www.czasopisma.pan.pl PAN www.journals.pan.pl

ARCHIVES
of
FOUNDRY ENGINEERING
DOI: 10.2478/afe-2014-0037
Published quarterly as the organ of the Foundry Commission of the Polish Academy of Sciences

DE GRUYTER OPEN
ISSN (2299-2944)
Volume 14
Issue 2/2014
57 – 62

The Production of Material with Ultrafine Grain Structure in Al-Zn Alloy in the Process of Rapid Solidification

M. Szymanek[a]*, B. Augustyn[a], D. Kapinos[a], S. Boczkal[a], J. Nowak[b]
[a] Institute of Non Ferrous Metals, Light Metals Division, 19 Pilsudskiego Street, 32-050 Skawina, Poland
[b] AGH University of Science and Technology Faculty of Foundry Engineering in Cracow Poland
*Corresponding author. E-mail address: mszymanek@imn.skawina.pl

Received 20.03.2014; accepted in revised form 30.03.2014

Abstract

In the aluminium alloy family, Al-Zn materials with non-standard chemical composition containing Mg and Cu are a new group of alloys, mainly owing to their high strength properties. Proper choice of alloying elements, and of the method of molten metal treatment and casting enable further shaping of the properties. One of the modern methods to produce materials with submicron structure is a method of Rapid Solidification. The ribbon cast in a melt spinning device is an intermediate product for further plastic working. Using the technique of Rapid Solidification it is not possible to directly produce a solid structural material of the required shape and length. Therefore, the ribbon of an ultrafine grain or nanometric structure must be subjected to the operations of fragmentation, compaction, consolidation and hot extrusion.

In this article the authors focussed their attention on the technological aspect of the above mentioned process and described successive stages of the fabrication of an AlZn9Mg2.5Cu1.8 alloy of ultrafine grain structure designated for further plastic working, which enables making extruded rods or elements shaped by the die forging technology. Studies described in the article were performed under variable parameters determined experimentally in the course of the alloy manufacturing process, including casting by RS and subsequent fragmentation.

Keywords: Innovative foundry technologies and materials, Aluminium alloys, Solidification process, High aluminium zinc alloys, Rapid solidification

1. Introduction

Aluminium alloys from the 7XXX series are structural materials attractive to designers because of high mechanical properties and low values of apparent density ranging between 2.7 and 2.8 g/cm^3. Materials with such parameters are used in the automotive industry and in transport by land and air. Recalling the beginnings of the development of aluminium and its alloys we should go back to 1808 when Sir Humphrey Devy discovered aluminium. The following years raised its popularity, and it started to be used on a wide scale to make jewellery, and later dishes. The beginnings of the twentieth century led to rapid development of aluminium alloys, when Alfred Wilm accidentally discovered the possibility of increasing the strength of alloys by spontaneously occurring aging process. The driving force for further development of aluminium alloys was the demand from aviation for new lightweight metallic materials. In forties of the past century, studies conducted on the process of alloy strengthening and designing of new alloys resulted in the formation of Al-Zn alloy systems [1-2]. Introduced to the market, these materials fuelled a dynamic development of the casting and plastic working technologies, while sixties of the past century saw the invention of Rapid Solidification, where during casting the liquid metal solidification rate can reach 10^6 K/s. The solidification rate so high allows obtaining a material with the submicron or amorphous structure [3-8]. The basic empirical backgrounds of the process are comprised in the Hall-Petsch equation (1) expressing a relationship between the grain size and strength properties of the material obtained:

$$\sigma_{yd} = \sigma_0 + K_y d^{-1/2} \qquad (1)$$

where:
σ_0 - the stress required to set individual dislocations in motion independent of the grain size,
K_y - the Hall-Petch parameter, the stress intensity factor for plastic working, depending mainly on the temperature and strain rate; it strongly increases with the increasing amount of alloying elements,
d - the average grain diameter [9].

The grain size in the alloy obtained by this method assumes the values from nano- to micrometres. Such materials can be strengthened by heat treatment, if they contain reinforcing phases, or by grain boundaries, according to a) dislocation pile-up model, or b) dislocation density model [9].

