester (nominal test force: 19,61 N, dwell time 10 s.). It must be noted, that this experiment is only an approximation of the industrial conditions. During the experiment, similarly as in the extrusion process, the material was heated within a few minutes to the temperature of the alloy at a die exit (540°C). In the laboratory test, however, the deformation did not take place and the temperature course during billet preheating (dependent on heater type, extrusion speed etc.) was not simulated.

3. Results and discussion

In the microstructure of both as-cast alloys (Fig. 3) two types of particles are observed: Mg_2Si (black) and AlFe(Mn)Si (white). The particles have characteristic sharp, elongated shapes and are located mainly on grains boundaries.

On the DSC curves (Fig. 4.), incipient melting peaks with the onset temperature 571°C for alloy 1 and 576°C for alloy 2 are observed (TABLE 3). They are the result of mixture areas melting that have formed during solidification reactions [1, 17].

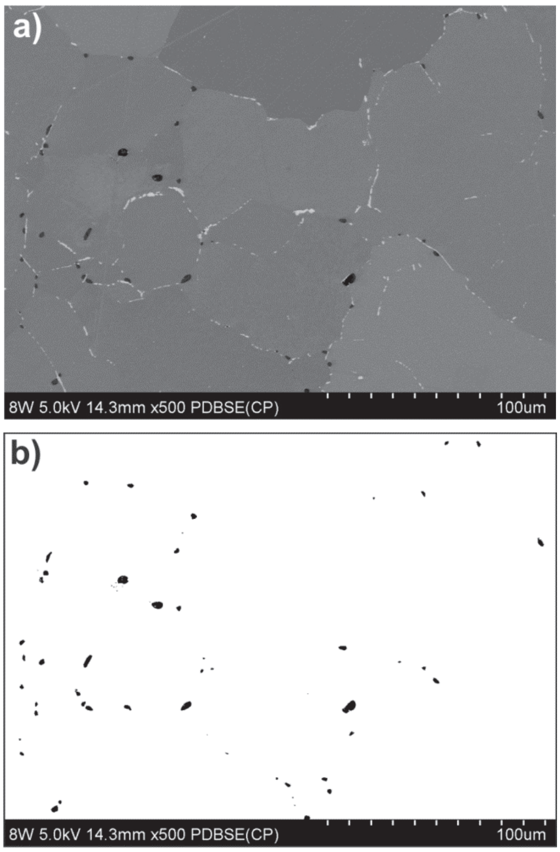

Fig. 2. An example of binarization transformation for number and area determination of Mg2Si particles in the microstructure

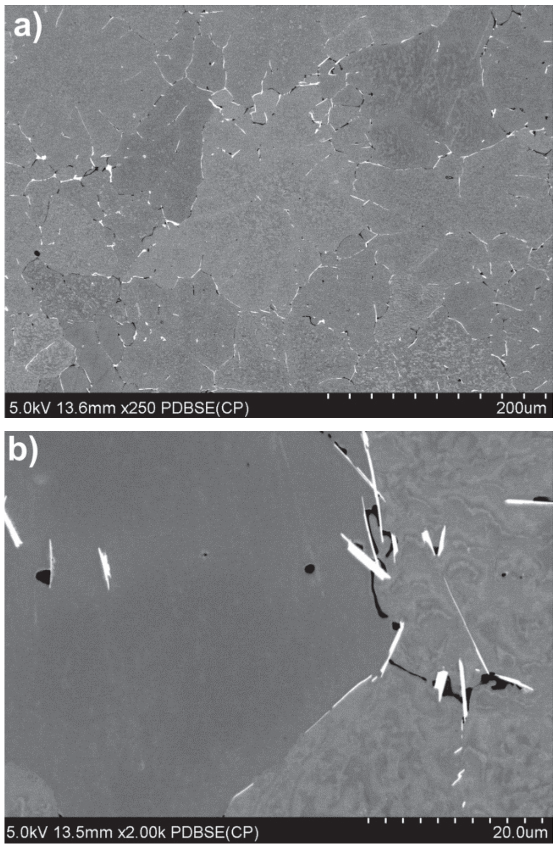

Fig. 3. Microstructure of investigated billets in as-cast state: alloy 1 (a, b)

