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INVESTIGATION OF SOLIDIFICATION PROCESS AND MICROSTRUCTURE EXAMINATION OF AlCu4Ni2Mg2 ALUMINIUM ALLOY

Grażyna Mrówka-Nowotnik, Małgorzata Wierzbińska, Jan Sieniawski, Andrzej Nowotnik

S u m m a r y

The main objective of this work was to examine a solidification process of AlCu4Ni2Mg2 alloy and analyze morphology and composition of the complex microstructure of intermetallic phases in as-cast condition. To study the solidification process differential scanning calorimetry (DSC) was used. To identify intermetallics in AlCu4Ni2Mg2 alloy optical light microscopy (LM), X-ray diffraction (XRD), scanning (SEM) and transmission (TEM) electron microscope and were used. The results show that the as-cast microstructure of AlCu4Ni2Mg2 alloy after slow solidification at a cooling rate 5°/min, consisted: dendrites of α-Al and intermetallic phases Al_7Cu_4Ni, Al_6Fe, S-Al_2CuMg, and $Al_3(CuFeNi)_2$ and Al_2Cu.

Keywords: aluminium alloys, solidification, microstructure, intermetallic phases

Analiza procesu krystalizacji i kształtowania mikrostruktury stopu aluminium AlCu4Ni2Mg2

S t r e s z c z e n i e

Prowadzono analizę procesu krystalizacji oraz składu chemicznego i morfologii składników fazowych mikrostruktury stopu AlCu4Ni2Mg2 w stanie lanym. W analizie procesu krystalizacji stopu stosowano metodę różnicowej kalorymetrii skaningowej (DSC). Identyfikację składników fazowych mikrostruktury stopu AlCu4Ni2Mg2 prowadzono metodami mikroskopii świetlnej (LM), elektronowej: skaningowej (SEM) i transmisyjnej (TEM) oraz dyfrakcji rentgenowskiej (XRD). Analiza uzyskanych wyników badań pozwoliła ustalić, że mikrostruktura stopu AlCu4Ni2Mg2, kształtowana w procesie krystalizacji z małą prędkością chłodzenia 5°C/min, składa się z dendrytów roztworu stałego α-Al oraz wydzieleń faz międzymetalicznych: Al_7Cu_4Ni, Al_6Fe, S-Al_2CuMg, $Al_3(CuFeNi)_2$ i Al_2Cu.

Słowa kluczowe: stopy aluminium, krystalizacja

1. Introduction

Al-Cu-Ni-Mg aluminium alloys due to their excellent properties have a widespread application, especially in the aircraft, marine structures and automotive industry [1-4]. The main alloyin elements – Cu, Ni and Mg, partly dissolve in the primary α-Al matrix, and to some amount present in the form of intermetallic phases. A range of different intermetallic phases may form during

Address: Prof. Jan SIENIAWSKI, Grażyna MRÓWKA-NOWOTNIK, DSc Eng., Małgorzata WIERZBIŃSKA, PhD Eng., Andrzej NOWOTNIK, PhD Eng., Rzeszow University of Technology, Department of Materials Science, 2 W. Pola St., 35-959 Rzeszów, Phone (+48, 0-17) 865 36 46, Fax (+48, 0-17) 854 48 32, e-mail: mrowka@prz.edu.pl

solidification, depending upon the overall alloy composition and crystallization condition. Their relative volume fraction, chemical composition and morphology exert significant influence on a technological properties of the alloys [1, 5-10]. In the aluminium alloys besides the alloying elements, transition metals such as Fe, Mn and Cr are always present. Even small amount of these impurities causes the formation of a new phase component [1, 4-10]. These phases are the consequence of equilibrium and nonequilibrium reactions occurred during casting of aluminium alloy. It worth to mention that good interpretation of microstructure relies on heaving a complete history of the samples for analysis. Therefore, the particle characterization is essential not only for choosing the best processing routes, but also for designing the optimized alloy composition.

