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The Production ofMaterial withUltrafine Grain Structure in Al-Zn Alloyin the Process ofRapid Solidification

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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 ofan 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 fabricationof anAlZn9Mg2.5Cu1.8 alloy of ultrafinegrain 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 castingby RS and subsequent fragmentation.

Keywords: Innovative foundry technologies and materials, Aluminiumalloys, Solidification process, High aluminium zinc alloys, Rapidsolidification

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, studiesconducted onthe process of alloy strengthening and designing of new alloys resulted in the formation of Al-Zn alloysystems [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:

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

where:
σ_0 - the stress required to set individualdislocationsin motion independent of the grain size,
K_y - the Hall-Petchparameter, 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 valuesfrom nano- to micrometres.Such materialscanbestrengthenedbyheat treatment,if they containreinforcing phases,or bygrain boundaries, according to a)dislocation pile-upmodel, orb) 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 alloyscast 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 productfor further processing steps such as fragmentation, cold compaction, consolidation and hot extrusionyielding 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 conditionwhich is obtained bysolutionising 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 ultrafinegrain 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 ultrafinegrain 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 tomanufacture the alloyin the form ofribbons of ultrafinegrainstructure proceeds according to a flow diagramshown below(Figs. 1, 2, 3).The stockobtained in thethree consecutive stagesof the process isan 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 toa gas refining treatment (Fig. 1).

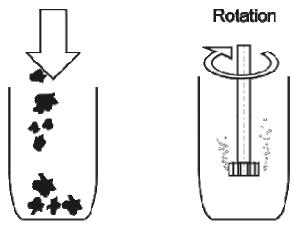

Fig. 1. Alloy manufacture

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 meltspinning 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

STEP2 The second stepin the technological process was casting of ribbons from the non-standard AlZn9Cu1.8Mg2/5 alloy in theRapidSolidificationprocessusing a water-cooled copper wheel with a diameter of500mm and a width of70mm. The meltspinning device used in the experimentwas provided with an optional systemtocastaluminium alloys, designed andconstructedin earlierprojects (Fig. 2).

Feeding ofliquid alloyonto the surfaceof the rotatingwheelis donefrom the topviaanejector nozzleanda gas cushion pushing the melt up. The RS holding furnaceis used in the casting processtostabilise thetemperature of moltenalloy, while meltingof ingotsis carried out in an induction furnace co-operatingwith the RS device toallowrapid melting ofthe charge combined with intense stirring of the melt. Owing to this melting regime it is possible to reduce holding of metal andsegregation ofalloying elementsin aresistance furnace which forms part of the equipment.

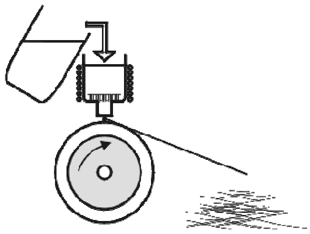

Fig. 2. Alloy cast by melt spinning

Thetemperature 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.

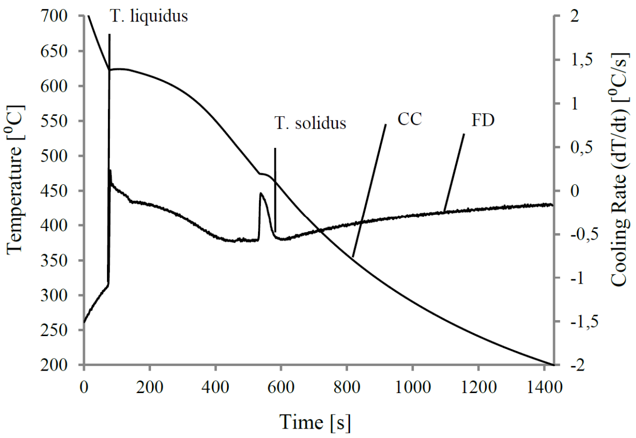

Fig.3. Summary of the cooling curve (CC) and its first derivative (FD) plotted for the AlZn9 alloy cast into metal mould

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.

