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# Al-Si-RE ALLOYS CAST BY THE RAPID SOLIDIFICATION PROCESS

## STOPY AI-SI-RE ODLEWANE METODĄ RAPID SOLIDIFICATION

The aim of the studies described in this article was to present the effect of rare earth elements on aluminium alloys produced by an unconventional casting technique. The article gives characteristics of the thin strip of Al-Si-RE alloy produced by Rapid Solidification (RS). The effect of rare earth elements on structure refinement, i.e. on the size of near-eutectic crystallites in an aluminium–silicon alloy, was discussed. To determine the size of crystallites, the Scherrer X-ray diffraction method was used. The results presented capture relationships showing the effect of variable casting parameters and chemical composition on microstructure of the examined alloys. Rapid Solidification applied to Al-Si alloys with the addition of mischmetal (Ce, La, Ne, Pr) refines their structure.

Keywords: aluminium alloy, Rapid Solidification, grain refinement, ultrafine structure.

Celem badań opisanych w artykule było przedstawienie wpływu pierwiastków metali ziem rzadkich na stopy aluminium otrzymywane niekonwencjonalną metodą odlewniczą. W artykule scharakteryzowano cienką taśmę ze stopu Al-Si-RE wytworzoną metodą szybkiej krystalizacji (Rapid Solidification). Przedstawiono wpływ pierwiastków metali ziem rzadkich na rozdrobnienie struktury - wielkość krystalitów okołoeutektycznego stopu aluminium - krzem. Do oznaczenia wielkości krystalitów wykorzystano rentgenowską dyfrakcyjną metodę Scherrera. Zaprezentowane wyniki ujmują zależności zmiennych parametrów odlewania i składu chemicznego na mikrostrukturę badanych stopów. Zastosowanie szybkiej krystalizacji dla stopów Al-Si z dodatkiem miszmetalu (Ce, La, Ne, Pr) powoduje rozdrobnienie ich struktury.

#### 1. Introduction

Aluminium alloys are an attractive construction material offering high mechanical properties and low specific gravity. For this reason they are used in all those cases where the reduced weight of components is a must. The weight of produced components depends on the type of material, on the product design and on the manufacturing process. These factors contribute to the continuous search for modern solutions, finding new alloys, new constructions and new processes for their manufacture [1,2].

One of these modern casting techniques is melt spinning. It involves rapid cooling of liquid metal fed in the form of a thin stream to the rotating wheel. The wheel made of copper is in most cases water-cooled and removes heat from the solidifying alloy at a rate of  $10^4$ – $10^6$  °C/s. Rapid Solidification allows producing metal strips (ribbons) with ultrafine structure promoting the increased strength properties[3,4].

#### 2. Methodology

As a result of completed studies, the effect of the addition

of rare earth elements to near-eutectic aluminium-silicon alloy cast in a melt spinning device was determined (Fig. 1).



Fig. 1. The flow diagram of ribbon manufacture by RS

- step 1 alloy preparation for RS,
- step 2 alloy refinning and filtration,
- step 3 casting of ribbon by the technique of Rapid Solidification

Ribbons are made in three stages, which include preparation of alloy of proper chemical composition, gas refining, and casting ribbons in a dedicated device for the Rapid Solidification process implementation.

The near-eutectic silumin was examined without and with the addition of rare earth elements in the form of mischmetal. To refine the structure of Al-Si alloys, the addition of 4% and

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8% of mischmetal, comprising a mixture of Ce, La, Pr and Nd, was introduced. The results of the chemical analysis of produced ribbons are compared in Table 1.

