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MICROSTRUCTURE AND MECHANICAL PROPERTIES OF SILICON-FREE Al-Cu-Ni CASTING ALLOYS USED IN HEAVY DUTY AIRCRAFT ENGINE PARTS

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

S u m m a r y

The main task of this work was to study the microstructure and mechanical properties of silicon-free Al-Cu-Ni casting alloys. The microstructure of tested samples was evaluated in terms of fracture mechanism using an optical microscope – Nikon 300, scanning electron microscope HITACHI S-3400 (SEM) in a conventional back-scattered electron mode and JEOL – JEM 2100 ARP TEM/STEM electron microscope. The mechanical (R_m and $R_{0.2}$) and plastic (A,Z) properties of the examined alloy were evaluated by uniaxial tensile test at room temperature. The results shows that the casting method and the applied thermal processing did not have a significant influence on the primary (Al_6Fe, Al_2CuMg, Al_7Cu_4Ni, $Al_3(CuFeNi)_2$ and AlCuMn) intermetallic phase composition and microstructure phase component morphology. However, during prolonged heating growth and change in the shape of separations of the θ'-Al_2Cu reinforcing phase occurs proportionally to temperature and heating time. Sand casts show higher mechanical properties: R_m $R_{0.2}$. The stability of the mechanical properties of the investigated alloys in higher temperatures is the consequence of the increased Cu content.

Keywords: aluminium alloy, microstructure, heat treatment, mechanical properties

Mikrostruktura i właściwości mechaniczne odlewniczych bezkrzemowych stopów Al-Cu-Ni stosowanych na silnie obciążone elementy silników lotniczych

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

Celem pracy była analiza mikrostruktury i właściwości mechanicznych odlewniczych, bezkrzemowych stopów Al-Cu-Ni. Obserwacje mikrostruktury prowadzono przy użyciu mikroskopu świetlnego – Nikon 300, elektronowego mikroskopu skaningowego HITACHI S-3400 z systemem EDS do mikroanalizy rentgenowskiej i elektronowego mikroskopu transmisyjnego JEOL – JEM 2100 ARP z systemem STEM/EDS. Właściwości wytrzymałościowe (R_m, $R_{0.2}$) i plastyczne (A,Z) badanych stopów wyznaczono w próbie statycznej rozciągania w temperaturze pokojowej. Analiza uzyskanych wyników badań pozwoliła stwierdzić, że sposób odlewania oraz obróbka cieplna nie mają istotnego wpływu na skład fazowy oraz morfologię pierwotnych cząstek faz międzymetalicznych (Al_6Fe, Al_2CuMg, Al_7Cu_4Ni, $Al_3(CuFeNi)_2$ i AlCuMn. Długotrwałe wygrzewanie, w podwyższonej temperaturze powoduje jednak znaczny wzrost cząstek umacniającej fazy θ'-Al_2Cu. Ustalono, że lepsze właściwości mechaniczne: R_m i $R_{0.2}$ wykazują stopy odlane do form piaskowych. Stabilność właściwości mechanicznych badanych stopów w podwyższonej temperaturze jest spowodowana większą zawartością Cu.

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Słowa kluczowe: stopy aluminium, mikrostruktura, obróbka cieplna, właściwości mechaniczne

1. Introduction

Use of aluminum-based alloys as construction materials is determined by their properties, mostly low density and high relative durability (R_m/δ), good electrical and thermal conductivity and good technological properties – castability and susceptibility for plastic deformation. The limited costs of producing aluminum are factors that stimulate the constant development of aluminum alloy manufacturing and processing technology [1-6].

Development of the Polish aerospace industry, including light and ultra-light aircraft, is inevitably connected with the manufacturing and application of aluminum alloys that are characterized by greater durability and lesser density in comparison with alloys used earlier. This necessitates the development of new solutions in chemical composition and thermal processing of high durability aluminum alloys, both those used for casting and those used for plastic working in the load-bearing structures of aircraft. Simultaneously, the development of aircraft drives created the demand for aluminum alloys with improved heat resistance (elements of combustion engines, turbine rotors and others) that is controlled by a process of heat treated solid solution decomposition. This is a group of precipitation hardened aluminum alloys. It is mostly composed of alloys containing copper with a small admixture of nickel and magnesium. These alloys are highly durable, while maintaining good plastic properties. However, they have worse castability compared with aluminum alloys containing silicon [2-7]. An important role in shaping durability properties of these alloys is played by their microstructure and phase composition, and especially relative volume and morphology of microstructure phase elements, including precipitates of stable reinforcing phases Al_2Cu with significant dispersion, separating from heat treated solid solution during aging [5-11].

Al-Cu-Ni alloys are used to cast engines less often than Al-Si-Cu alloys because of the technological difficulties [3-4,12-14], but they are the basis for the production of multi component alloys. An important element of this group are Al-Cu-Ni-Mg alloys with up to 4.5% Cu, approx. 2% Mg and approx. 2% Ni.

The performed tests confirmed that the proposed alloys with a nickel admixture are characterized by significant stability of mechanical properties with high tensile strength, as well as good plasticity at temperatures of 250 to 350°C and that they can be used to cast heavy duty elements of combustion aircraft engines for light and ultra-light aircraft.

2. Material and methodology

2.1. Material

Two casting group 2xxx aluminium alloys with a nickel admixture were selected for testing: AlCu4Ni2Mg2 and AlCu6Ni1 (Table 1). The AlCu4Ni2Mg2 alloy is a standard alloy currently used for heavy duty engine elements. The AlCu6Ni1 alloy is an experimental alloy that is being tested in order to ascertain the influence of an increased amount of Cu on the phase composition, the morphology of the microstructure components, and, consequently, on mechanical, technological and operational properties.

