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The Influence of Mg/Si Ratio on Phase Compositions in 2014 Aluminium Alloys After Casting and Heat Treatment.

Marzena Lech-Grega
The Institute of Non-Ferrous Metals, Light Metals Division; Pilsudskiego 19, 32-050 Skawina, Poland

Al-Mg-Si-Cu systems are not considered by Aluminium Association and Polish Standard PN-EN 573-3 a separate group of alloys but are formed by adding silicon to Al-Cu-Mg alloys from 2xxx series (e.g. 2014 with Si= 0,5-1,2%).
In the case of ternary Al-Mg-Si systems, the phases present there are Al, Mg2Si (β) and Si. The addition of copper introduced to alloy results in the formation of additional Al_2Cu (θ) phases and of the Q phase (described as Al5Cu2Mg8Si or Al4CuMg5Si4 or Al4Cu2Mg8Si7 or Al3Cu2Mg9Si7). What matters here is the Mg/Si ratio. If Mg/Si>1, the phases that occur are Al, Q, θ and β.
When Mg/Si<1, the alloy structure holds Al, Q, θ and Si.
The fraction of the intermetallic phases increases in the alloy when the presence of Fe and Mn is additionally allowed for.
The aim of the investigations was to characterise the intermetallic Al-Mg-Si-Cu and Al-Fe-Si-Mn phases present in commercial 2014 aluminium alloys in as-cast state and after heat treatment, allowing for the Mg/Si ratio (0.2, 0.4, 1.0 and 1,7) and solidification rate (from 0.2 to 0.8^0/sec.). The analysis was made using the results of examinations carried out under an optical microscope, and performed by SEM with EDX and EBSD, and by XRD. The material produced in laboratory was compared with billets made by industry.

Keywords: *Aluminium alloys, 2014 aluminium alloy, AlCuSiMg alloy, intermetalic phases, EBSD analysis*

1. Introduction

Aluminium alloys from 2xxx series and an AlCuMg system form the group of most popular heat-treated materials assigned for plastic forming. Their popularity they owe to high formability, relatively high mechanical properties, and easy application of anodic coatings of both decorative-protective and technical character. They are widely used by the automotive and building industry, and are precipitation hardened with Al_2Cu (Θ) and Al_2CuMg (S) phases, while their mechanical properties can be adjusted by control of the formed precipitates.
Added to 2xxx series alloys, silicon results in the formation of an additional Mg_2Si (β) phase and the Q phase described as a hexagonal $Al_3Cu_2Mg_9Si_7$ phase [1-3].
Alloys used in industry have a natural addition of up to 0,7% Fe and 0,4-1,2% Mn, forming with Si various α-AlFeSi phases of a regular structure ($Al_{12}Fe_3Si$, $Al_{15}Fe_3Si_2$, $Al_{12}(FeMn)_3Si$, $Al_{15}(FeMn)_3Si_2$, $Al_{12}Mn_3Si$, $Al_{15}Mn_3Si_2$, Al_8Fe_2Si,). Depending on alloy chemical composition, these phases either assume a more coagulated form of the "Chinese script" in as-cast state, or develop the form of dispersoids after a homogenising treatment. Another phase present in the system is a β-AlFeSi phase, which is a monoclinic Al_5FeSi phase present in the form of large lamellae. The β-AlFeSi phase occurs in cast ingots; its presence is highly undesirable when the alloy is assigned for further plastic forming [4-5].
The intermetallic phases in model materials have been described in every detail. However, the conditions of ingot solidification in industrial plants differ quite considerably from laboratory conditions. The result is the formation of different phases depending on the solidification rate (the distance from ingot surface). The consequence is the presence of different intermetallic phases after the homogenising treatment, and hence different formability of alloy assigned for plastic working.

2. Experimental procedure

The test material was 2014A aluminium alloy with different content of silicon (Si =0,5 and 1,0%) and magnesium (Mg = 0,2 and 0,8%), to make the Mg/Si ratio amount to 0.2, 0.4, 0.8, and 1,5, respectively. Other alloying elements were similar (Table 1). The chemical compositions of these alloys were comprised in a range developed for 2014A alloy by the PN-EN 573-3.