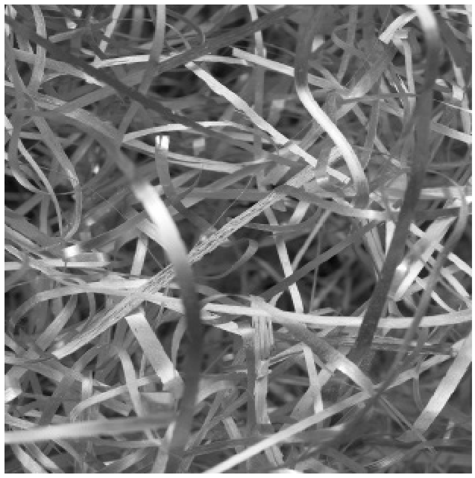

Fig. 4. Bulk form of the ribboncast

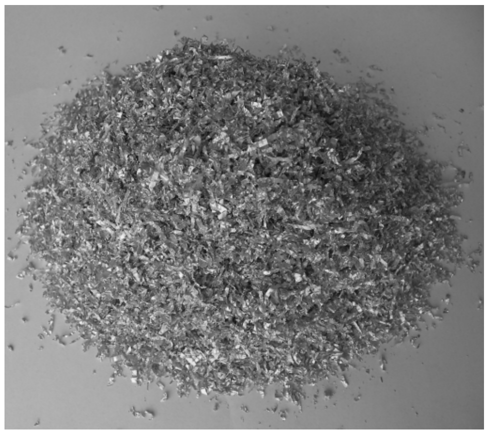

Fig. 5. Cast ribbons fragmented to the form of chips

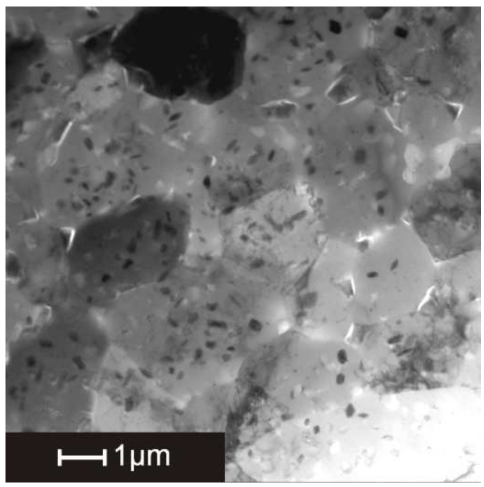

Fig. 6. Microstructure of ribbon cast by RS from the AlZn9 alloy

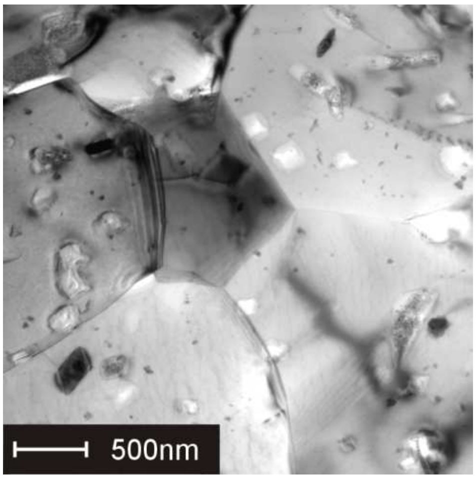

Fig. 7. Microstructure of ribbon cast by RS fromthe AlZn9 alloy. Examination made by TEM TECNAI G2 with EDX attachment, STEM and HAADF detector

STEP3 The laststep in the processis fragmentation ofthe cast ribbonobtainedby meltspinning.Theribbon in as-cast state is not fit forconsolidation and must be sectioned into smaller fragments.This operation isperformed in amilloperatingon the principle ofcuttingscissors witha rotorspeed of580rev/min(Fig. 8).

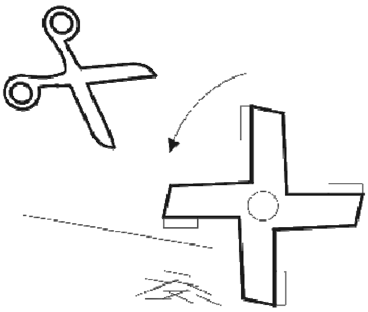

Fig. 8.Ribbon fragmentation

Additionally,the millchamberwas equippedwith a 1mm mesh classifying screen, allowing manufacture of the materialwith 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 ahomogeneous 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, produceda ribbon of the best quality described withgraphs shown in Figs.10, 11 and 12 andin Tables 2 and 3. Ribbonscast 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 asverified 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 ribbonsurface was obtained using an Olympus GX71optical microscope at a magnification of 50x.