Table 1. Chemical composition of the tested alloys (% of weight)

Alloy	Mn	Ni	Cu	Zr	Fe	Si	Mg	Zn
AlCu4Ni2Mg2	<0.1	2.1	4.3	-	0.1	0.1	1.5	0.3
AlCu6Ni1	0.9	1.1	6.36	0.01	0.2	0.1	0.05	-

In order to simplify the marking of the test samples, the alloys were marked as follows: alloy A – AlCu4Ni2Mg2 and alloy B – AlCu6Ni1. In the mark of each alloy an additional letter was introduced, signifying the casting method: K – chill casting, P – sand casting. The casts underwent T6 thermal processing – solution heat treatment (alloy A: $520^{\pm 5o}$C/5h/water, alloy B: $545^{\pm 5o}$C/10h/water) + artificial aging (alloy A: $250^{\pm 5o}$C/5h/air, alloy B: $225^{\pm 5o}$C/8h/air). Alloys after T6 thermal processing were additionally heated at a temperature of 250°C, 300°C and 350°C, for a time of 100 h, 150 h, 300 h, 500 h and 750 h.

2.2. Methodology

In order to observe the changes in the microstructure of the tested alloys resulting from the casting method (chill, sand) and the used thermal processing, the microstructure of the alloys was observed using light microscope (LM) NikonEpiphot 300, scanning electron microscope (SEM) Hitachi S-3400N with EDS system for X-ray microanalysis and transmission electron microscope (TEM) Jeol-2100 with STEM/EDS and Tesla BS 540 systems.

Metallographic specimens were prepared using standard methods; the samples were etched using modified Keller reagent: 2cm^3 HF + 3cm^3 HCl + 20cm^3 HNO_3 + 175cm^3 H_2O. Thin foils were prepared using a method based around double sided thinning by electrochemical polishing in a reagent with the following chemical composition: 260 ml CH_3OH + 35 ml of glycerin + $5HclO_4$

using a Tenupol-3 device by Struers and ion beam milling device by Cressington. The temperature of phase transformations in the tested alloys was determined using the DSC method (differential scanning calorimetry), using a SETARAM SETSYS Evolution-1200 calorimeter. The used samples weighed 0.22-0.30 g and the reference sample was pure aluminum (99.7%). The samples were heated from the surrounding temperature to 650°C at a rate of 4°C/min. The mechanical properties (tensile and yield strength; elongation) of the tested alloys were determined using a static tensile test. Round, fivefold samples with a diameter of 6 mm were tensile tested using a universal tester Instron-8801 [15].

3. Results

The observations of the tested alloys in as cast state (Fig. 1, 2) led to the conclusion that the casting method of the AlCu4Ni2Mg2 (A) and AlCu6Ni1 (B) alloys had no significant impact on the phase composition and the morphology of phase components of the microstructure of the tested alloys.

a) b)

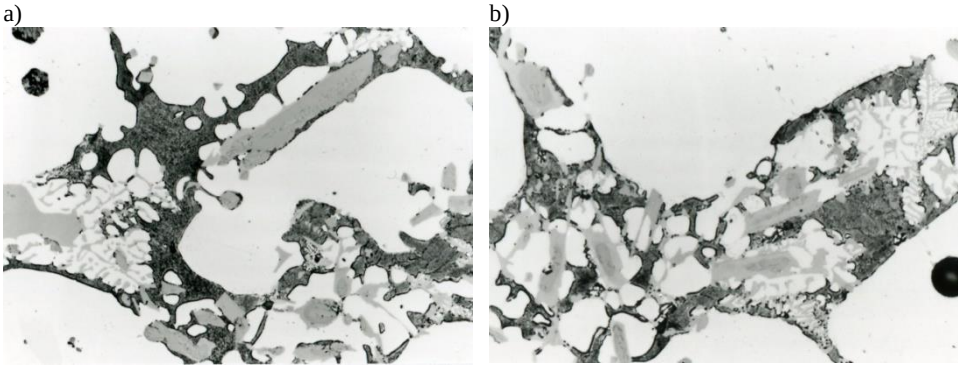


Fig. 1. Microstructure of the AlCu4Ni2Mg2 (A) alloy: a) sand casting (P), b) chill casting (K)

a) b)

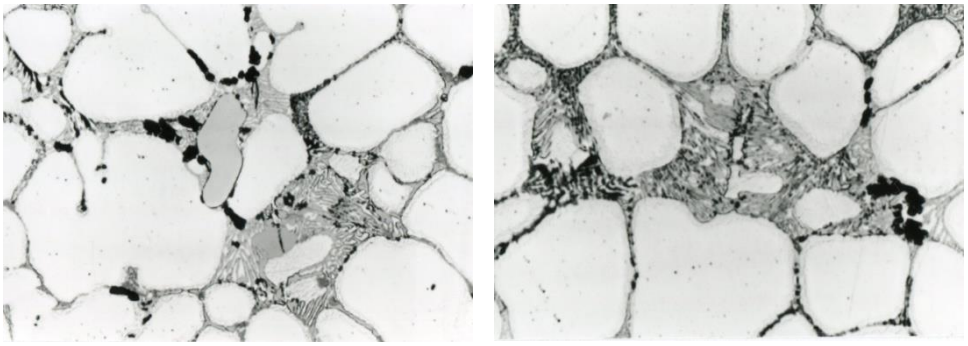


Fig. 2. Microstructure of the AlCu6Ni1 (B) alloy: a) sand casting (P), b) chill casting (K)

However, it was ascertained that in the both tested alloys after T6 thermal processing large separations of intermetallic phases that separate at the boundaries of the dendrites of the solid solution α were partly dissolved and changed shape (Fig. 3, 4). Preliminary observations showed that in the A alloy in the T6 state there are three types of separations: spheroid with wide surfaces, ellipsoid that separate in both the A(K) alloy and the A(P) alloy at the boundaries of the dendrites of the solid solution α, and spheroid with a large dispersion – in the A(P) alloy there is a slightly larger concentration of these in areas at the dendrite boundaries (Fig. 3a), and in the A(K) alloy the situation is opposite – boundary areas are partly devoid of these separations (Fig. 3b).