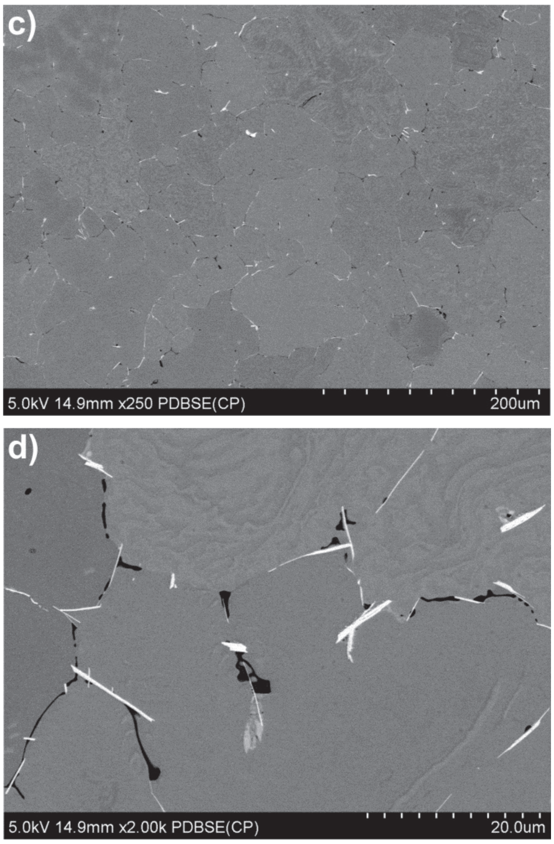

Fig. 3. (continued) Microstructure of investigated billets in as-cast state: alloy 2 (c,d)

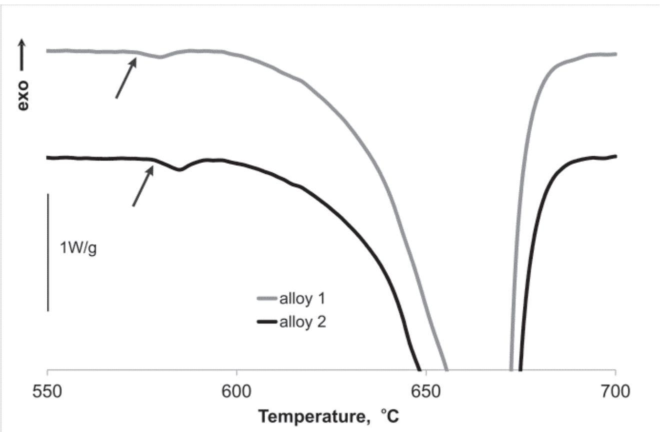

Fig. 4. DSC curves of as-cast alloys

TABLE 3

DSC analysis results of as-cast alloys

	Solidus temperature, °C	Incipient melting heat, J/g
alloy 1	571	1,1
alloy 2	576	1,7