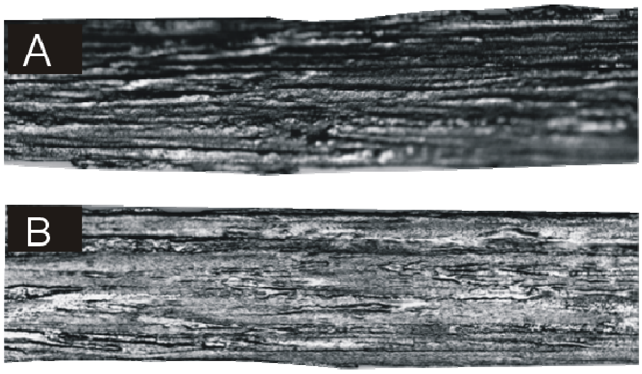

Fig. 9. View ofthe ribbon surface: A – atmosphere side; B –Cu wheel side

It is easy tonote the difference in surfacemorphologybetween the ribbonside contacting the atmosphere(Fig.9A) andthe wheel(Fig.9B).

The ribbon thickness was determinedbymeasurements taken with a micrometer, while the widthwas determinedby measurements taken withan optical microscope. The valuesobtained were compared andsubjected tostatistical analysis.The selectedresultsare shown below.

Table 3.
The thickness of ribbons cast from AlZn9 alloy

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

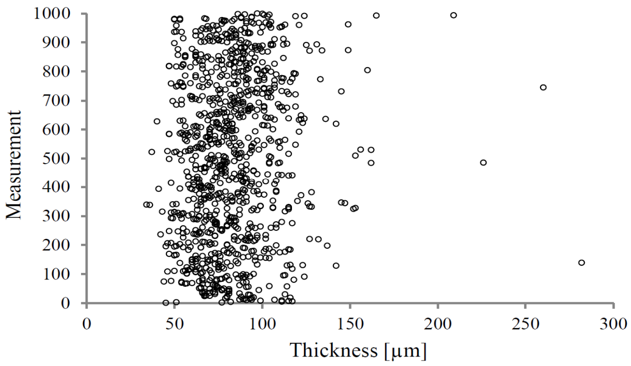

Fig. 10. A scatterplot of the ribbon thickness values

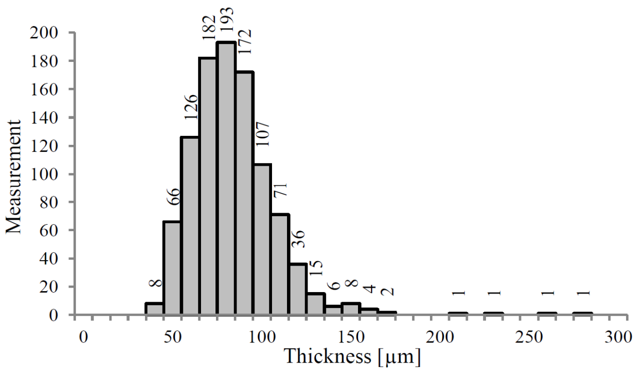

Fig. 11. Histogramshowing thedistribution ofclass sizesofthe cast ribbonthicknessvalues

Table 4.
The width of ribbons cast from AlZn9 alloy

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

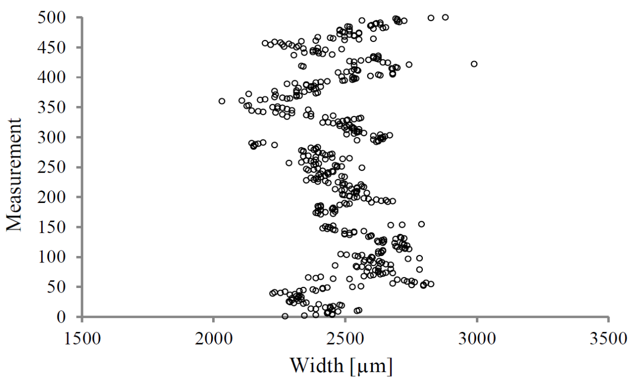

Fig. 12. A scatterplot oftheribbon width values

Analysing thescatterplotsit can be concludedthat themeasurement resultsform aband comprisedin the range of2200-2800µm for the thickness(Fig. 10)and 40-150um for the width (Fig. 12). The desterminednumber ofclassintervals for the ribbon thicknessmakes onemode(modal value), and also showsthe range ofthe prevalence of variable (measured value).