Chemical composition of produced ribbons

TABLE 1

Elamont	Content, wt%					
Element	AlSi11.6	AlSi11.6 AlSi11.6Mm4				
Si	12.46	12.65	12.66			
Fe	0.12	0.18	0.16			
Mn	0.03	0.09	0.01			
Mg	0.00	0.00	0.03			
Ni	0.00	0.01	0.01			
Zn	0.02	0.02	0.02			
Pr	0.00	0.17	0.26			
Ce	0.00	2.00	4.50			
La	0.00	1.24	2.21			
Nd	0.00	0.57	0.97			
RE (total)	0.00	3.98	7.94			

To determine the best casting temperature for alloys processed by Rapid Solidification, thermal analysis was performed (Table 2). The AlSi11.6 alloy was cast at a temperature of 620°C, while alloys with the addition of mischmetal were cast at 680°C (4%) and 720°C (8%). Optimum casting parameters of the examined alloys were determined for the selected rotational speeds of the wheel. The linear velocity at which the ribbons were cast was 15 m/s, 22.5 m/s and 30 m/s.

The effect of rare earth elements was investigated maintaining constant diameter of the pouring nozzle outlet, constant gas pressure in ejecting cushion and constant temperature of the wheel.

### 3. Evaluation of the cast ribbon surface

Depending on the casting speed or linear speed of the wheel, the obtained ribbons were characterized by different surface topography and different thickness. The wheel facing surface of the cast strip was more smooth. Images of strips cast from the tested alloy, showing both their surfaces, i.e. the surface facing the copper wheel and the surface facing the atmosphere (air), obtained at an extreme casting speed of 15 and 30 m/s, are shown in the drawings below (Figs. 2-5).



Fig. 2. Image of the AlSi11,6Mm4 alloy ribbon surface cast at a speed of 15 m/s, wheel-facing side

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Fig. 3. Image of the AlSi11,6Mm4 alloy ribbon surface cast at a speed of 15 m/s, atmosphere-facing side

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Fig. 4. Image of the AlSi11,6Mm4 alloy ribbon surface cast at a speed of 30 m/s, wheel-facing side



Fig. 5. Image of the AlSi11,6Mm4 alloy ribbon surface cast at a speed of 30 m/s, atmosphere-facing side

### 4. Measurement of the cast ribbon thickness

The cast ribbons were stacked in a loose heap, from which representative samples of different lengths were randomly selected. On strips cast at different speeds of the wheel, thickness was measured with a micrometre to find a relationship between this parameter and the casting speed.

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Carting nonrestant	Alloy					
Casting parameters	AlSi11,6	AlSi11,6Mm4	AlSi11,6Mm8			
Temperature of the end of solidification (solidus point), °C	567	557	557			
Temperature of the eutectic, °C	-	573	573			
Temperature of the beginning of solidification (liquidus point), °C	578	622	690			
Melt pouring temperature, °C	640	680	750			
Temperature of nozzle - valve, °C	640	680	750			
Rotational speed of the wheel, rev/min	560; 840; 1120					
Linear speed of the cast ribbon, m/s	15; 22,5; 30					
Diameter of the nozzle, mm	1.2					
Gas overpressure during casting, MPa	0.03-0.04					
Temperature of the wheel during casting, °C	20					

Casting parameters of the investigated alloys



High scatter of the thickness values in produced ribbons was the reason why for each alloy one thousand thickness measurements were taken. So large number of measurements was necessary to accurately define the range of the strip thickness values obtained for selected casting speeds. The resulting thickness bands (Fig. 6) characterize the examined material and allow estimating the grain size gradient.

Below some examples are given of the plotted relationship between linear casting speed and scatter of thickness values in the tested AlSi11,6Mm8 alloy strips.



Fig. 6. The scatter of thickness values in an AlSi11,6Mm8 alloy strip  $[\mu m]$  as a function of the linear casting speed [m/s]



Fig. 7. The frequency of occurrence of different AlSi11,6Mm8 alloy strip thickness values as a function of the linear casting speed

With the increasing casting speed of the examined alloys, a decrease in the thickness of the ribbons and in the scatter of the measurement results was observed.

The thickness of ribbons cast from the Si11,6 alloy was measured for the five different casting speeds; in the case of the Si11,6Mm4 alloy it was measured for the three speeds (Table 3).

