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Following the precipitation process during homogenization by correlative microscopy in aluminium manganese alloy

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Abstract. The correlative microscopy provides an opportunity for combining two visual impressions based on different principles. By this method, the taking of optical microscopic and electron microscopic images become possible about the same experimental area in an appropriate hardware-software environment.
In the aluminium-manganese alloys, precipitation form during homogenization which is one of the processes in the rolling technology sequence. The features, morphology and composition of developing precipitation depend on the homogenisation temperature and time, these characteristics change within wide limits.
In this study, the precipitates forming during homogenization were examined by correlative method. In 3003 manganese alloy, the appearance of particles at different homogenization temperature and time was followed. The microstructure of sample before and after homogenization was investigated, the phases were identified in the microstructure and the formation processes were followed.

1. Introduction

In the 3xxx aluminium alloys, the main alloying element is Mn with about 1w% (3003), besides it can contain maximum 0.6% Si, maximum 0.2 % Cu and maximum 0.7% Fe. The manganese in dissolved state or in different intermetallics/precipitates has a strong effect on formability; the formed phases have an influence on the dynamic recrystallization during hot rolling and recovery process after cold rolling and subsequent annealing. The nature, the composition, the morphology and the dispersion of precipitates can be changed in wide range by changing the homogenization time and temperature. In the manganese aluminium alloy, intermetallic phases appear as e.g. Al_6Mn, ($Al_6(Mn,Fe)$ as a function of the cooling rate during casting. Depending on the time and temperature of the homogenization, other processes can also take place in the course of the homogenization process, such as the destabilization of non-equilibrium phases or the spheroidization of equilibrium phases. The orthorhombic $Al_6(Fe,Mn)$ often transforms to cubic α-(Fe,Mn)-Si („6 to α" transformation). [1, 2, 4]

The manganese precipitation then the dissolution from the aluminium solid solution starts at about 300°C in the 3003 aluminium. The eutectic in the cast microstructure mostly consists of $Al_6(Fe,Mn)$, the fragmentation of which starts at about 400°C. The $Al_6(Fe,Mn)$ phase transforms to α-Al(Fe,Mn)Si and the amount of this phase increases by increasing the temperature. The fragmentation of eutectic phases is the strongest at 560°C in addition spheroidization and coarsening can also be observed. The volume fraction of primary phases does not change significantly during homogenization, the manganese has not any effect on it but the finest secondary precipitation has. The formation of $Al_6(Fe,Mn) \rightarrow \alpha$-$Al_{12}(Fe,Mn)_3Si$ eutectoid transformation and the fine dispersed precipitation begin at the same temperature (300°C) and their kinetic is the same up to 500°C. Above 500°C, the small precipitates dissolve, while the amount of the ones having big size increase.

The average size of precipitation grows by increasing the homogenization temperature, but the homogenization has an effect on the morphology of precipitation as well. In the eutectic structure formed during the crystallization, spheroidization takes place during homogenization in addition small manganese contented precipitates also appear within the homogenised grains [4, 6, 7]. During growing of the precipitates, particle-free zones (PFZ) form in the matrix which do not contain any dispersoids. The increasing of PFZ has a beneficial effect on the subsequent formability. The size of particle-free zone grows by increasing the heating time especially in case of the alloys with high Si and Mn content which can be explained by the coarsening of fine disperse and primary big size particles [3, 4].

As the character, composition, morphology and distribution of precipitations appearing during the homogenizing heat treatment applied in the course of the rolling technology of manganese aluminium alloys can widely change as a function of the time and temperature influencing by this the properties of final product. A method suitable for describing the structure-changes taken place during the homogenizing heat treatment and for following the progress of these changes in time was searched. The correlative microscopy seemed to be suitable for this purpose. Various heat treatments were performed at different homogenization temperatures and times by using 3003 manganese aluminium alloy. The formed microstructure of homogenized samples was investigated by a correlative microscopic method.

2. Experimental

2.1. Correlative microscopy

In correlative microscopy, the possibilities offered by visual impressions operating at different principles are combined. As the visual imaging is always based on the information obtained from the interaction between the sample and the radiating source, therefore light contrast is used in optical microscopy, while material contrast is applied in electron microscopy for imaging. By applying correlative microscopy, optical and electron microscopic images can be made sequentially in a special hardware and software environment, which is free from the anomalies of mapping techniques.

The Zeiss method uses a special sample holder and software to connect the two investigation methods (Figure 1.). The sample holder has a frame which can be attached both to the optical microscope and to the electron microscope as well.

The three markers placed at certain distances on the frame help recording the accurate measuring coordinates. During calibration, the coordinates of markers are recorded in the software. After calibration, the actual coordinates are recorded in each case near the image taken of the investigated area of sample. After completing the heat treatment or other treatment result a structural change, the sample is returned to the holder and the software finds the examined area on the basis of the recorded coordinates.