2. Experimental procedure

The investigation was carried out on the AlCu4Ni2Mg2 casting aluminium alloy (composition: 4.3% Cu, 2.1% Ni, 1.5% Mg, 0.3% Zn, 0.1% Fe, 0.1% Si, Al-ball). The microstructure of examined alloy was observed using an optical microscope - Nikon 300, scanning electron microscope (SEM) HITACHI S-3400, and the transmission electron microscopes (TEM) Tesla BS-540 and Jeol-2100. The intermetallic particles from investigated AlCu4Ni2Mg2 alloy were extracted chemically in phenol [11-12]. The extracted powder was analyzed by using X-ray to determine the exact phase composition of the alloy. The X-ray diffraction analysis of the powder was performed using ARL-XTR'a diffractometer – Cu Kα radiation at 40kV. DSC measurements were performed using a SETARAM Setsys Evolution 1200 with a sample weight of approximately 80-90 mg. Temperature scans were made from room temperature ~25°C to 800°C at constants heating rates of 5°C/min in a dynamic argon atmosphere. The heat effects associated with the transformation (dissolution/precipitation) reactions were obtained by subtracting a super purity Al baseline run and recorded.

3. Results and discussion

DSC curves obtained by heating (Fig. 1a) and cooling (Fig. 1b) as-cast specimens of the examined AlCu4Ni2Mg2 alloy are shown in Fig. 1. DSC curves demonstrate precisely each reactions during heating and solidification process of as-cast AlCu4Ni2Mg2 alloy. One can see from the figures that during cooling the reactions occurred at lower temperatures (Fig. 1b) compared to the values recorded during heating of the same alloy (Fig. 1a). The solidification process of this alloy is quite complex (Fig. 1) and starts from formation of aluminum reach (α-Al) dendrites and Al_6Fe phase. Possible reactions (the exact value of temperature) which followed during solidification of AlCu4Ni2Mg2 alloy obtained during DSC investigation are presented in Tab. 1.

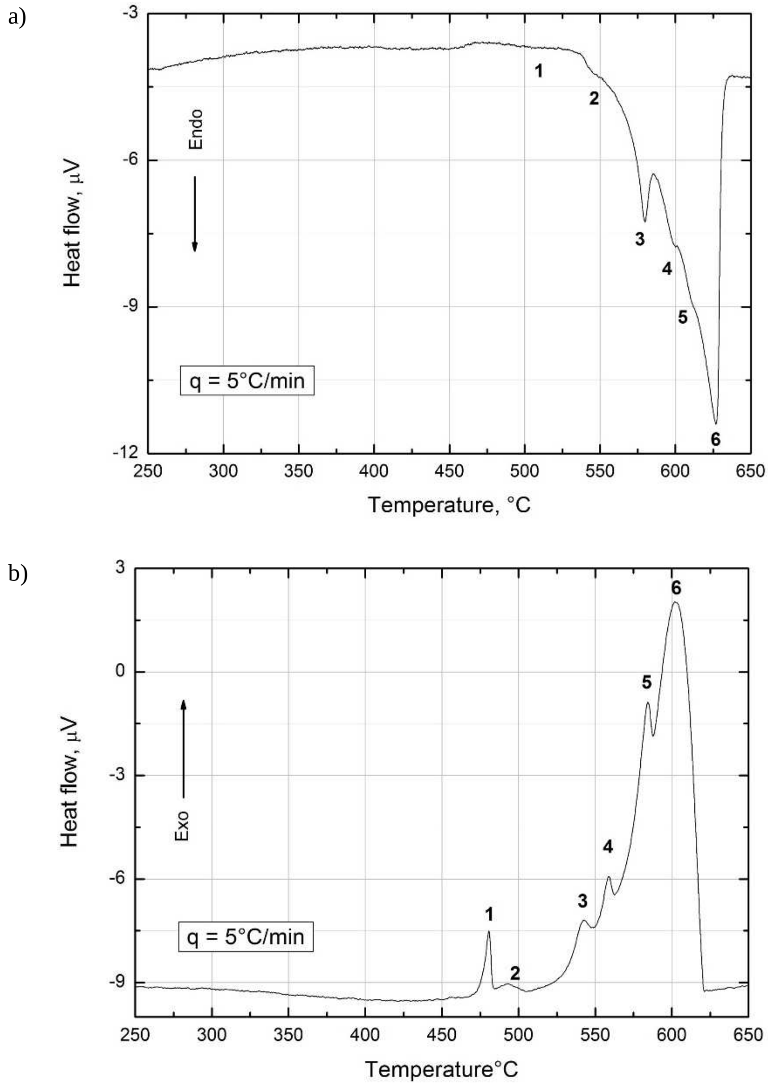


Fig. 1. DSC thermograms obtained during a) heating and b) cooling at a rate of 5°C/min