The averaged results of the thickness measurements taken on strips made from the tested alloys and examined as a function of the linear speed of casting are plotted in Figure 15.



Fig. 8. Average thickness of strips vs linear casting speed

With double increase in the linear casting speed from 15 m/s to 30 m/s, both tested alloys showed a nearly two-fold decrease in the thickness of the cast strips, i.e. from about 120  $\mu$ m to about 60  $\mu$ m. The thickness of strips produced from the three different alloys assumed the values of 40-200  $\mu$ m (0.05-0.2 mm), while their width was in the range of 1000-2500  $\mu$ m (1-2.5 mm).

#### 5. X-ray diffraction method

Using X-ray diffraction method (Scherrer method), the size of the crystallites was measured. Below there are X-ray diffraction patterns of an Al-Si alloy cast by the conventional method (Fig. 9) and by the method of Rapid Solidification (Fig. 10); red colour indicates the diffraction reflections from silicon.



Fig. 9. The X-ray diffraction pattern of Al-Si alloy cast by conventional process

TABLE 3

The results of measurements of the cast strip thickness

Alloy	AlSi11,6		AlSi11,6Mm4			AlSi11,6Mm8			
The linear strip casting speed, m/s	15	22,5	30	15	22,5	30	15	22,5	30
Average thickness, µm	127	80	68	121	71	64	119	77	64
Maximum thickness, µm	197	119	91	188	115	87	176	106	90
Minimum thickness, µm	85	62	50	80	56	44	80	55	44
Standard deviation, µm	26	8	9	24	9	11	17	11	10

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Fig. 10. The X-ray diffraction pattern of Al-Si alloy cast by RS (35Hz) at a linear casting speed of 30 m/s

Tests have proved that with the increasing casting speed, the silicon diffraction reflections are significantly broadened first and disappear next, which means that as a result of rapid solidification silicon undergoes partial or total amorphization.

For various casting speeds and for different alloys (where "0 [m/s]" means sample cast in a conventional manner into a foundry mould), the grain size was measured in nanometres on both sides of the strip (Table 4).

TABLE 4 The size of crystallites in ribbons cast at different speeds

	Linear speed, m/s					
Alloy / Side	0	15	22,5	30		
	Size of crystallite, nm					
AlSi11,6 - atmosphere	331	234	178	178		
AlSi11,6 - wheel	331	228	154	149		
AlSi11,6Mm4 - atmosphere	276	209	168	174		
AlSi11,6Mm4 - wheel	276	147	125	124		
AlSi11,6Mm8 - atmosphere	273	187	178	167		
AlSi11,6Mm8 - wheel	273	148	133	121		



Fig. 11. Casting speed vs size of aluminium crystallites in alloys of AlSi11,6, AlSi11,6Mm4 and AlSi11,6Mm8 - atmosphere-facing side and wheel-facing side

Using X-ray diffraction method it has been demonstrated that the increase of casting speed from 0 to 20 m/s reduces the size of aluminium crystallites. At higher speeds, the crystallite size was observed to undergo certain stabilization. In the strips produced by rapid solidification it was possible to achieve two times smaller size of the aluminium crystallites compared with the size obtained in conventional casting (Fig.17). Crystallites in the wheel-facing surface layer reached the size of about 150 nm in the AlSi11,6 alloy without the addition of mischmetal and approx. 125 nm in a near-eutectic silumin with the addition of mischmetal. In the sub-surface layer of strips on the atmosphere-facing side, crystallites in the tested alloys have reached the size of about 180 nm. The addition of rare earth elements has reduced the size of the aluminium crystallites. The measurements and calculations were conducted in accordance with the fundamental principles of the Scherrer method [5, 6, 7].

# 6. Metallography of microstructure on the strip cross-section

Microstructure on the strip cross-section was observed under an Olympus GX 71 optical microscope and under an FEI\_Tecnai G2 20 X-Twin transmission electron microscope. Figures 19 and 20 show the microstructure of alloys cast in a conventional manner into a metal mould with the casting cooling rate of ~ 5°C/s.