Figure 1. Zeiss sample holder with markers for correlative microscopy

The method can be applied for (1) the determination of morphology and composition on a given area by two different methods (optical and electron microscopic) and for (2) observing in high magnification the process taken place on the given area.
In the course of our research work, the results of processes taken place during homogenization were followed by using the electron microscopic method based on the correlative microscopy method.

2.2. Examinations
The experiments were performed on 3003 aluminium alloy. The composition of alloy can be seen in Table 1.

Table 1. Composition of 3003 aluminium alloy [w%]

	Al	Mn	Cu	Fe	Si	Zn	Residuals
3003	Balanced	1.0-1.5	0.05-0.20	max 0.7	max 0.6	max 0.1	max 0.15

Polished samples were used for the measurements. The investigations were performed by using Zeiss EVO MA 10 scanning electron microscope in the Institute of Physical Metallurgy, Metalforming and Nanotechnology, University of Miskolc. In case of each sample, the images were taken in by a magnification of 500 and 1000. Following the heat treatment, the phases developed on the areas investigated earlier could be observed on the basis of the recorded and saved coordinates. The homogenization heat treatment was performed at 500 and 600°C for 1, 5 and 10 hours period. In this study, the results obtained during the homogenization process of max. 10 hours are presented.

3. Results and Discussion

Due to the resolution limit, optical microscopy is not suitable for observing small precipitates with a size of few hundred nanometre developing during the homogenization process. In case of the sample homogenized at 500°C for 10 hours, the information content of images taken of the investigated area by optical and electron microscopy method is clearly visible on **Figure 2**. The small particles cannot be seen on the image taken by optical microscope. Since the purpose of our study is to observe the precipitates forming during homogenization, the changes will only be followed in the electron microscopy images.

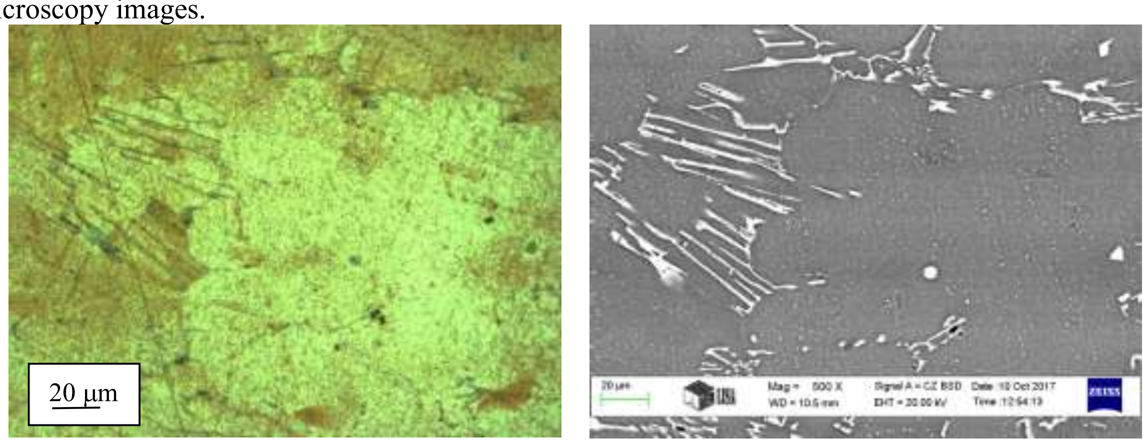

Figure 2. Optical microscopic (left) and electron microscopic (right) images; Sample 500°C 10h

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3.1. Results of homogenization

The microstructure of a homogenized 3003 aluminium sample is shown in **Figure 3**.