a) b)

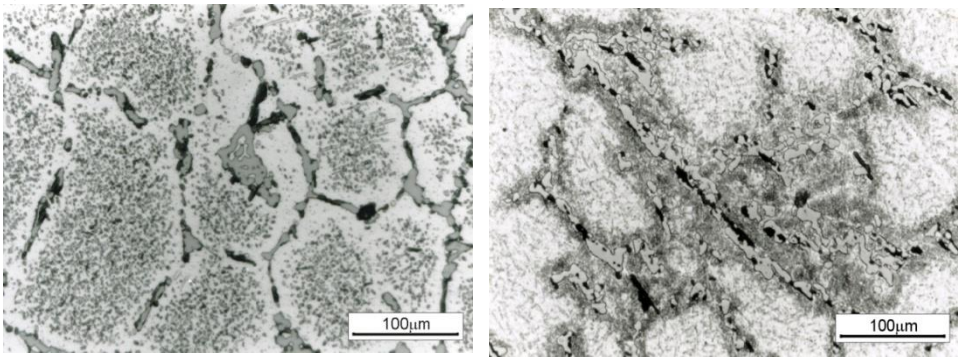


Fig. 3. Microstructure of the AlCu4Ni2Mg2 (A) alloy: a) sand casting (P), b) chill casting (K)

a) b)

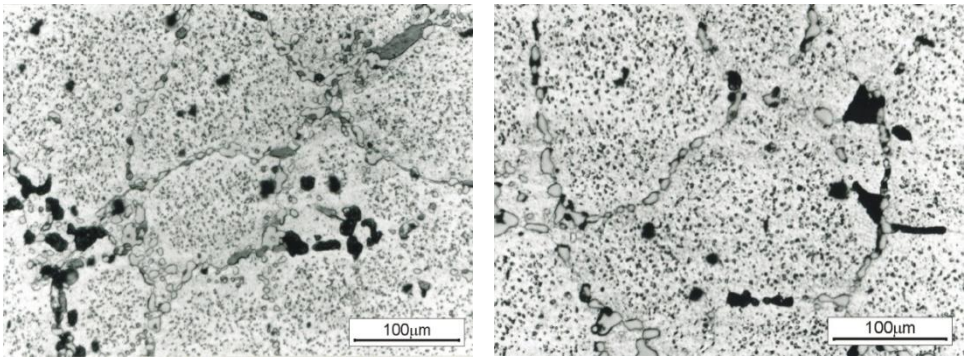


Fig. 4. Microstructure of the AlCu6Ni1 (B) alloy: a) sand casting (P), b) chill casting (K)

In alloy B, both spheroid and irregular separations were observed, with varied shapes and proportionally large sizes, separating at the boundaries of the dendrites of the solid solution α, and spheroid precipitates with a large dispersion evenly placed in the solid solution α in the whole volume of the alloy (Fig. 4).

Observation of the tested alloys performed using a scanning electron microscope in combination with EDS microanalysis of the chemical composition (point method) led to the conclusion that spheroid separations visible in the AlCu4Ni2Mg2 (A) alloy are Al_7Cu_4Ni phase precipitates, and ellipsoid separations are $Al_3(CuFeNi)_2$ phase precipitates. The original large separations visible in the AlCu6Ni1 (B) alloy (Fig. 4 and 5) are Al_7Cu_4Ni phase precipitates, and ellipsoid separations are $Al_3(CuFeNi)_2$ and AlCuMn phase precipitates (Fig. 5, Table 2).

a) b)

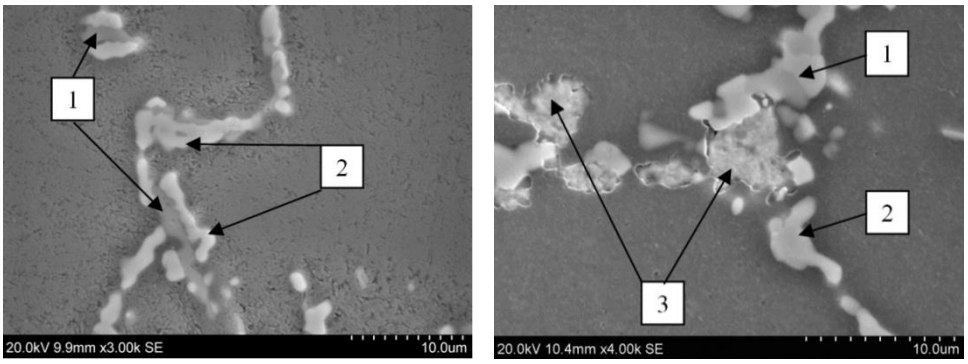


Fig. 5. Microstructure of the alloys a) AlCu4Ni2Mg2 (A) and b) AlCu6Ni1 (B) SEM image

Table 2. Chemical composition and relative volume of separations identified in the tested alloys: AlCu4Ni2Mg2 –A and AlCu6Ni1 – B

Alloy	**Points from fig. 5**	**Phase type**	**Chemical composition of the separations (% at)**	V_V **[%]**
AlCu4Ni2Mg2	***1***	Al_7Cu_4Ni	Al 52.9-65.2; Cu 23.7-30.2; Ni 7.1-8.6	2.3
	2	$Al_3(CuFeNi)_2$	Al 65.9-73.1; Cu10.5-19.3; Ni 7.1-10.3; Fe4.5-7.5	1.1
AlCu6Ni1	**1**	Al_7Cu_4Ni	Al 52.9-63.1; Cu 23.7-29.2; Ni 8.0-11.2	0.9
	2	$Al_3(CuFeNi)_2$	Al 65.9-73.1; Cu 10.5-19.3; Ni 7.1-10.3; Fe 4.5-7.5	1.2
	3	AlCuMn	Al 65.9-73.1; Cu 30.7-35.3; Mn 10.06-15.3	1.75