Fig. 12. Images of the microstructure of AlSi11,6 alloy cast into metal mould observed at three different magnifications



Fig. 13. Images of the microstructure of AlSi11,6Mm4 alloy cast into metal mould observed at three different magnifications



Fig. 14. Images of the microstructure of AlSi11,6Mm8 alloy cast into metal mould observed at three different magnifications

The microstructure of the examined alloys is observed to contain the precipitates of primary silicon, alpha phase and silicon eutectic. The structure of the mischmetal-containing alloy is more refined compared to the alloy without mischmetal (the assessment relates to the effect of an additive on changes in the three structural constituents). The precipitates of primary





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silicon change their shape from globular to lamellar (dark grey precipitates). The dendrites of the alpha phase (globular white fields) are more evenly distributed with the increasing content of RE elements, and the principal axes of dendrites and their arms are shorter. RE elements were also observed to exert an effect on the eutectic modification ( $\alpha$  + Si, the finest dark grey precipitates), resulting in a significant refinement and reduced length of the silicon plates.

Figures 15-16 show images of the microstructure of strips cast from the tested alloys at the two extreme speeds of 15 and 30 m/s. The cross-section of the strips shows differences in the microstructure. Areas in the lower part of the image show the strip structure from the casting wheel side, while areas in the upper part show the strip structure from the atmosphere side.



Fig. 15. Microstructure of strips cast by RS at a speed of 15 m/s, observed at a magnification of 1000×: a) AlSi11,6; b) AlSi11,6Mm4; c) AlSi11,6Mm8



Fig. 16. Microstructure of strips cast by RS at a speed of 30 m/s, observed at a magnification of 1000x: a) AlSi11,6; b) AlSi11,6Mm4; c) AlSi11,6Mm8

It was found that as a result of higher speed of the heat dissipation, the structure of the strip from the wheel side was more refined compared to the strip structure from the atmosphere side.

It was also found that an increase in the strip casting speed has contributed to the reduction of the strip thickness and width, and increased the structure refinement on the cross-section. The addition of rare earth elements to AlSi11,6 alloy has contributed to a significant structure refinement and changed the morphology of the precipitates of different structural constituents compared with alloys solidifying in a metal mould.

To illustrate the microstructure on the cross-section of an RS strip and to confirm the size of aluminium crystallites measured by the Scherrer method, photographs were taken with an FEI\_Tecnai G2 20 X-Twin transmission electron microscope.



Fig. 17a, b, c, d. Microstructure of AlSi11,6Mm8 alloy strips cast at a speed of 15 m/s  $\,$ 

The images in Figure 17 a, b, c, d show the occurrence of areas of aluminium grains with the silicon eutectic and finedispersed particles inside these grains. The image in Figure 17b, 17c shows the same area of the structure, but taken at a different angle of sample inclination relative to the incident electron beam. The measured average grain diameter in a selected area of the sample was 1300 nm, while the average crystallite size, which is the area of the aluminium grains with undefected internal structure, was close to 200 nm. This statement is consistent with the results of the crystallite size measurement taken by the X-ray diffraction method known as Scherrer method.

#### 7. Summary and conclusions

- 1. Changing the casting speed (the peripheral speed of the wheel) affects the surface topography of cast strips.
- 2. With the casting speed increasing in the range of 15-30 m/s, a decrease was observed in the thickness of strips cast from the examined alloys.
- 3. The X-ray diffraction method showed that with the casting speed increasing to 20 m/s, the size of aluminium crystallites was decreasing. At higher speeds, a stabilization was observed to occur.
- 4. The addition of rare earth elements to AlSi11,6 alloy reduced the size of the crystallites.
- 5. Tests and X-ray examinations of the microstructure obtained in produced strips show that both the increased casting speed as well as the addition of rare earth elements result in a significant structure refinement.

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