The method for obtaining the RS material in a casting process comprises feeding of a thin stream of molten copper onto the rotating copper wheel with the commonly used casting speed of 50 m/s. The product of melt spinning is a thin ribbon, which in the case of alloys cast is characterised by a width comprised in the range of 1400-3500 µm and a thickness of 50-150 µm. In this form it is an intermediate product for further processing steps such as fragmentation, cold compaction, consolidation and hot extrusion yielding the solid components or, eventually, parts shaped by die forging [7, 8, 10-12]. To improve its strength properties, the alloy is heat treated, e.g. to the T6 condition which is obtained by solutionising and artificial aging, producing the structural material with high mechanical properties. Due to the specific conditions, the method of Rapid Solidification allows the manufacture of materials with ultrafine grain structure, using alloys of standard and non-standard chemical composition. Rapid Solidification has also another advantage, and it is the possibility to make materials with properties unattainable by conventional processes [11-14].

2. Methodology of the research

The authors have undertaken the task of making an aluminium-based alloy characterised by ultrafine grain structure with additions of zinc and magnesium, which is expressed by the following formula: AlZn9Cu1.8Mg2.5 (numbers refer to the average content of elements in mass percent). The resultant alloy with a non-standard chemical composition (Table 1) was cast in a melt spinning device yielding a product in the form of ribbon. The next operation was fragmentation of the ribbon in a mill in a special scissor cutting system to obtain chips of required granulation.

2.1. Steps of the technological process

The method to manufacture the alloy in the form of ribbons of ultrafine grain structure proceeds according to a flow diagram shown below (Figs. 1, 2, 3). The stock obtained in the three consecutive stages of the process is an intermediate product for further plastic working to obtain a solid item.

STEP 1 Alloy components were pure metallic constituents such as A8 primary aluminium (99.8% Al - 1080), Zn - as a main alloying element, and Cu and Mg. The alloy was melted in an induction crucible furnace from a 25 kg charge, observing the required thermal regime, the sequence of introducing the individual alloying elements, and the time of their melting (Fig. 1). After the introduction and melting of alloying elements, the melt was additionally subjected to a gas refining treatment (Fig. 1).

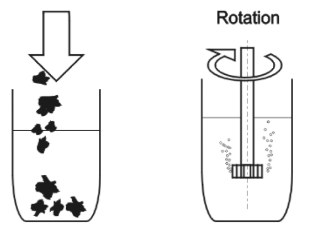

Fig. 1. Alloy manufacture

After sampling, the content of individual elements was determined using a Baird DV6 optical emission spectrometer. The resulting chemical composition shown in Table 1 was determined in a final chemical analysis of the material poured into ingot moulds to form 2 kg ingots, used next as a feedstock in the melt spinning process.

Table 1.
Chemical composition

Elemen	Zn	Cu	Mg	Cr	Fe	Si	Ni
[wt. %]	9.00	1.81	2.55	0.22	0.10	0.07	0.01

STEP 2 The second step in the technological process was casting of ribbons from the non-standard AlZn9Cu1.8Mg2/5 alloy in the Rapid Solidification process using a water-cooled copper wheel with a diameter of 500mm and a width of 70mm. The melt spinning device used in the experiment was provided with an optional system to cast aluminium alloys, designed and constructed in earlier projects (Fig. 2).

Feeding of liquid alloy onto the surface of the rotating wheel is done from the top via an ejector nozzle and a gas cushion pushing the melt up. The RS holding furnace is used in the casting process to stabilise the temperature of molten alloy, while melting of ingots is carried out in an induction furnace co-operating with the RS device to allow rapid melting of the charge combined with intense stirring of the melt. Owing to this melting regime it is possible to reduce holding of metal and segregation of alloying elements in a resistance furnace which forms part of the equipment.