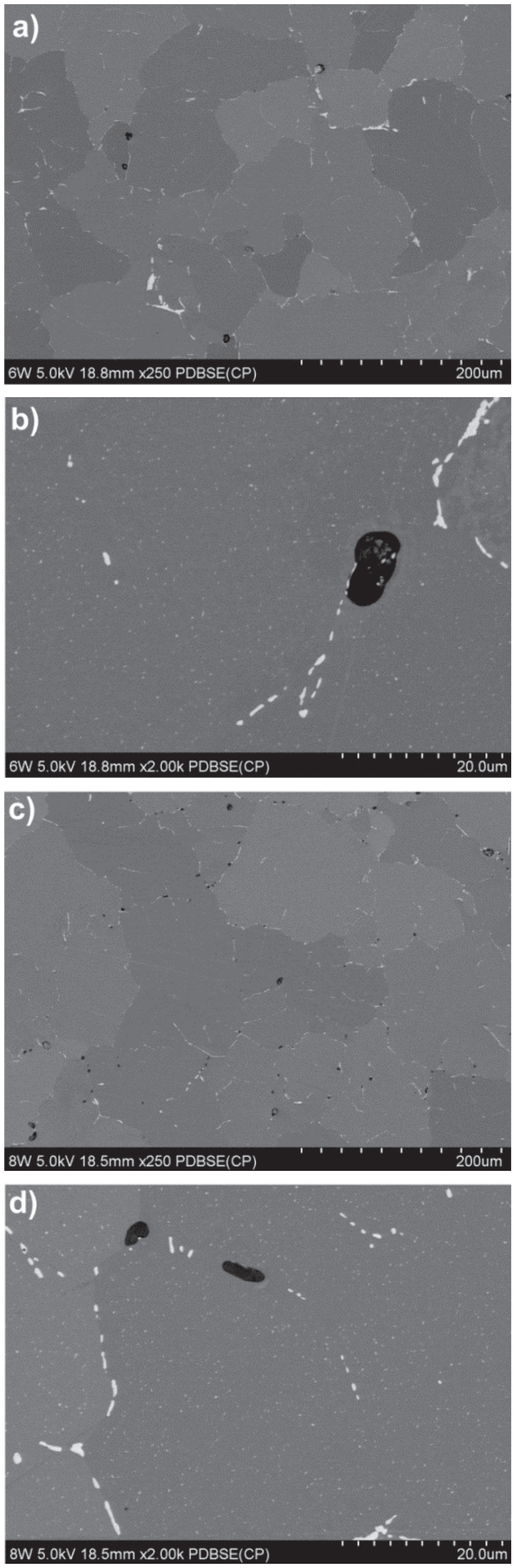

Fig. 5. Typical microstructure of the investigated alloys after homogenization and water quenching: alloy 1 homogenized at 535°C for 8h (a, b), alloy 2 homogenized in 535°C for 8h (c, d, e), alloy 2 homogenized at 550°C for 8h (f, g, h)

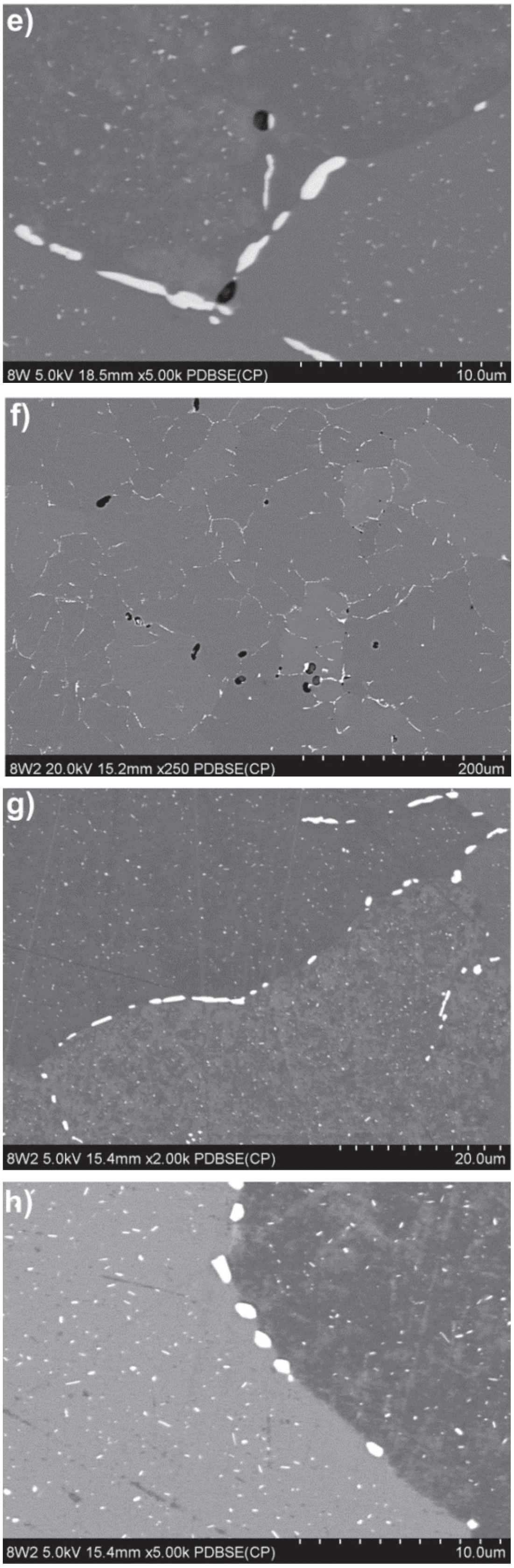

Fig. 5. (continued) Typical microstructure of the investigated alloys after homogenization and water quenching: alloy 1 homogenized at 535°C for 8h (a, b), alloy 2 homogenized in 535°C for 8h (c, d, e), alloy 2 homogenized at 550°C for 8h (f, g, h)