Table 1 Chemical composition of the laboratory samples of 2014A aluminium alloys.

Sample	Cooling rate	Si	Fe	Cu	Mn	Mg	Ni	Zn	Zr+Ti	Al	Mg/Si
	[K/sek]	[wt%]									
1.1	20	0,49	0,36	5,71	0,57	0,20	0,020	0,032	0,11	balance	0,4
1.4	0,5										
2.1	20	0,52	0,42	6,13	0,57	0,76	0,020	0,032	0,11	balance	1,5
2.4	0,5										
3.1	20	0,98	0,43	6,10	0,58	0,19	0,022	0,035	0,11	balance	0,2
3.4	0,5										
4.1	20	0,97	0,45	5,76	0,56	0,78	0,022	0,037	0,11	balance	0,80
4.4	0,5										

The investigated alloy of predetermined chemical composition was cast in a metal mould, which provided different cooling rates. Samples cooled at a rate of 20 K/sec and 0,5 K/sec were finally chosen for the tests.
The homogenising treatment of selected samples assigned for laboratory tests was carried out at a temperature of 500°C for 8 hours.
The reference material was taken from an ingot of φ 330 mm, cast under the industrial conditions and characterised by the chemical composition given in Table 2. Samples for tests were cut out from the ingot edge (B) and centre (S), using ingots in as-cast state (C) and after the homogenising treatment (H). Under industrial conditions, ingots were homogenised at a temperature of 490°C for 12 hours.

Table 2 The chemical composition of samples taken from an 2014 aluminium alloy ingot.

Sample	Si	Fe	Cu	Mn	Mg	Ni	Zn	Zr+Ti	Al	Mg/Si
	[wt%]									
Ingot 2014A	0,85	0,18	5,4	0,78	0,84	0,007	0,051	0,022	balance	1

On thus prepared samples, the metallographic examinations were carried out under an OLYMPUS GX71 light microscope and a PHILIPS XL30 scanning electron microscope with chemical analysis in microareas by EDX. On selected samples, the phase analysis was made by EBSD technique to enable crystallographic identification of phases present in the examined alloys.
For LM and SEM observations, the metallographic samples were prepared in a standard mode by grinding and mechanical polishing. Additionally, for EBSD examinations, the samples were electropolished.

3. Results and discussion

In as-cast laboratory samples, four types of the precipitates were found, namely:

- AlCu copper phases, often containing Mg (about 0,2wt.%) and Si (about 0,2-0,3wt.%).

In samples where the Mg/Si ratio was 0,2 and 0,4 (samples nos. 3 and 1, respectively), the AlCu phases were present in the form of blocks with a variable Cu content (Fig.1a.). In samples with the

Mg/Si ratio equal to 0,8 and 1,5 (samples 2 and 4), the copper phases usually appeared as complex eutectic phases with Mg_2Si (Fig.1b).
The EBSD analysis has revealed that, irrespective of the chemical composition, in terms of crystallography, the copper phase was described an Al_2Cu phase (Fig. 2a).

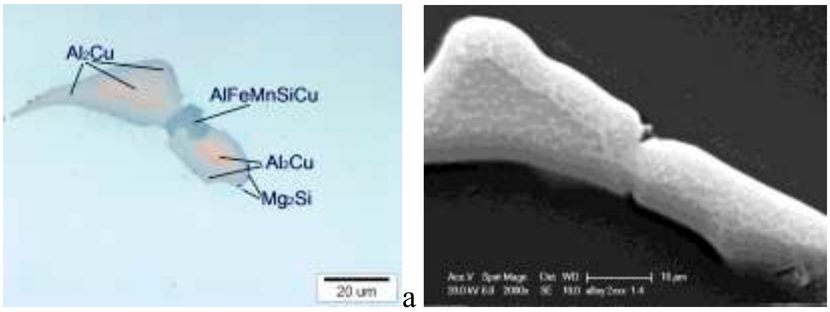
- AlFeMnSiCu iron-manganese phases containing silicon and copper.