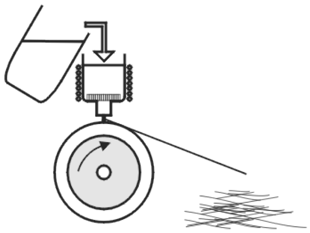

Fig. 2. Alloy cast by melt spinning

The temperature of alloy pouring was in the range of 720-725ºC (Fig. 3 and Table 2) and was selected from the CC and FD curves based on the results of the thermal analysis of the tested alloy performed on a UMSA5/MTC_MG Universal Metallurgical Simulator and Analyzer. The melt superheating temperature of 94-99°C protected zinc and magnesium contained in the alloy from rapid oxidation while maintaining good fluidity.

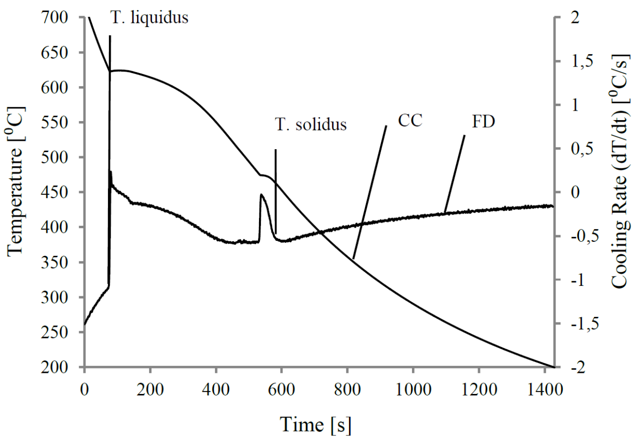

Fig. 3. Summary of the cooling curve (CC) and its first derivative (FD) plotted for the AlZn9 alloy cast into metal mould

Table 2.
The results of thermal analysis obtained for the cooling cycle of AlZn9 alloy

Thermal characteristic	[^{0}C]
Start of the crystallization (liquidus)	626
End of the crystallization (solidus)	470

The linear speed of the ribbon casting was selected by experiments and kept at a level of 36 m/s. Ribbons were also cast at a speed of 30 and 41 m/s. The pressure ejecting the melt through a dispensing nozzle did not exceed 0.35 bar. Owing to these casting parameters it was possible to produce each time the ribbon shown in a bulk form in Fig. 4.