The observation of the tested alloys microstructure using TEM and the analysis of the resulting diffraction patterns showed that in the A alloy, aside from the original separations of intermetallic phases observed using the light microscope and SEM, there are separations of the reinforcing phase θ'-Al_2Cu (Fig. 6).

a) b)

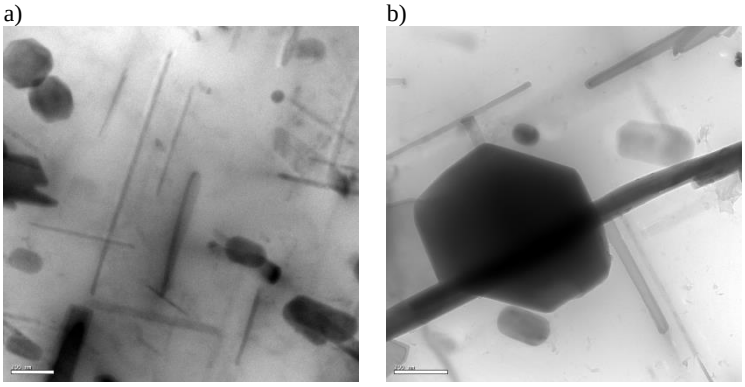


Fig. 6. Microstructure of the AlCu4Ni2Mg2 (A) alloy, visible separations of the θ'-Al_2Cu phase: a) needle shaped, b) compact shaped

The precipitates with a large dispersion observed using the light microscope (Fig. 3) were identified as separations of the Al_6Fe and Al_2CuMg phases (Fig. 7, 8).

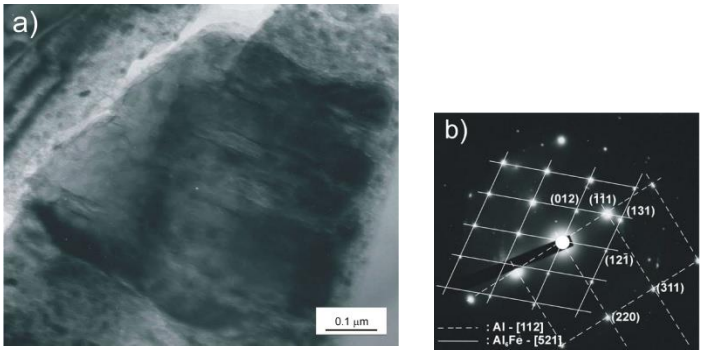


Fig. 7. Microstructure of the AlCu4Ni2Mg2 (A) alloy: a) Al_6Fe phase precipitate, b) electron diffraction image with a solution

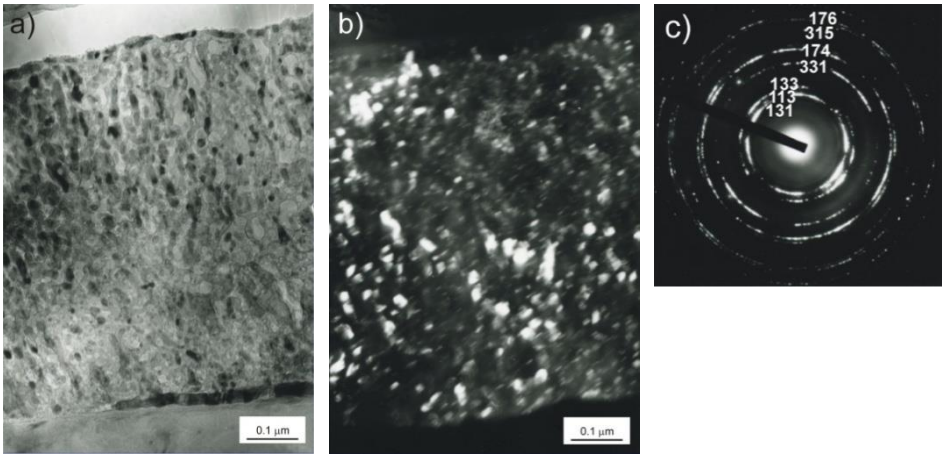


Fig. 8. Microstructure of the AlCu4Ni2Mg2 (A) alloy: a) S-Al_2CuMg phase precipitate, image in a bright field, b) image in a dark field, c) electron diffraction image with a solution

In alloy B, separations of the reinforcing phase θ'-Al_2Cu (Fig. 9) and the α-Al_2CuMg phase (Fig. 10) were also identified using diffraction analysis (TEM). The shape of the Al_2Cu phase precipitates is varied, from the compact, regular "crystals" to elongated "tiles" (Fig. 9).

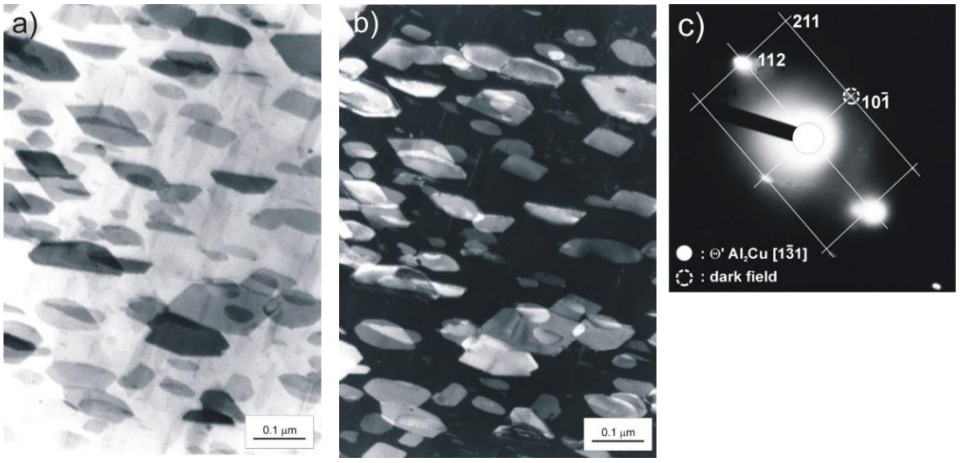


Fig. 9. Microstructure of the AlCu6Ni1 (B) alloy – separations of the θ'-Al_2Cu phase