Aluminum reach (α-Al) dendrites are formed at the beginning of solidification process. The addition of alloying elements to the alloys (Ni, Cu, Mg) as well as impurities (eg. Fe) changes the solidification path and reaction products. Therefore, as-cast microstructure of the tested alloy exhibits the appearance of mixture of intermetallic phases (Fig. 2a, b).

Table 1. Possible solidification reactions during nonequilibrium conditions in investigated AlCu4Ni2Mg2 alloy, at a heating rate 5°C/min

Possible reactions	**Temperature, °C**
L→ (Al) + Al_6Fe	612
L→ (Al) + Al_4CuMg	584
L→(Al)+Al_2Cu+Al_2CuMg	558
L→(Al)+Al_2Cu+Al_7Cu_4Ni	542
L→(Al)+Al_2Cu+ Al_2CuMg +$Al_3(CuFeNi)_2$	493
Solidus	480

The analyzed microstructure in as- cast state (Fig. 2) contains of primary aluminium dendrites and substantial amount of different intermetallic phases constituents varied in shape (i.e.: sphere-like, complex rod-like and ellipse-like), size and distribution. They are located at the grain boundaries of α-Al and form dendrites network structure (Fig. 2). This alloy besides the eutectic phases and primary intermetallics contains a certain amount of the fine needle shaped particles of strengthening phases located in the boundary zone of the dendritic network structure (Fig. 2).

a) b)

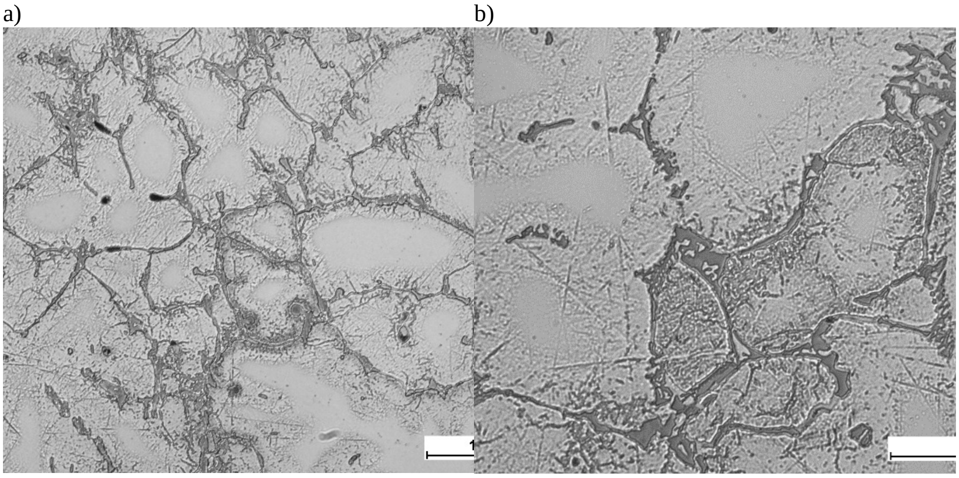


Fig. 2. The microstructure of AlCu4Ni2Mg2 alloy in as-cast state

In order to identify the intermetallic phases in the examined alloy, series of SEM observation and distribution maps were performed for the elements line Mg-K, Al-K, Fe-K, Ni-K, Cu-K (Fig. 3). The maximum pixel spectrum clearly shows the presence of Ni and Cu in the scanned microstructure. As seen in the elemental maps in Fig. 3, the regions enriched in Ni and Cu correspond to the formation of large, irregular eutectic type precipitates phases observed in Fig. 2.