The two-stageprocessof the ribbon fragmentation (Fig.8) in a milloperating in a scissor-cutting mode at a speed of 580rev/minenabled 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 evaluationof particulate materialwas performed by sieve analysistodetermine the effectiveness ofthe fragmentation process andpercent content ofindividual fractions.The two-stage fragmentation process enabledobtaining 94% of material fractionwith the grain sizecomprised in a range of0.2-1mm. By calculating the apparent andbulk density of thefragmentedribbons, an average density of570g/dm^{3}and782g/dm^3 was obtained (Fig.14).Ultimately,theprocessof compaction and plastic working should give the density of the solid materialat a level of 2600-2800g/dm^3.

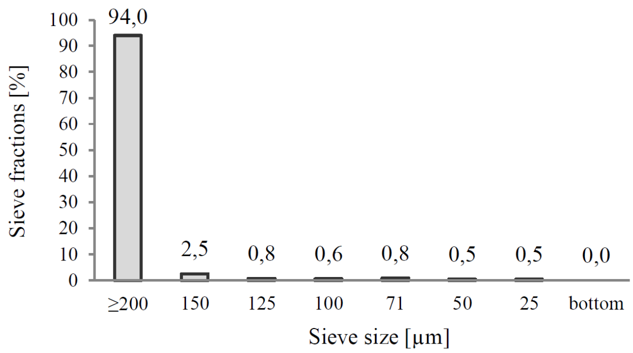

Fig. 13. Graph showing the content of ≤200 µm fractions calculated by the granulometric measurements conducted on the crushed AlZn9 alloy chips

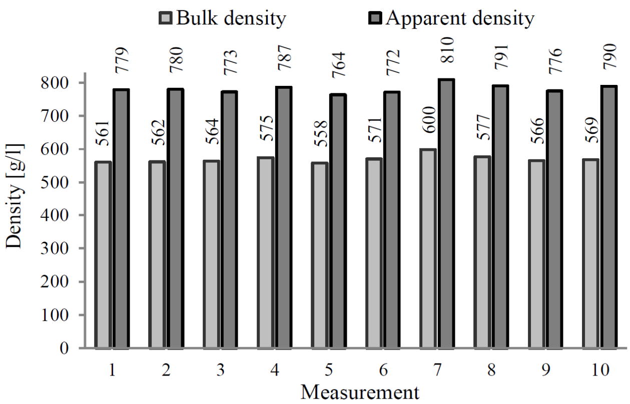

Fig. 14. Graph showing bulk and apparent density of crushed chips determined for10 measurements

Evaluating theparticulate material, it can be statedthat this material meets therequirementsimposed bythe targetapplication, which is the manufacture of compacts in a250T press andconsolidationin a 500T press orextrusion byCRE(ContinuousRotaryExtrusion) to the form of solidrod.

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 alloywill 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 a500T 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 reducedcurb weight of a vehicle, components and parts included, will reduce the level offuel consumption, air emissions and operating costs.

Acknowledgements

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References

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

[2] Dobrzański, L. A. (2006). *Engineering materials and materials design.* Warszawa: WNT.

[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. 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*. 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*. 3, 123-126.

[6] Chen, H. & Yang, B. (2008). Effect of Precipitations on Microstructures and Mechanical Properties of Nanostructured Al-Zn-Mg-Cu Alloy. *Materials Transactions.* 49(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 forTransport Applications. *Light Metals.* 2014, 975-980.

[9] Muszka, K. (2008). *The effect ofstructure refinementonthe strengthening mechanism of low-carbon plastic allydeformable steel.* Unpublished doctoral dissertation, AGH University of Science and Technology, Kraków, Polska.

[10] Carpenter, J. K. & Steen, P. H. (1992). Planar-flow spin-casting of molten metals: process behavior. *Journal of Materials Science.* 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*. 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.* 11(2), 153-158.