In comparison to as cast state, after homogenization and water quenching (Fig. 5) dissolution of significant amount of the Mg_2Si particles and defragmentation of AlFe(Mn)Si particles is observed. In the case of alloy 2, with higher Mg and Si content, after homogenization at the temperature of 535°C, the number of Mg_2Si particles is four times greater and the area is two times bigger than after homogenization at the temperature of 550°C, as is shown in Fig. 6 a, b. In the microstructure of alloy 1, despite the low homogenization temperature, the smallest amount of the Mg_2Si particles remains undissolved.

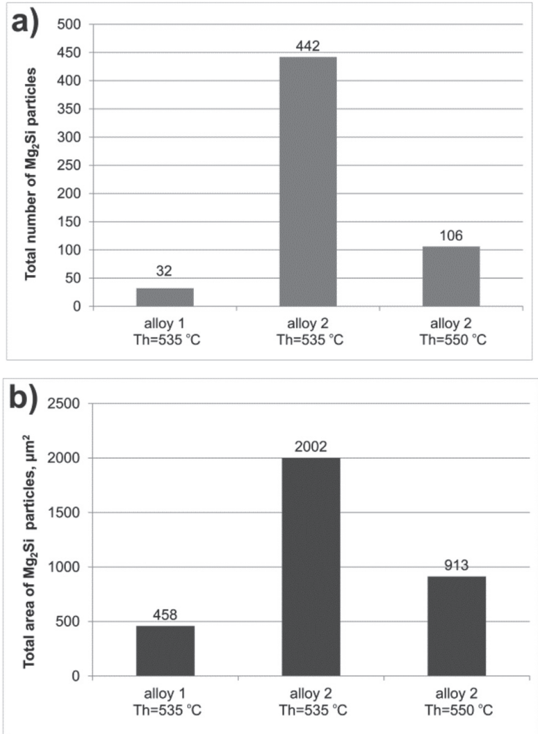

Fig. 6. Results of the quantitative microstructure analysis for alloys after homogenization and water quenching: total number (a) and area (b) of the Mg_2Si particles

Concentrations of Mg and Si in the grains interiors, for both alloys homogenized at the temperature of 535°C are similar (when values are compared with standard deviation taken into consideration). After homogenization of alloy 2 at the temperature of 550°C, the main additions concentration in grains interiors slightly increases, compared to the lower temperature annealing (Fig. 7). This observation corresponds to the described above decrease of Mg_2Si amount in the structure of alloy 2 after applying the higher homogenization temperature.

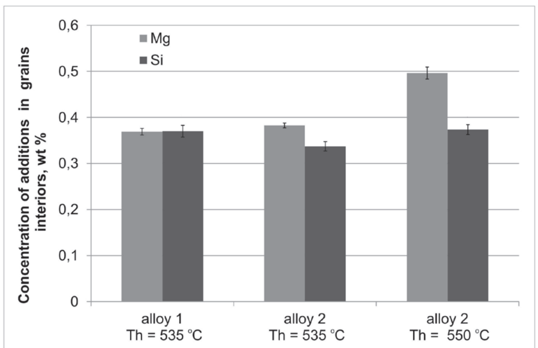

Fig. 7. Concentration of Mg and Si in the grains interiors after homogenization (average values from ten measurements in randomly selected grains)

On the DSC curve for alloy 1 after homogenization with water quenching (Fig. 8a.), incipient melting peak is no longer observed. It indicates that the mixture causing incipient melting in as-cast state was removed during homogenization. In the case of alloy 2, incipient melting peaks are found after both variants of annealing with water quenching (Fig. 8b.), but their onset temperature is about 10°C higher than in as-cast state (T 4). It should be noted that after homogenization at the temperature of 550°C, heat of incipient melting (estimated from the peak area) is over 6 times smaller than after lower-temperature homogenization. It may be therefore stated that the fraction of structure component causing melting, is essentially smaller after homogenization at the higher temperature.