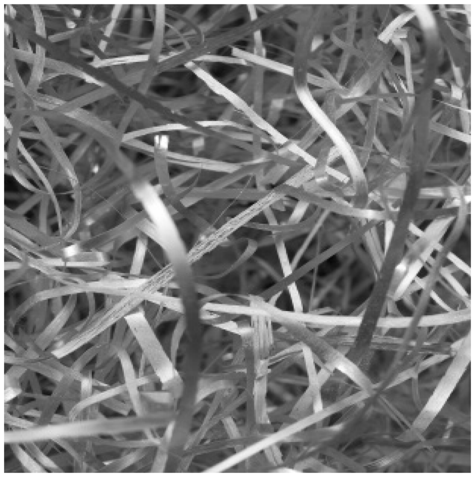

Fig. 4. Bulk form of the ribbon cast

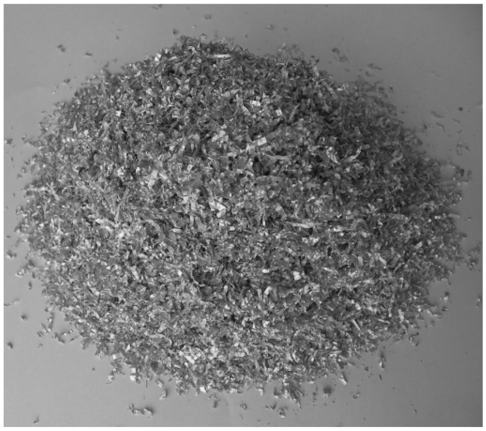

Fig. 5. Cast ribbons fragmented to the form of chips

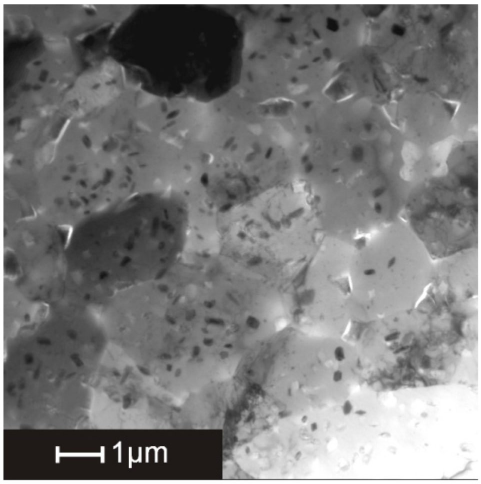

Fig. 6. Microstructure of ribbon cast by RS from the AlZn9 alloy

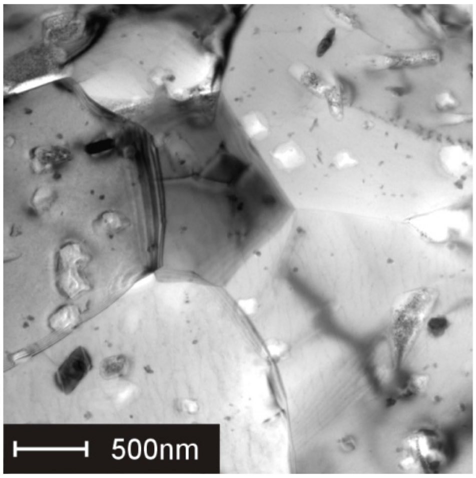

Fig. 7. Microstructure of ribbon cast by RS from the AlZn9 alloy. Examination made by TEM TECNAI G2 with EDX attachment, STEM and HAADF detector

STEP 3 The last step in the process is fragmentation of the cast ribbon obtained by melt spinning. The ribbon in as-cast state is not fit for consolidation and must be sectioned into smaller fragments. This operation is performed in a mill operating on the principle of cutting scissors with a rotor speed of 580 rev/min (Fig. 8).

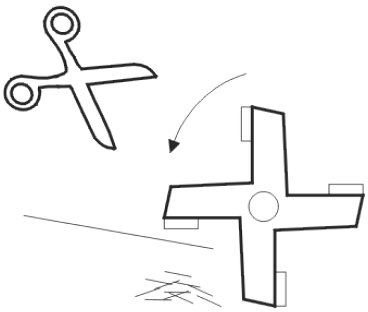

Fig. 8. Ribbon fragmentation

Additionally, the mill chamber was equipped with a 1mm mesh classifying screen, allowing manufacture of the material with grain size below this value (Fig. 12).

3. Discussion of results

During various stages of the manufacturing process, the required materials were successfully produced, including alloy of a homogeneous chemical composition, produced in the form of ingots, ribbons cast by RS, and chips for further plastic working. The auxiliary equipment has met the anticipated requirements. It is worth mentioning that alloys for the melt spinning process must be of high purity, while the process of melting and remelting should be as short as possible to reduce oxidation. Casting of the AlZn9Cu1.8Mg2.5 alloy in a melt spinning device produced ribbons shown in Fig. 9.

The casting parameters adopted in the process of the ribbon fabrication, i.e. the casting temperature of 720-725°C, the gas cushion pressure of 0.35 bar, the selected nozzle diameter and the casting speed of 36 m/s, produced a ribbon of the best quality described with graphs shown in Figs. 10, 11 and 12 and in Tables 2 and 3. Ribbons cast at a higher speed of 41 m/s had jagged edges, uneven width and local material discontinuities. On the other hand, the lower casting speed of 30 m/s has yielded the material of higher thickness, unstable in respect of the product length as verified by the gauge measurements and visualised by the methods of statistical analysis. During casting at the lowest speed, some problems occurred associated with handling of the liquid alloy onto the mould surface, ultimately resulting in the process instability. Therefore, the authors selected the best material for further research, which has proved to be the ribbon cast at a speed of 36 m/s (Fig. 9).

An image of the cast ribbon surface was obtained using an Olympus GX71 optical microscope at a magnification of 50x.