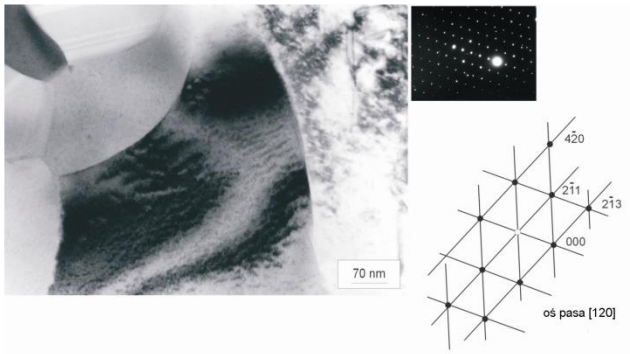


Fig. 10. Microstructure of the AlCu6Ni1 (B) alloy: α-Al_2CuMg phase precipitate

Microscopic observation with large zoom values (TEM) showed that the microstructure of the alloys with regard to morphology of reinforcing phase separations is identical for both sand casts and chill casts. Therefore, the microstructure of these alloys after prolonged heating was treated as identical for groups A and B, without separation into sand and chill casts (Fig. 11-13). Image of the microstructure of the AlCu4Ni2Mg2 (A) alloy after heating at a temperature of 250 to 350°C for a time of 100 h, 500 h and 750 h is shown in Fig. 12.

a) b)

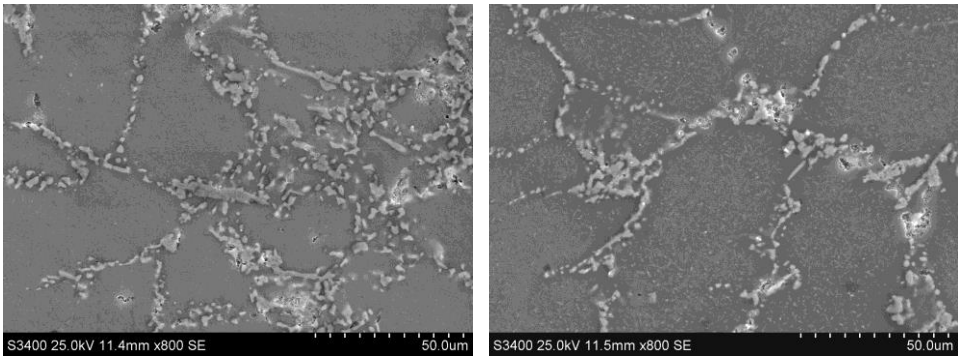


Fig. 11. Microstructure of the alloys A a) and B b) after heating at a temperature of 350°C/750 h

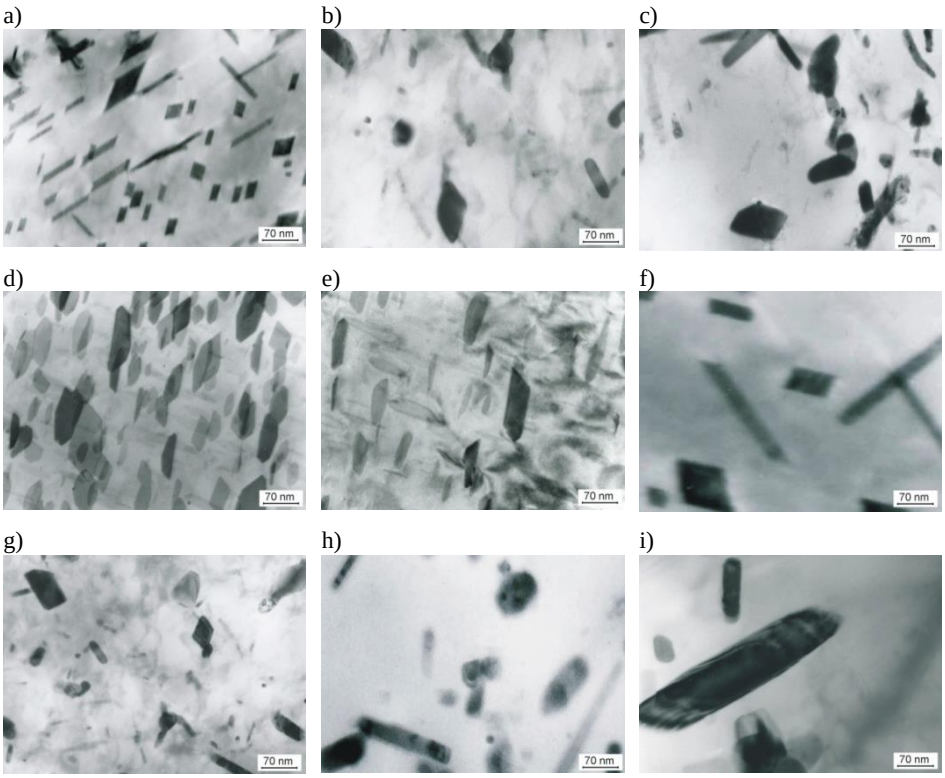


Fig. 12. Microstructure of the AlCu4Ni2Mg2 (A) alloy – separations of the θ'-Al_2Cu phase after heating at a temperature of 250°C for a time of: a) 100 h, b) 500 h, c) 750 h; 300°C for a time of: d) 100 h, e) 500 h, f) 750 h and 350°C for a time of: g) 100 h, h) 500 h, i) 750 h

The observations led to the conclusion that heating at an increased temperature had no significant impact on the morphology of the original separations of the phase components of the microstructure of the tested alloys A and B. These separations did not change their shape and size even after a long time of heating – up to 750 h (Fig. 11). The image of the microstructure of the AlCu6Ni1 (B) alloy after heating at a temperature of 250 to 300°C for a time of 100 h, 500 h and 750 h is shown in Fig. 13.