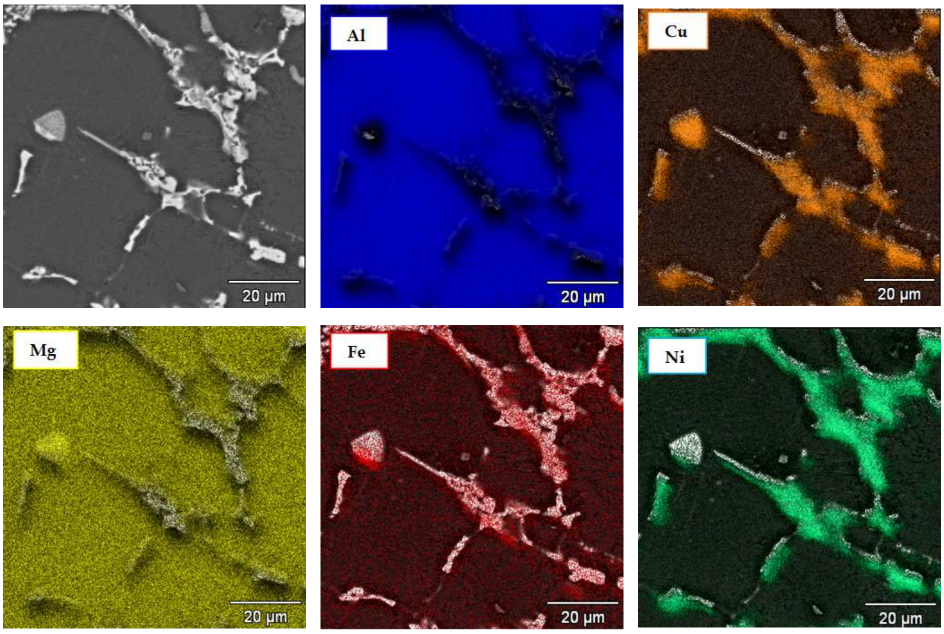


Fig. 3. SEM image of the AlCu4Ni2Mg2 alloy and corresponding elemental maps of: Al, Mg, Fe, Ni and Cu

Figure 4 shows scanning electron micrographs of the particles extracted from the as-cast AlCu4Ni2Mg2 alloy by using the phenolic dissolution technique. The EDS analysis (Tab. 2) performed on the extracted phases present in microstructure of the alloy revealed, that the phase with complex morphology is the Al_7Cu_4Ni one (Fig. 4a, c), whereas the plate-like is $Al_3(CuFeNi)_2$ (Fig. 4b, d).

The phenol extraction method was successfully applied to the examined alloy in the as-cast condition. However, the problem we came across whilst extracting a very fine particles. The residue is separated by centrifuging and since some of the particles are very fine and available sieves are having too big outlet holes there is no chance prevents them from being flowing out from a solution. Therefore transmission electron microscopy technique TEM observation (Fig. 5) and X-ray diffraction analysis (Fig. 6) of samples of the examined AlCu4Ni2Mg2 alloy were used in order to compare and confirm present results.

TEM micrographs and electron diffraction patterns analysis proved that the dispersed precipitates showed in Fig. 2 are the particles of intermetallic phases S-Al_2CuMg (Fig. 5).