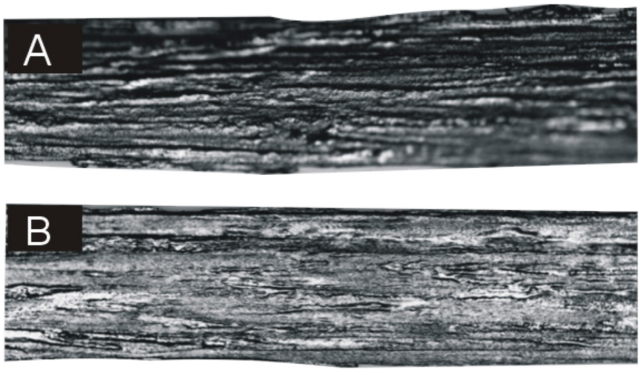

Fig. 9. View of the ribbon surface: A – atmosphere side; B – Cu wheel side

It is easy to note the difference in surface morphology between the ribbon side contacting the atmosphere (Fig. 9A) and the wheel (Fig. 9B).

The ribbon thickness was determined by measurements taken with a micrometer, while the width was determined by measurements taken with an optical microscope. The values obtained were compared and subjected to statistical analysis. The selected results are shown below.

Table 3.
The thickness of ribbons cast from AlZn9 alloy

Unit	[μm]
Mean	83
Standard deviation	23
Maximum	282
Minimum	34

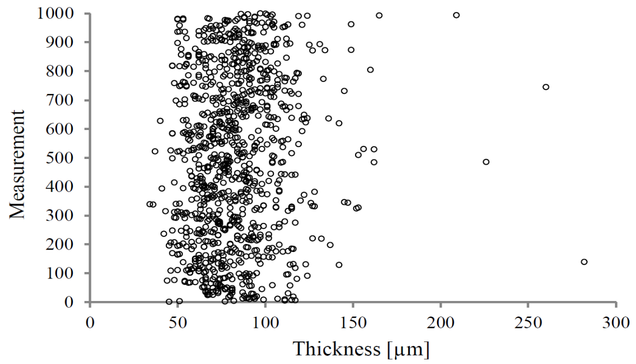

Fig. 10. A scatterplot of the ribbon thickness values

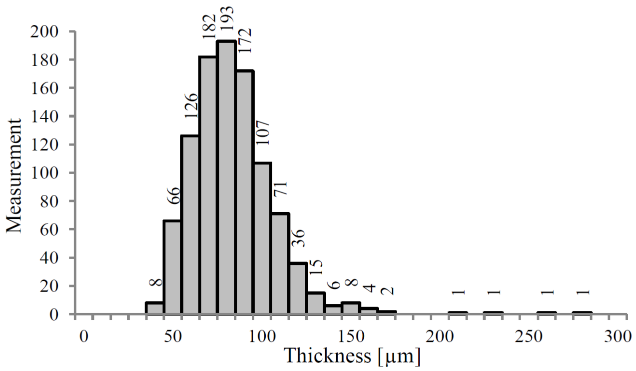

Fig. 11. Histogram showing the distribution of class sizes of the cast ribbon thickness values

Table 4.
The width of ribbons cast from AlZn9 alloy

Unit	[µm]
Mean	2482
Standard deviation	150
Maximum	2990
Minimum	2031

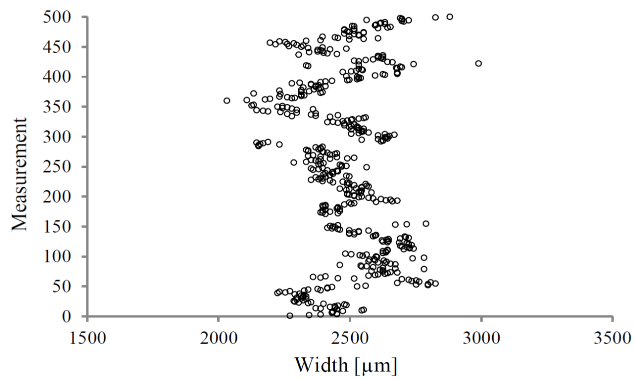

Fig. 12. A scatterplot of the ribbon width values

Analysing the scatterplots it can be concluded that the measurement results form a band comprised in the range of 2200-2800 µm for the thickness (Fig. 10) and 40-150 um for the width (Fig. 12). The destermined number of class intervals for the ribbon thickness makes one mode (modal value), and also shows the range of the prevalence of variable (measured value).