It was found that both in alloy A and alloy B the prolonged effects of heat cause the precipitates of reinforcing phases to enlarge in proportion to temperature and heating time. A greater tendency to increase the number of separations is shown by alloy B. It was determined that microstructure degradation resulting from prolonged heating applies mostly to the growth of the precipitates of reinforcing phases and the changes in their shape.

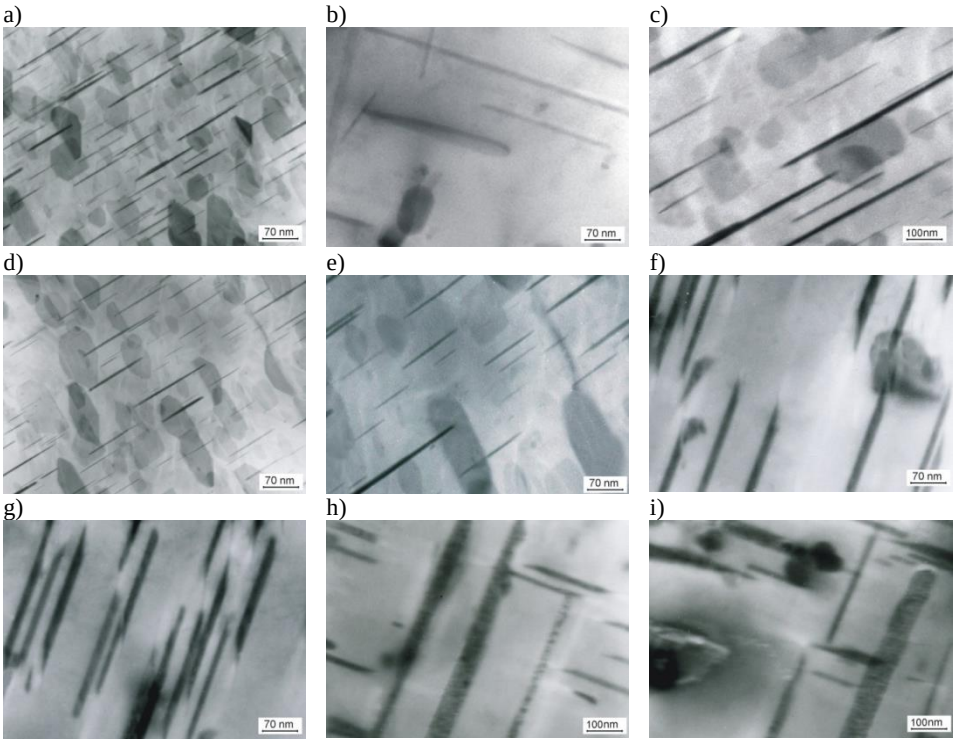


Fig. 13. Microstructure of the AlCu6Ni1 (B) alloy – separations of the θ'-Al_2Cu phase after heating at a temperature of 250°C for a time of: a) 100 h, b) 500 h, c) 750 h; 300°C for a time of: d) 100 h, e) 500 h, f) 750 h and 350°C for a time of: g) 100 h, h) 500 h, i) 750 h

Determining the temperature of phase transformations and the thermal expansion coefficient of the tested alloys was determined using the DSC method (differential scanning calorimetry) and the DIL 805 BÄHR THERMO System dilatometer.

Table 3. Temperature of phase transformations in the alloys AlCu4Ni2Mg2 (A) and AlCu6Ni1 (B)

Alloy	**Temperature of phase transformations °C**					
A(K)	582	605	636	-	-	-
A(P)	583	605	632	-	-	-
B(K)	537	556	567	590	612	641
B(P)	–	–	573	590	609	640

The temperature values of phase transformations taking place in the tested alloys, determined using the resulting DSC curves, are shown in Table 3. After the analysis of the results, no significant difference in the value of the temperature of phase transformations connected with the casting method (sand casting (P) and chill casting (K)) was found.

Values of the thermal expansion coefficient obtained at three temperature ranges: 20-100°C (293-373K); 20-200°C (293-473K); 20-300°C (293-573K) are presented in Table 4.

Table 4. Coefficient of thermal expansion of the AlCu4Ni2Mg2 and AlCu6Ni1 alloys

Alloy	**Coefficient of thermal expansion $\alpha \cdot 10^6$, 1/K**		
	Temperature range, K		
	293 – 373	**293– 473**	**293– 573**
A(K)	18.5	20.2	21.2
A(P)	22.4	22.8	22.7
B(K)	24.1	21.2	23.9
B(P)	22.2	22.6	23.4

Analysis of the results of dilatometric tests showed that the thermal expansion coefficients of the tested alloys cast using both the sand and the chill method are comparable and their values do not change significantly in the tested

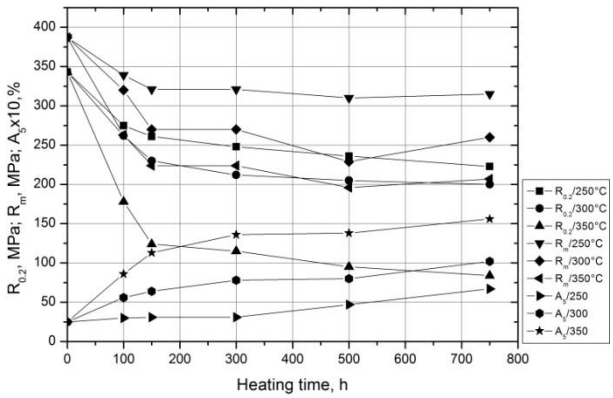
temperature ranges. Mechanical properties of the tested alloys after T6 thermal processing are shown in Table 5.