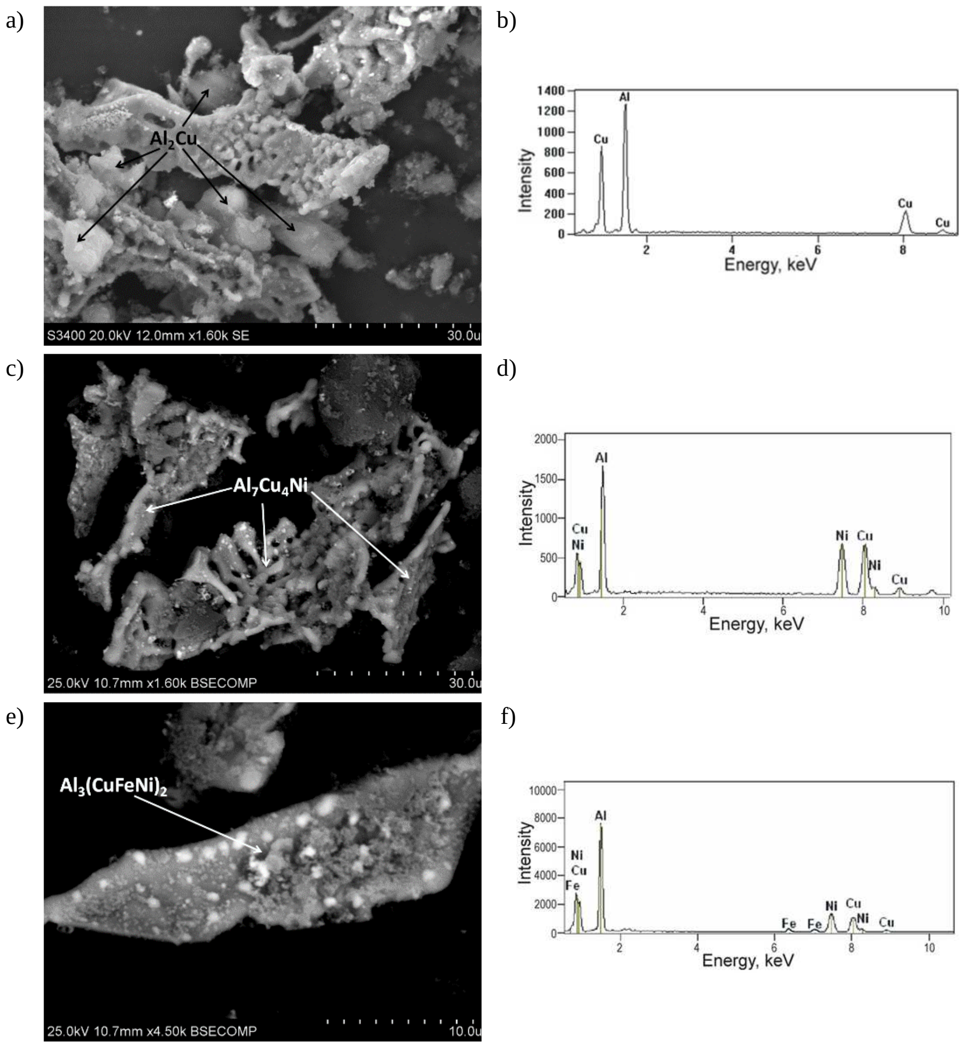


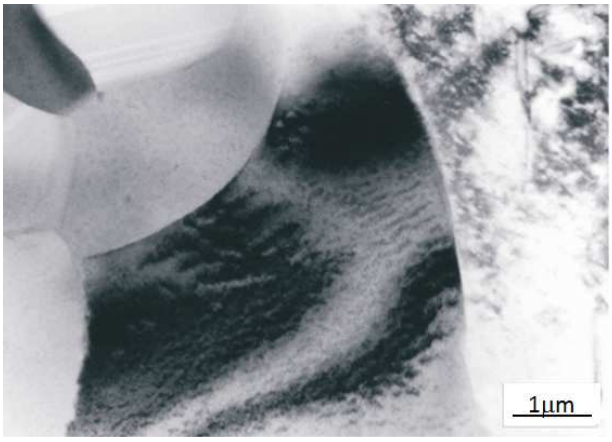
Fig. 4. SEM micrographs of the particles extracted from the AlCu4Ni2Mg2 alloy along with EDS spectra: Al_2Cu (a,b) Al_7Cu_4Ni (c,d) and $Al_3(CuFeNi)_2$ (e,f)

The results of the SEM/EDS analysis of the particles extracted with boiling phenol from AlCu4Ni2Mg2 alloy were compared with X-ray diffraction pattern (Fig. 6). The observed peaks confirmed SEM and TEM results. The majority of the peaks were from Al_7Cu_4Ni, Al_6Fe, S-Al_2CuMg, and $Al_3(CuFeNi)_2$.