The two-stage process of the ribbon fragmentation (Fig. 8) in a mill operating in a scissor-cutting mode at a speed of 580 rev/min enabled sectioning the ribbon to a required fraction thickness (Fig. 13). The ribbon cutting operation was performed without the occurrence of any adverse effects, such as the ribbon seizure in a fragmentation chamber, clustering of fine particles into lumps, and welding to the mill elements. The evaluation of particulate material was performed by sieve analysis to determine the effectiveness of the fragmentation process and percent content of individual fractions. The two-stage fragmentation process enabled obtaining 94% of material fraction with the grain size comprised in a range of 0.2-1 mm. By calculating the apparent and bulk density of the fragmented ribbons, an average density of 570 g/dm^3 and 782 g/dm^3 was obtained (Fig. 14). Ultimately, the process of compaction and plastic working should give the density of the solid material at a level of 2600-2800 g/dm^3.

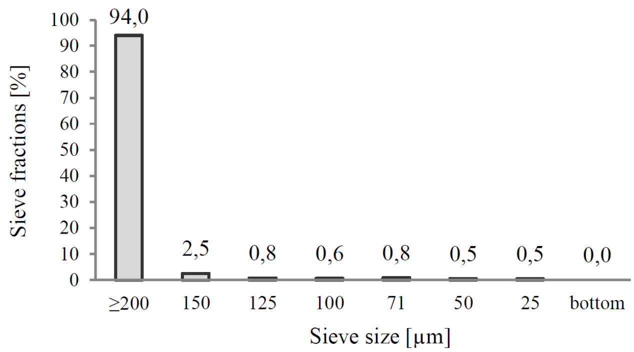

Fig. 13. Graph showing the content of ≤200 µm fractions calculated by the granulometric measurements conducted on the crushed AlZn9 alloy chips

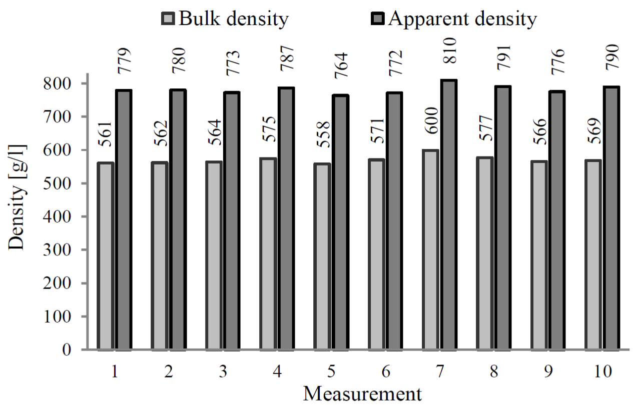

Fig. 14. Graph showing bulk and apparent density of crushed chips determined for 10 measurements

Evaluating the particulate material, it can be stated that this material meets the requirements imposed by the target application, which is the manufacture of compacts in a 250T press and consolidation in a 500T press or extrusion by CRE (Continuous Rotary Extrusion) to the form of solid rod.

4. Conclusions

The stock obtained in the subsequent stages of the process was compacted and subjected next to plastic working. The process of casting in a melt spinning device carried out at a crystallisation rate of up to 106 K/s yielded the material with a submicron grain size (Fig. 6). Using Hall-Petsch relation, it can be expected that, consolidated into a solid rod, the alloy will reach the strength higher than the strength obtainable by common methods. One can also assume that a well-conducted strengthening heat treatment will further raise the mechanical properties.