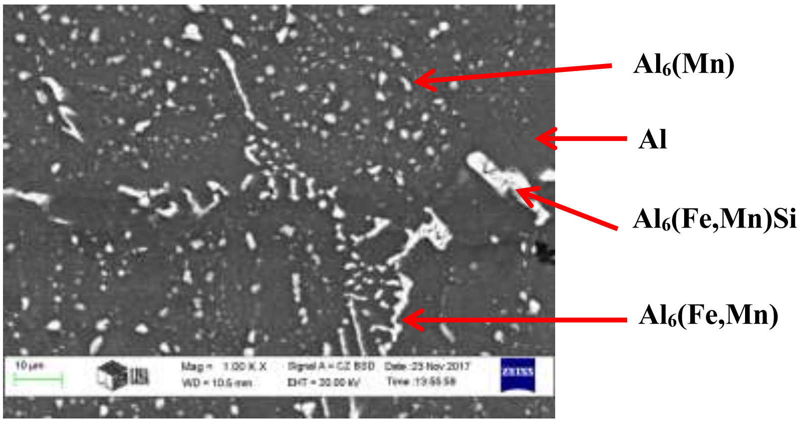
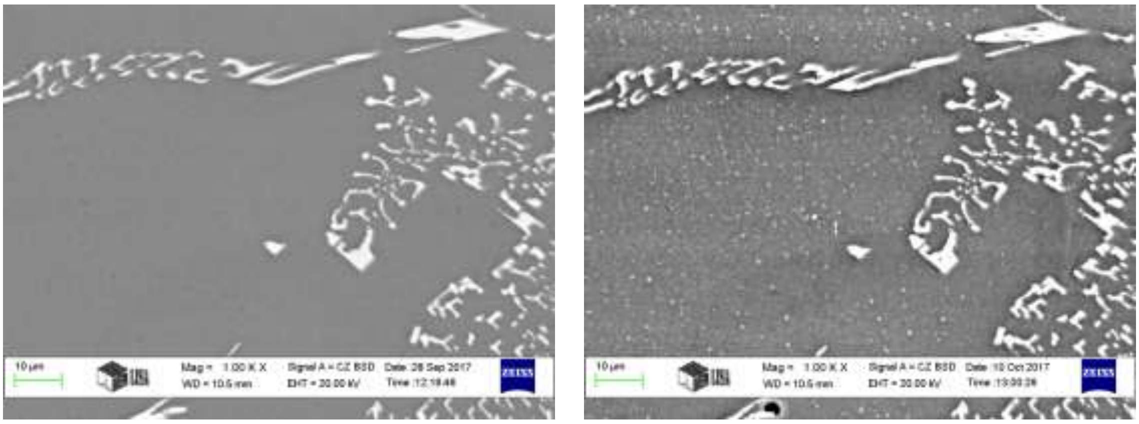


Figure 3. The microstructure of homogenized 3003 aluminium alloy

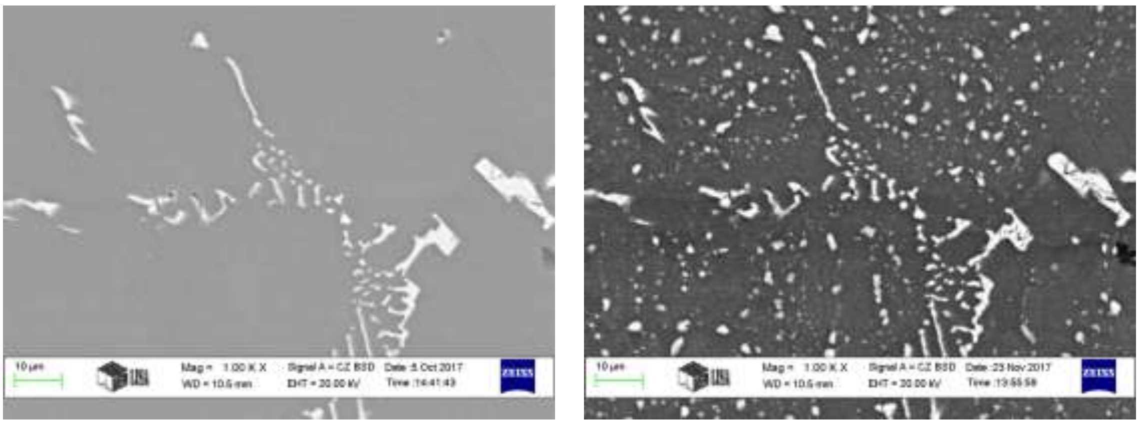
In the 3003 alloy, the phases having a larger size similar to the light colour Chinese characters are $Al_6(FeMn)$ primary intermetallics. The small particles appearing in aluminium solid solutions are Al_6Mn precipitates. The amount of precipitates increases as a function of the homogenization time (Figure 4.).



cast homogenised

Figure 4. Microstructure of sample after homogenisation (500°C, 10h)

The precipitates are coarsening by increasing the homogenization temperature (Figure 5.).



cast homogenised

Figure 5. Microstructure of Sample after homogenisation (600°C 10h)

3.2. Precipitates in the homogenised samples
High magnification images were performed for characterising the precipitation. In case of the sample homogenised at 500°C, many small particles of a size of about 200-300 nm and some larger particles with a size of about 0.7-0.8 μm next to the small particles can be observed. The distribution of small dispersed phases is uniform and it surrounds the previously formed particles having a larger size. (Figure 6.).