Irrespective of the chemical composition in laboratory prepared alloys, iron phases had the form of „Chinese script" and were enriched with copper in an amount of about 8-10wt.% and with magnesium in an amount of about 0,5wt.% .
Depending on cooling rate, the Fe+Mn/Si ratio differed and amounted to about 1,5–3 for the cooling rate of 20K/sec and to about 4,5–5 for the cooling rate of 0,5K/sec. The Fe+Mn/Si ratio also depended on silicon content in the examined alloy.
Irrespective of the chemical composition, in terms of crystallography, the iron phase was described as an $Al_{4,01}MnSi_{0,74}$ phase (Fig. 2b).

- MgSi magnesium-silicon phases

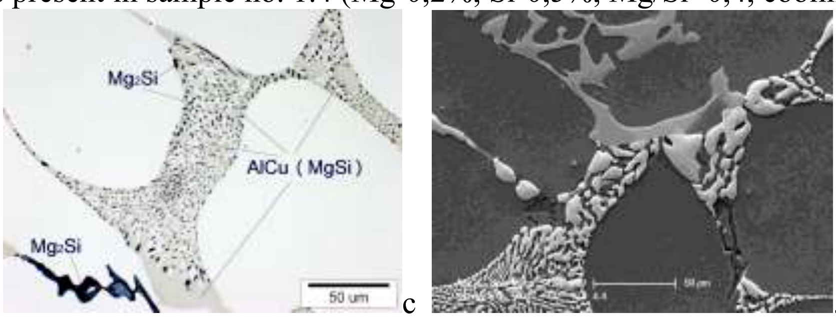
In samples where the Mg/Si ratio was 0,2 and 0,4 (samples nos. 3 and 1, respectively), the MgSi phases were very scarce in occurrence (they did not occur in sample no. 3 with the cooling rate of 20K/sec). In samples with the Mg/Si ratio equal to 0,8 and 1,5 (samples nos. 4 and 2, respectively), the MgSi phases were present as complex eutectic phases containing AlCu and also as large precipitates located at grain boundaries (Fig. 1b).

- Precipitates of primary silicon in sample no. 3 with an excess content of silicon (Mg - 0,2%, Si – 1%).



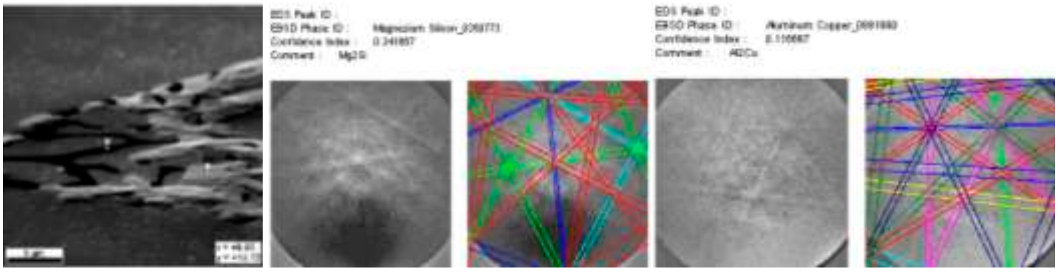
a b

1a. AlCu phases present in sample no. 1.4 (Mg-0,2%, Si-0,5%, Mg/Si=0,4, cooling rate of 0,5K/s).



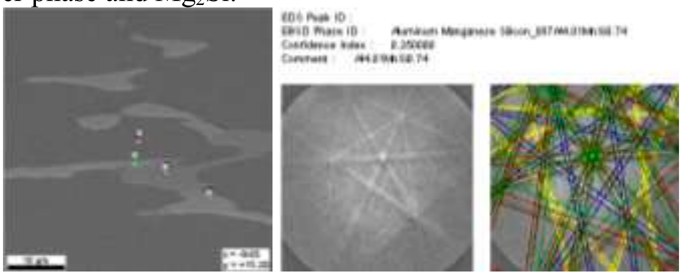
c .d

1b. AlCu phases present in sample no. 4.4 (Mg-0,8%, Si-1%, Mg/Si=8, cooling rate 0,5K/s).
Fig. 1. The structure of precipitates in as-cast state.



a

a. The analysis of copper phase and Mg_2Si.



b

b. The analysis of iron phase.

Fig. 2 The EBSD analysis of intermetallic phases.