Evaluating the particulate material, it can be stated that it meets the requirements imposed by the target application, which is the production of compacts in a 250T press and consolidation in a 500T press or extrusion by CRE to the form of solid rod.

If further studies will result in still higher mechanical properties of the alloy, it can be used as a structural material. The use of material with higher strength for a given element automatically reduces the weight of this element. The reduced curb weight of a vehicle, components and parts included, will reduce the level of fuel consumption, air emissions and operating costs.

Acknowledgements

The study was financed from a Strategic „ZAMAT" Project No. POIG.01.03.01-00-015/09 entitled "Advanced materials and technologies for their production", co-financed from the structural fund; the project implementation period is 2010-2014.

References

[1] Kurzydłowski, K. J. (2010). The development of structural materials; the history of aluminium alloys. *Przegląd Mechaniczny* Proceedings of Jubilee Session (in Polish).

[2] Dobrzański, L. A. (2006). *Engineering materials and materials design* (pp. 642-658). Warszawa: WNT (in Polish).

[3] Praisner, T. J. Chen, J. S. & Tsenga, A. A. (1994). Experimental Study of Process Behavior in Planar Flow Melt Spinning. *Metallurgical and Materials Transactions B. Vol. 26B,* 1995-1199.

[4] Lityńska-Dobrzyńska, L. Dutkiewicz, J. Maziarz, W. & Góral, A. (2011). Microstructure of Rapidly Solidified Al-12Zn-3Mg-1.5Cu Alloy with Zr and Sc Additions. *Materials Transaction*, *Vol. 52,* 309-314.

[5] Cieślak, G. Latuch, J. & Kulik, T. (2006). Producing massive nanocrystalline alloys from the Al-Si-Ni-Mm (mischmetal) system. *Inzynieria Materiałowa Rok XXVII. Vol. 3,* 123–126 (in Polish).

[6] Chen, H. & Yang, B. (2008). Effect of Precipitations on Microstructures and Mechanical Properties of Nanostructured Al-Zn-Mg-Cu Alloy. *Materials Transactions. Vol. 49, No. 12,* 2912-2915.

[7] Szymanek, M. Augustyn, B. Szymański, W. & Kapinos, D. (2013). The Production of Wrought AlSi30Cu1.5Mg1.2Ni1.5 Alloy with Ultrafine Structure. *Light Metals 2013*, 521-525.

[8] Augustyn, B. Szymanek, M. Kapinos, D. & Pakieła, W. (2014). Evaluation of Functional Properties of the Rapidly Solidified Cast AlSi30 Alloy as a Material for Transport Applications. *Light Metals 2014*, 975-980.

[9] Muszka, K. (2008). *The effect of structure refinement on the strengthening mechanism of low-carbon plastically deformable steel.* Unpublished doctoral dissertation, AGH University of Science and Technology, Kraków, Polska. 9-41 (in Polish).

[10] Carpenter, J. K. & Steen, P. H. (1992). Planar-flow spin-casting of molten metals: process behavior. *Journal of Materials Science. Vol. 27*, 215-225.

[11] Szymański, W. Szymanek, M. Żelechowski, J. Bigaj, M. Gawlik, M. & Płonka, B.(2013) Alloy AlSi30 Cast in the Process of Rapid Solidification and Consolidated in the Process of Plastic Forming. *Light Metals 2013*, 333-337.

[12] Kapinos, D. Szymanek, M. Augustyn, B. & Gawlik M. (2013). Effect of heat treatment on the mechanical properties of wrought Al-Zn-Mg-Cu alloy cast by Rapid Solidification. *Materials Science Forum. Vol. 765*, 496-500.

[13] Sundararajan, A. & Thomas, B. G. (2008). Heat Transfer During Melt Spinning of Al-7%Si Alloy on a Cu-Be Wheel. *Light Metals 2008,* 793-810.

[14] Naplocha, K. & Kaczmar, J. W. (2011). Tribological properties of Al 7075 alloy based composites strengthened with Al_2O_3 fibres. *Archives Of Foundry Engineering 2/2011. Vol. 11,* 153-158.