Table 5. Mechanical properties of the alloys AlCu4Ni2Mg2 (A) and AlCu6Ni1 (B) after standard T6 thermal processing

Mechanical properties	**Alloy A**		**Alloy B**	
	cast P	**cast K**	**cast P**	**cast K**
$R_{0.2}$, MPa	343	305	331	285
R_m, MPa	387	318	400	323
A_5, %	2.5	0.8	2.7	0.7

Results of mechanical tests of the A and B alloys after heating at temperatures of 250°C, 300°C and 350°C for a time of 100 h, 150 h, 300 h, 500 h and 750 h are shown in Fig. 14 and 15.

a)



b)

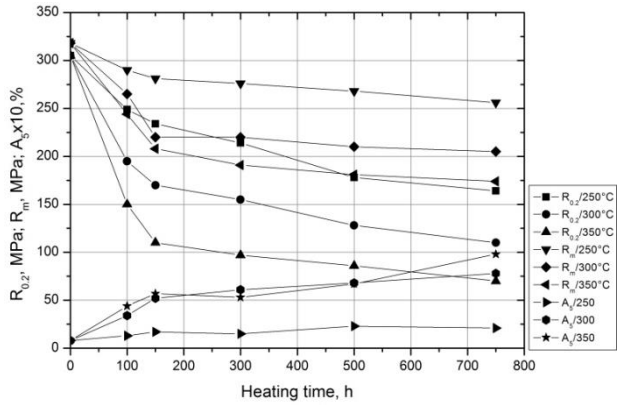


Fig. 14. Relation of tensile strength R_m, yield point $R_{0.2}$ and relative elongation A_5 of alloys: a) AP, b) AK to the time of heating at a temperature of 250°C, 300°C and 350°C

The analysis of the results of mechanical properties tests showed that all the alloys that were heated for a prolonged period show a large decrease in durability properties, regardless of temperature. The numerical values (in %) were determined on the basis of the following formula:

$$[(R - R_{(t)}) \text{ x } R^{-1}) \text{ x}100]$$

where: R – value of R_m or $R_{0.2}$ after T6 thermal processing, $R_{(t)}$ – value of R_m or $R_{0.2}$ after heating at a temperature t = 250/300/350°C – Fig. 16.

The rise in heating temperature caused a significant decrease in mechanical properties of the tested alloys (Fig. 16). However, it was observed that despite a large decrease in the tensile strength R_m with the increased time, its value does not change in fact, even after 750 h of heating. This is evidence of the high stability of durability properties of the tested alloys at temperatures up to 350°C.

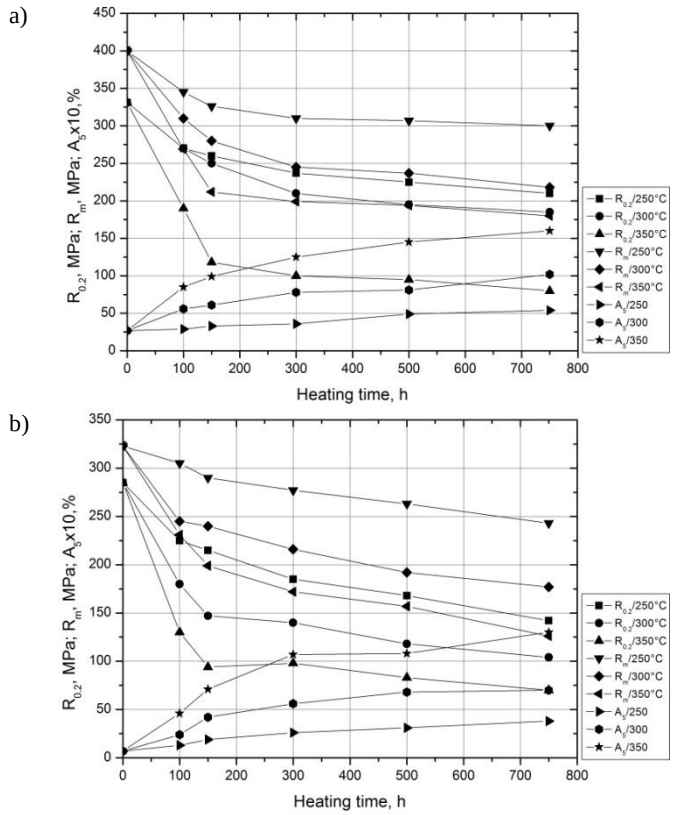


Fig. 15. Relation of tensile strength R_m, yield point $R_{0.2}$ and relative elongation A_5 of alloys: a) AP, b) AK, c) BP, d) BK to the time of heating at a temperature of 250°C, 300°C and 350°C

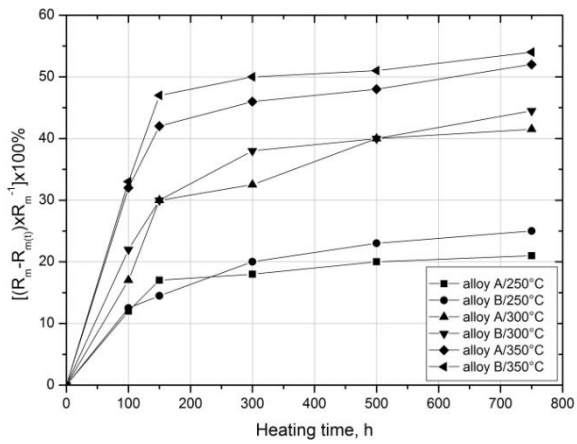


Fig. 16. Change of tensile strength R_m of the AlCu4Ni2Mg2 (A) and AlCu6Ni1 (B) alloys after heating at a temperature of 250°C, 300°C and 350°C, depending on time