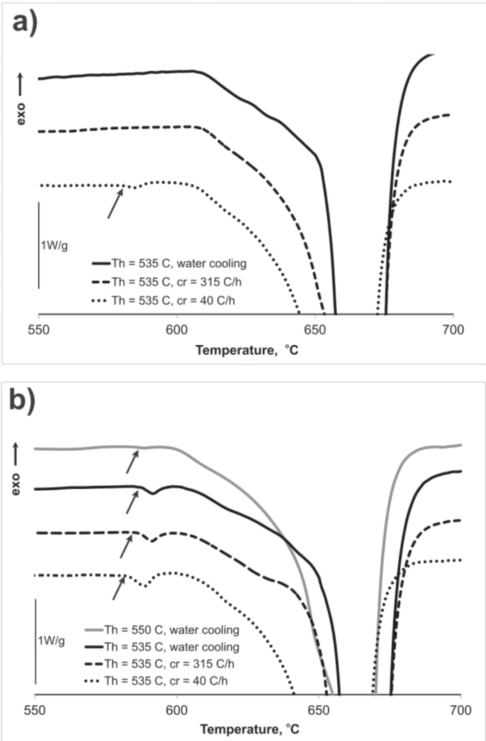

Fig. 8. DSC curves for investigated alloys after homogenization: alloy 1 (a), alloy 2 (b)

The high concentration of Mg and Si in the grains interiors, small amount of undissolved Mg_2Si particles and removal of incipient melting peak noted for alloy 1 allow to state that soaking at the temperature of 535°C for 8h is sufficient for this material. In the case of alloy 2, comparison of parallel results obtained for the two annealing variants indicates that homogenization at the temperature of 550°C for 8h is more beneficial than at the temperature of 535°C. Due to improved dissolution of Mg_2Si particles, it should allow to take advantage of higher Mg and Si content in the alloy. However, as it is shown in the Fig. 5 e and h, after homogenization at the higher temperature, tendency to growth of dispersoids is found. As it was mentioned earlier on the basis of literature data [5, 6], recrystallization resistance of the material is then lowered. When thin-walled hollow profiles are extruded, a recrystallization may occur, mainly in welds regions, which contributes to lowering of extrudates strength. For that reason, despite the benefits mentioned earlier, homogenization atthe temperature of 550°C for 8h cannot be recognized as recommended.

The influence of the cooling rate from homogenization temperature, on the structure of investigated materials is presented in Fig. 9. In the microstructure of alloys cooled at 315°C/h, on the grains boundaries a few coarse Mg_2Si particles are found. In the grains interiors, fine particles with about 0,5 μm in size are visible (Fig. 9. a, b, e, f). Based on comparison of these images with the microstructure observed after homogenization and water quenching (Fig. 5.) it may be stated that coarse Mg_2Si particles remain undissolved after soaking. The fine particles, which are observed in the grains interiors, precipitated during cooling from the homogenization temperature. Their small size justifies expectation that they will easily dissolve during subsequent reheating and extrusion process.

During cooling at 40°C/h, significant amount of main alloying additions precipitated in unfavourable form of numerous large Mg_2Si particles on the grains boundaries, although some fine particles in the grains interiors are also found (Fig. 9. c, d, g, h).

TABLE 4

DSC analysis results for alloys after homogenization

Material, homogenization conditions	Solidus temperature, °C	Incipient melting heat, J/g
alloy 1, 535°C/8h, water quenching	609	-
alloy 1, 535°C/8h, 315°C/h	608	-
alloy 1, 535°C/8h, 40°C/h	580	0,8
alloy 2, 550°C/8h, water quenching	583	0,2
alloy 2, 535°C/8h, water quenching	586	1,3
alloy 2, 535°C/8h, 315°C/h	585	1,5
alloy 2, 535°C/8h, 40°C/h	583	2,4