Table 2. The chemical composition of the intermetallic phases in the AlCu4Ni2Mg2 alloy

No. of analyzed particles	Suggested type of phases	Chemical composition of intermetallic phases, (%at)			References
		Cu	Ni	Fe	
14	Al_2Cu	47.7÷51.9 52.5	-	-	This work [9]
25	Al_7Cu_4Ni	29.7÷45.2 38.7÷50.7 34.33	14.2÷22.6 11.8÷22.2 18.08	-	This work [10] [8]
30	$Al_3(CuFeNi)_2$	10.5÷19.3 9÷15	17.1÷20.5 18÷22	7.2÷9.5 8÷10	This work [9]

a)



b)

c)

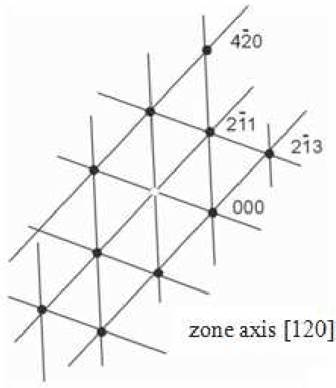


Fig. 5. TEM micrograph of AlCu4Ni2Mg2 alloy showing the precipitate of the S-Al_2CuMg phase (a), and corresponding electron diffraction pattern (b, c)

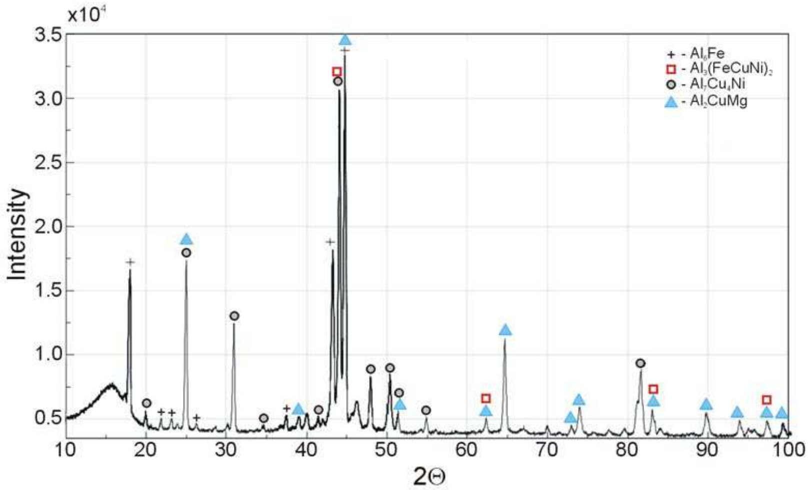


Fig. 6. The X-ray diffraction from the particles extracted from AlCu4Ni2Mg2 alloy

4. Conclusions

The solidification process of investigated AlCu4Ni2Mg2 aluminium alloy is quite complex. DSC curves obtained by heating and cooling as-cast specimens of the examined alloy demonstrate precisely each reactions during heating and solidification process of as-cast AlCu4Ni2Mg2 alloy:

- L→ (Al) + Al_6Fe – 612 °C
- L→ (Al) + Al_4CuMg - 584°C
- L→(Al)+Al_2Cu+Al_2CuMg – 558°C
- L→(Al)+Al_2Cu+Al_7Cu_4Ni – 542°C
- L→(Al)+Al_2Cu+ Al_2CuMg +$Al_3(CuFeNi)_2$ – 493°C
- Solidus – 480°C

Microstructure examination by different technique (LM, SEM/EDS, TEM, XRD) of as-cast alloy cooling with constants heating rates of 5°C during solidification confirmed DSC results. The analyzed microstructure in as- cast state contains of primary aluminium dendrites and substantial amount of different intermetallic phases constituents Al_7Cu_4Ni, Al_6Fe, S-Al_2CuMg, and $Al_3(CuFeNi)_2$. varied in shape (i.e.: sphere-like, complex rod-like and ellipse-like), size and distribution located at the grain boundaries of α-Al. This alloy also contain some amount of the fine needle shaped particles of strengthening Al_2Cu and S-Al_2CuMg phases located in the boundary zone.

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