4. Discussion and conclusions

Microscope observations (LM, SEM, TEM) of the tested alloys in as-cast state and after T6 thermal processing did not show a significant impact of the casting method and the applied thermal processing on the phase composition and microstructure phase component morphology. It was determined that in the microstructure of the tested alloys, on the boundaries of the dendrites of the solid solution α-Al the following intermetallic phases occur: Al_6Fe, Al_2CuMg, Al_7Cu_4Ni and $Al_3(CuFeNi)_2$ (alloys A and B). Additional separations of the AlCuMn were found in alloy B. It was determined that T6 thermal processing does not affect microstructural construction of the alloys as regards the occurrence of intermetallic phases (the only change is in the shape of separations – sharp edges are rounded). The presence of separations of dispersive reinforcing phase θ'-Al_2Cu was found in the dendrites of the α-Al solid solution. As a result of prolonged heating at an increased temperature, a large increase in the size of reinforcing phase precipitates occurred, but no significant changes to their shape were observed. However, prolonged heating did not affect the morphology of intermetallic phases (Al_6Fe, Al_2CuMg, Al_7Cu_4Ni, $Al_3(CuFeNi)_2$ and AlCuMn) that were created during primary crystallization. Growth and change in the shape of separations of the θ'-Al_2Cu reinforcing phase in the AlCu4Ni2Mg2 and AlCu6Ni1 alloys occurs proportionally to temperature and heating time and results in a slight decrease in their durability properties. Tests of the mechanical properties of the tested alloys showed that the casting method: sand casting (P) and chill casting (K) has a slight effect on their values. Sand casts show higher stretching strength R_m and conventional plasticity boundary $R_{0.2}$. In addition, it was determined that alloys A and B are characterized by a high stability of mechanical properties, while simultaneously maintaining high tensile strength and plasticity, in conditions of prolonged heat, especially at temperatures of 250 to 350°C. On the basis of the test results it can be ascertained that the stability of the mechanical properties of the AlCu4Ni2Mg2 and AlCu6Ni1 alloys in higher temperatures is the result of the increased Cu content in comparison with the widespread Al-Si-Cu. Furthermore, the results of calorimetric tests show that at temperatures of 250-350°C there is no occurrence of phase transformations that could modify the microstructure, and therefore the mechanical properties of the alloys. The values of the thermal expansion coefficients in the temperature range that corresponds to the operating conditions of the structure elements cast from the tested alloys are also practically constant. On the basis of the analysis of the results, it is possible to ascertain that these alloys meet the requirements for heavy duty aircraft engine part materials and that they can be used successfully for parts operating at temperatures up to 350°C.

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References

[1] Aluminium Handbook. vol.1: Fundamentals and Materials. Aluminium-Verlag Marketing & Kommunikation GmbH, Düsseldorf 1999.
[2] J.W. MARTIN: Precipitation Hardening. Oxford, Pergamon Press 1968.
[3] I.J. POLMEAR: Light alloys. Metallurgy of the light metals. Arnold, London-New York-Sydney-Auckland 1995.
[4] F. KING: Aluminium and its alloys. John Willey & Sons, New York-Chichester-Brisbane-Toronto, 1987.
[5] C. RAVI, C. WOLVERTON: First-principles study of crystal structure and stability of Al-Mg-Si-(Cu) precipitates. *Acta Mater*., **52** (2004) 4213-4227.
[6] G. MRÓWKA – NOWOTNIK: Intermetallic phase particles in cast AlSi5Cu1Mg and AlCu4Ni2Mg2 aluminium alloys. *Arch. of Mater Sci. Eng.*, **38**, **2** (2009) 69-77.
[7] G. MRÓWKA – NOWOTNIK: Analysis of intermetallic phase particles in cast AlCu4Ni2Mg2 aluminium alloy in T6 condition. Proc. Inter. Conf.: Mashinostroenie i tekhnosfera XXI veka, Donieck 2009, 177-181.
[8] G. MRÓWKA - NOWOTNIK, J. SIENIAWSKI: The chemical phenol extraction of intermetallic particles from casting AlSi5Cu1Mg alloy. *J. Microscop*, **237**(2010) 407-410.
[9] Z. LI, A.M. SAMUEL, C. RAYINDRAN, S. VALTIERRA, H.W. DOTY: Parameters controlling the performance of AA319-type alloys: Part II. Impact properties and fractography. Mater. Sci. Eng., **A367** (2004) 111-122.
[10] L.F. MONDOLFO, Aluminium Alloys: Structure and Properties. Butterworths London-Boston 1976.
[11] M. WIERZBIŃSKA, J. SIENIAWSKI: Effect of morphology of eutectic silicon crystals on mechanical properties and cleavage fracture toughness of AlSi5Cu1 alloy. *J. Ach. Mater Manuf. Eng*., **14** (2006) 217-220.
[12] F.O. TRAEKNER: Factors Affecting the physical characteristics of aluminium magnesium silicon alloy extrusions. Proc. Inter. Conf. Atlanta 1977, 339-347.
[13] L. BÄCKERUD, G. CHAI: Solidification characteristics of aluminum alloys 3. American Foundry Society, Des Plaines, Illinois 1992.
[14] N.A. BELOV, A.A. AKSENOV, D.G ESKIN: Iron in aluminium alloys. Taylor & Francis Inc, New York 2002.
[15] PN-EN 10002-1+AC1. Metals. Standard method of tensile test at room temperature.

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