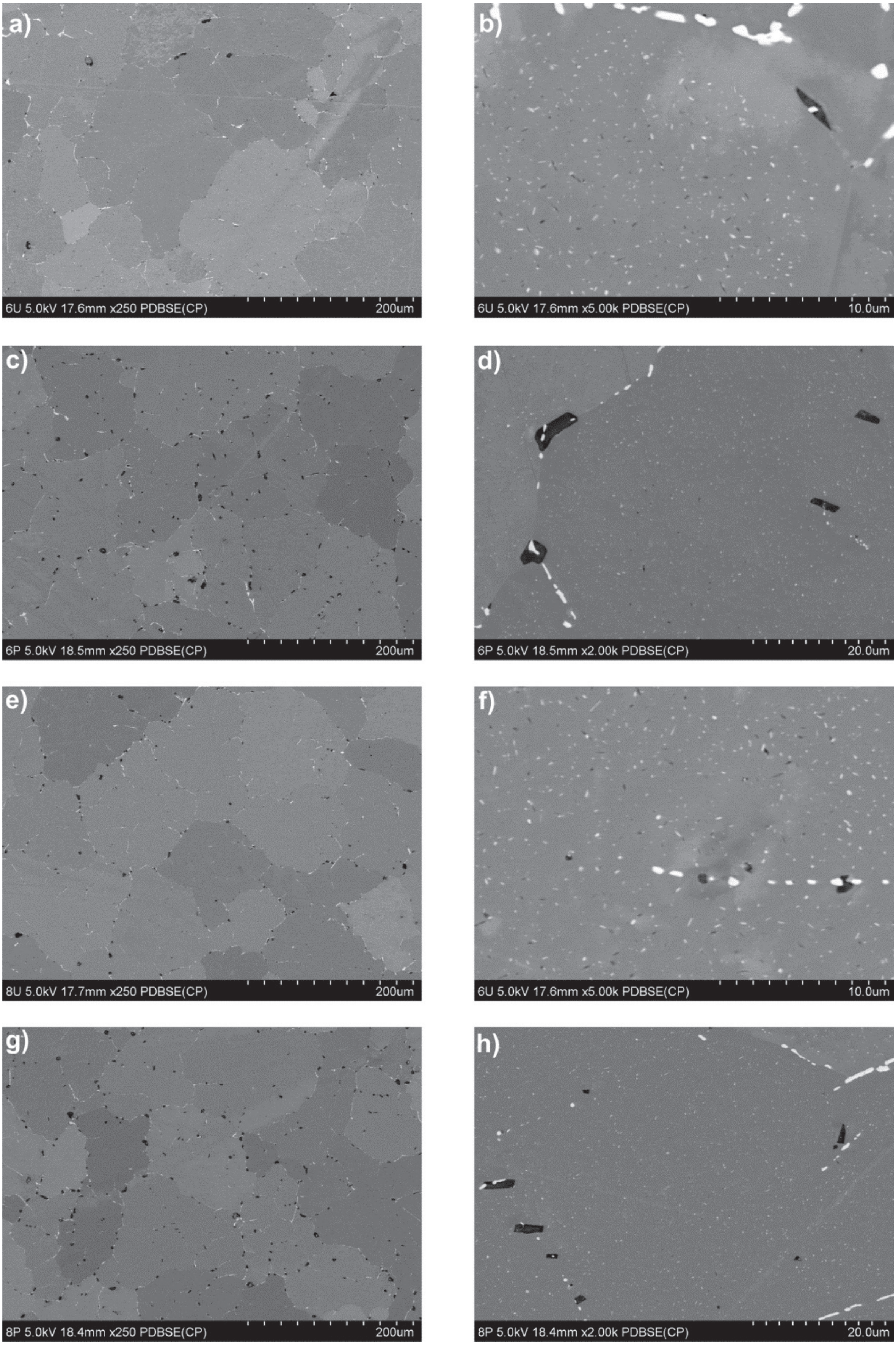

Fig. 9. Microstructure of investigated alloys after homogenization at the temperature 535°C for 8h: alloy 1 cooling rate 315°C/h (a, b), alloy 1 cooling rate 40°C/h (c, d), alloy 2 cooling rate 315°C/h (e, f), alloy 2 cooling rate 40°C/h (g, h).