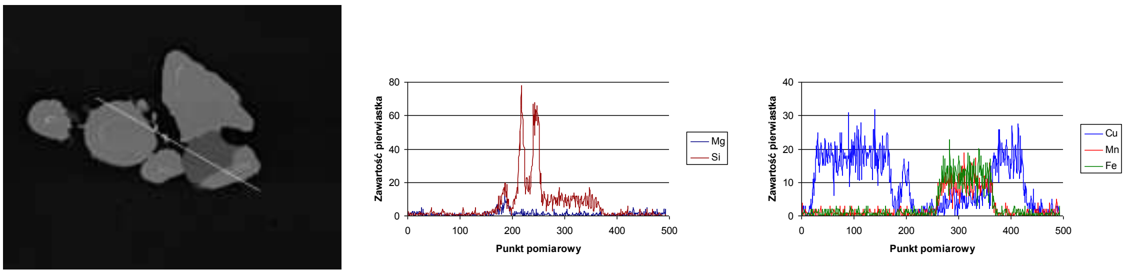


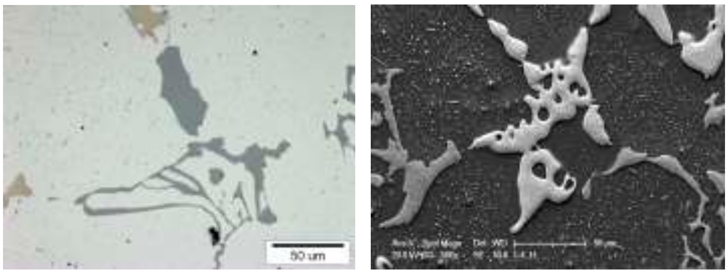
Fig. 3 The linear analysis of chemical composition running through the precipitates of copper, iron, Mg_2Si and Si in as-cast sample no. 3.

The homogenising treatment resulted in the presence of fine-dispersed Al_2Cu phases precipitating from solution and in a low-degree precipitates coagulation. Around the large precipitates, an area free from the fine-dispersed precipitates occurred (Figs. 4 and 5). Moreover:

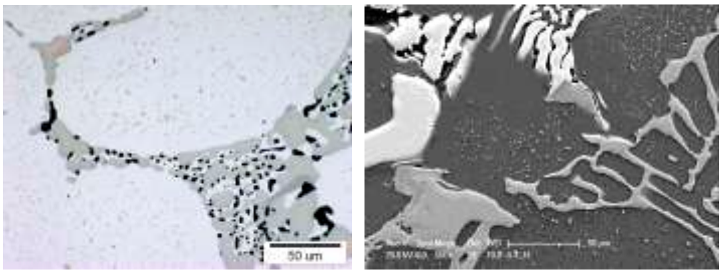
- in copper phases a compensation of the chemical composition occurred,
- in iron phases the content of magnesium decreased to even 0,2wt.%,
- the MgSi magnesium-silicon phases have undergone a coagulation process.

The structure of the precipitates in an ingot made from 2014 alloy should be, on account of the chemical composition, similar to the precipitates observed in laboratory sample no. 4.
It has been noted that in samples taken from an ingot, irrespective of the cooling rate, the precipitates of Mg_2Si occurred only on grain boundaries and not in the form of a copper phase-containing eutectic. The homogenising treatment has resulted in a coagulation of the copper eutectic, combined with precipitation (inside the grains) of fine-dispersed Al_2Cu phases. On the grain boundaries of copper precipitates, the Mg_2Si phases precipitated and coagulated.

In laboratory melts, the mechanical properties (R_m, $R_{p0,2}$) differed considerably in function of the chemical composition. The lowest properties (R_m =180MPa in as-cast state and 90MPA after homogenising treatment) were found in the alloy in which the Mg/Si ratio was 1,5, the highest (R_m =220MPa in as-cast state and 170MPA after homogenising treatment) offered the alloy in which the Mg/Si ratio was 0,2.



a. The intermetallic phases in sample no. 1.4 (Mg-0,2%, Si-0,5%, Mg/Si=0,4, cooling rate of 0,5K/s).



b. The intermetallic phases in sample no. 4.4 (Mg-0,8%, Si-1%, Mg/Si=8, cooling rate of 0,5K/s).