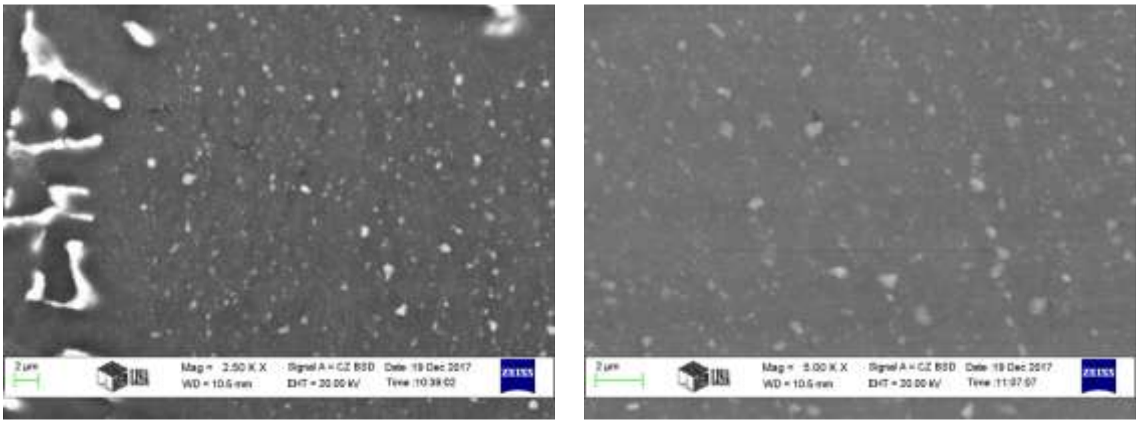


Figure 6. Precipitates in high magnification, Sample 500°C 10h

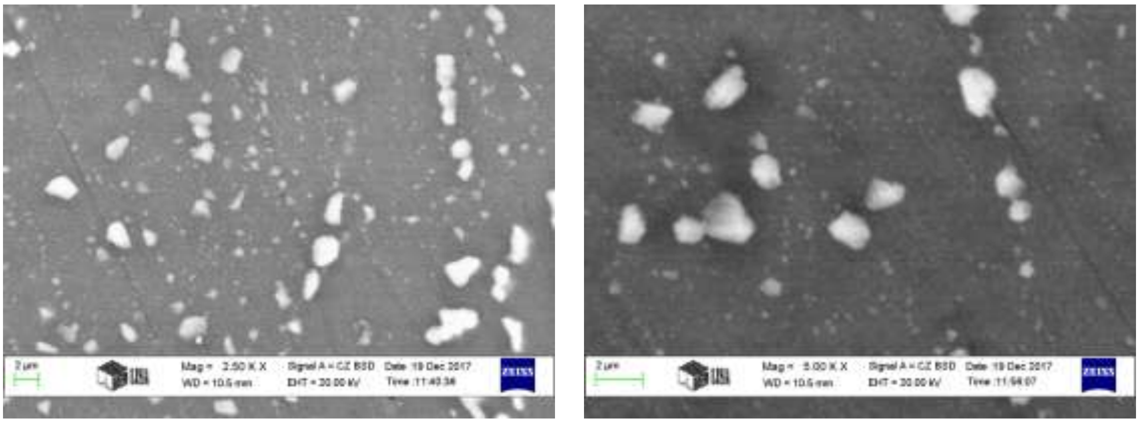


Figure 7. Precipitates in high magnification, Sample 600°C 10h

In the sample homogenised at 600°C, small precipitations of a size of about ~100-200 nm can be seen in an uneven distribution around the larger spherical particle of a size of ~1.5-2 μm but grains falling between the two sizes can hardly be observed. The signs of coarsening can obviously be seen in the microstructure: the bigger particles are grown, while the smaller precipitates are dissolved and the primary phases start to solve; it is shown by the change of curvature and spheroidization of the phase boundary surface. The presence of tiny particles shows the traces of precipitation process which proceeds parallel with the coarsening, but the coarsening process is dominant (Figure 7.).
The particle diameters measured in different areas in the homogenised samples are shown in Table 2.

Table 2. Particle diameters in the homogenised samples

Sample	**Examined area**	**Particle diameter [μm]**	**Average particle diameter [μm]**
500°C 10h	Field of view No. 1	0.826	0.890
		0.768	
	Field of view No. 2	0.945	
		0.903	
	Field of view No. 3	1.049	
		1.104	
	Field of view No. 4	0.744	
		0.784	
600°C 10h	Field of view No. 1	2.574	2.380
		2.727	
	Field of view No. 2	2.694	
		1.971	
	Field of view No. 3	2.437	
		2.916	
	Field of view No. 4	2.020	
		1.703	

4. Summary

It can be stated that the correlative microscopy is suitable for following and studying the processes taken place in aluminium alloys during homogenization. The correlative microscopy method provides an opportunity for detecting the precipitation, coarsening and dissolving processes too.

In the 3003 alloy, the precipitates appear at a homogenization temperature of 500°C, the number of particles increase by increasing the time of homogenization. The coarsening of precipitates starts at a homogenization temperature of 600°C, the size of particles increases by increasing the time of homogenization.

Acknowledgement

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