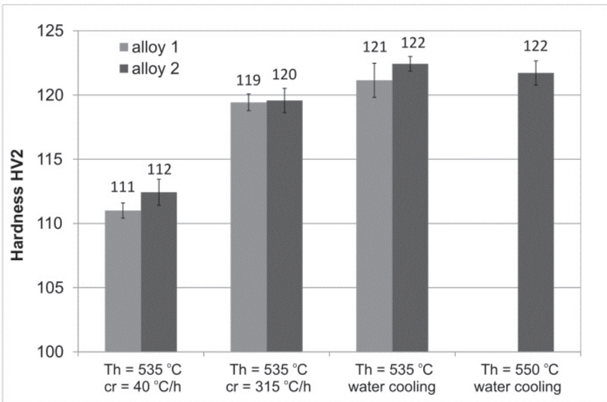

Fig. 10. Hardness of the investigated alloys after solution heat treatment (540°C/5 min.) and ageing (175°C/8h)

The ability of Mg_2Si particles, precipitated during homogenization cooling, for rapid dissolution was estimated by hardness values of solution heat treated (with short annealing) and aged samples. Final hardness obtained for samples quenched from homogenization temperature and cooled at 315°C/h is very similar: 121 and 119 HV for alloy 1, 122 and 120 HV for alloy 2, whereas hardness of the samples cooled from homogenization temperature at 40°C/h is lower by about 10 HV (Fig. 10.). It results from the difference in dissolution of Mg2Si particles during solution heat treatment: it is nearly full in the case of homogenization cooling at 315°C/h and only partial for the cooling at 40°C/h.

This explanation is confirmed by the results of DSC test with rapid heating, performed on the samples after different homogenization cooling variants. For the alloy 1, after homogenization with water quenching and cooling at 315°C/h, no incipient melting is observed, whereas after cooling at 40°C/h incipient melting peak with onset at the temperature of 580°C occurs. In the case of alloy 2, incipient melting at the temperature of about 585°C is observed after all homogenization variants. It should be noted that after homogenization at the temperature of 535°C for 8h with water quenching and cooling at 315°C/h, incipient melting heat is similar, about 1,5 J/g, whereas after the slowest cooling it is considerably greater: 2,4 J/g (Fig. 8. and TABLE 4). On the basis of the literature data mentioned earlier [7, 9, 14], it may be explained as follows: large Mg_2Si particles, present in the billets structure after slow homogenization cooling, do not fully dissolve during rapid heating and cause local enrichment of the surrounding matrix with Mg and Si. This causes appearance of unequilibrium melting reaction for alloy 1 and an increase of content of melting reaction components for alloy 2.

It should be stressed, that the hardness of both investigated alloys, after the homogenization at the temperature of 535°C, subsequent solution heat treatment and ageing is very similar. Moreover, the application of higher homogenization temperature for alloy 2, also has no influence on the final hardness, despite the described above improved dissolution of Mg2Si particles during soaking. This result allows to confirm the earlier statement that homogenization of this alloy at the temperature of 550°C for 8h is unnecessary.

4. Conclusions

On the basis of investigation results presented above, the following conclusions can be drawn:

1. Homogenization at the temperature of 535°C for 8h can be recommended for both investigated 6082 grade alloys. In the case of alloy 1 with lower Mg and Si content it is sufficient for significant Mg_2Si particles dissolution. For alloy 2 with higher Mg and Si content, after homogenization at the temperature of 550°C for 8h, the amount of undissolved Mg_2Si particles decreases, compared to homogenization at 535°C for 8h. However, an unfavourable tendency of dispersoids growth is observed and these soaking conditions are not found to be appropriate.
2. Cooling rate after homogenization at 315°C/h is sufficient for precipitation of fine Mg_2Si particles, whereas cooling at 40°C/h causes precipitation of Mg_2Si phase in the form of large particles on the grains boundaries.
3. The fine Mg_2Si particles, precipitated during cooling after homogenization at 315°C/h, dissolve during subsequent rapid heating. This is concluded on the basis of the high hardness of alloys cooled in this manner, subjected to solution heat treatment with short soaking (540°C/5 min.) and ageing. It can be expected, that in the case of application of this cooling rate in industrial conditions, the particles dissolution will also take place during the subsequent billets reheating and extrusion.
4. The large Mg_2Si particles, present in the microstructure of investigated alloys after homogenization cooling at 40°C/h, do not fully dissolve during subsequent rapid heating. It is confirmed by lower hardness after solution heat treatment and ageing as well as DSC test results, showing incipient melting in the case of alloy 1 and increase of melting heat for alloy 2, after this kind of homogenization cooling.

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