Fig. 4. The structure of precipitates after homogenising treatment.

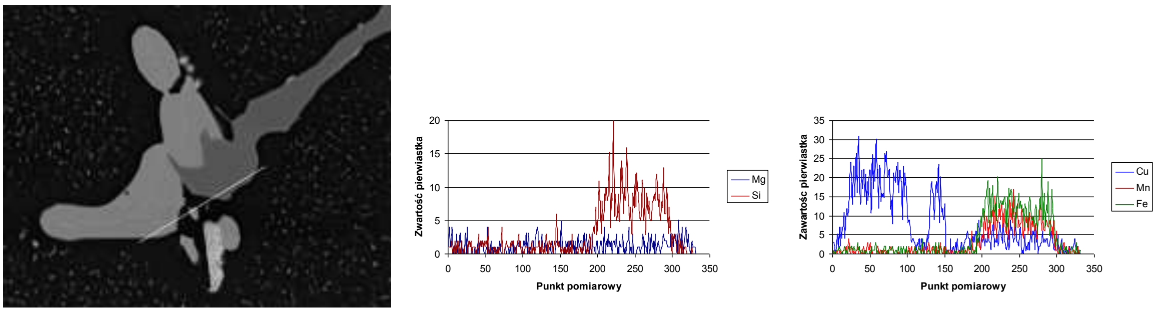
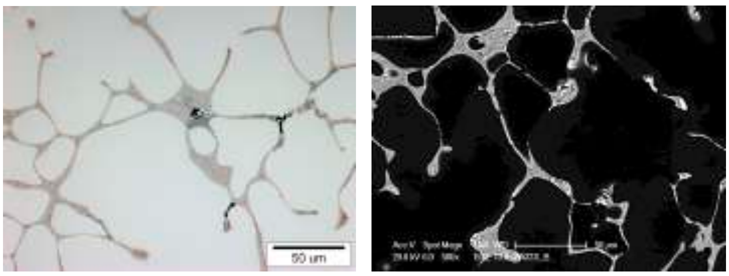
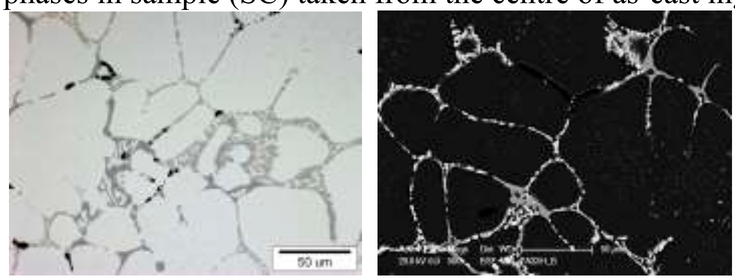


Fig. 5. The linear analysis of chemical composition running through the precipitates of copper and iron in sample no. 3 after homogenising treatment.



a. The intermetallic phases in sample (SC) taken from the centre of as-cast ingot.



b. The intermetallic phases in sample (SH) taken from the centre of ingot after homogenisation.

Fig 6. The structure of precipitates in ingot as-cast and after homogenising treatment.

4. Conclusions

1. In the family of 2014A alloys, it is possible to combine different chemical compositions with variable Mg/Si ratios and control in this way the alloy structure and mechanical properties.
2. In alloys manufactured under laboratory conditions, copper phases with additions of Si and Mg, iron-manganese-silicon phases with additions of Cu and Mg, Mg_2Si phase and primary silicon were present.
3. The content of these phases, and their chemical composition and morphology depended on the value of the Mg/Si ratio and on the cooling rate.
4. The crystallographic copper phases (EBSD analysis) were described as Al_2Cu phases, while iron phases were described as $Al_{4,01}MnSi_{0,74}$ phases. This statement has been confirmed by XRD examinations.
5. In laboratory samples, besides coagulation, the homogenising treatment resulted in a homogenisation of the chemical composition of the precipitates. The crystallographic structure remained unchanged.
6. The structure of the precipitates in ingots with Mg/Si=1 was similar to the structure observed in laboratory samples.

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