ARCHIVES of FOUNDRY ENGINEERING

ISSN (1897-3310)
Volume 2020
Issue 2/2020

111 – 117

19/2

Published quarterly as the organ of the Foundry Commission of the Polish Academy of Sciences

Gas Blowing Ultrasonic Aluminium Degassing Assessment with the Reduced Pressure Test (RPT) Method

H. Galarraga [a,] *, M.G. de Cortazar [a], E. Arregi [b], A. Artola [a], J.L. Oncala [a], M. Merchan [a]
[a] TECNALIA, Basque Research and Technology Alliance (BRTA,), Kurutz Gain Industrialdea 10, 20850 Mendaro, Spain
[b] Edertek S. Coop, Isasi Kalea 6, 20500 Arrasate-mondragón, Spain
* Corresponding author. E-mail address: haize.galarraga@tecnalia.com

Received 10.10.2019; accepted in revised form 13.03.2020

Abstract

Entrapped gases, solidification shrinkage and non-metallic compound formation are main sources of porosity in aluminium alloy castings. Porosity is detrimental to the mechanical properties of these castings; therefore, its reduction is pursued. Rotary degassing is the method mostly employed in industry to remove dissolved gases from aluminium melts, reducing porosity formation during solidification of the cast part. Recently, ultrasonic degassing has emerged as a promising alternative thanks to a lower dross formation and higher energy efficiency. This work aims to evaluate the efficiency of the ultrasonic degasser and compare it to a conventional rotary degassing technique applied to an AlSi10Mg alloy. Degassing efficiency was evaluated employing the reduced pressure test (RPT), where samples solidified under reduced pressure conditions are analysed. Factors affecting RPT were considered and temperature parameters for the test were established. The influence of ultrasonic degassing process parameters, such as degassing treatment duration and purging gas flow rate were studied, as well as treated aluminium volume and oxide content. Finally, ultrasonic degassing process was contrasted to a conventional rotary degassing technique, comparing their efficiency.

Keywords: Aluminium, Hydrogen, Degassing, Ultrasonic, Oxides, Reduced pressure test

1. Introduction

Dissolved hydrogen in aluminium melts is one of the main sources of porosity in aluminium alloy castings. The presence of hydrogen in aluminium melts is principally originated from the vapour in the atmosphere [1]. The water vapour reacts with the aluminium, forming aluminium oxide and gaseous hydrogen following the next equation [2]:

$$H_2O(v) + \frac{2}{3}Al = \frac{1}{3}Al_2O_3 + 2H(in\ Al)$$

During cooling and solidification of the aluminium alloy, dissolved hydrogen in excess of the extremely low solid solubility may precipitate in molecular form, resulting in the formation of porosity [3]. According to some authors, oxides can work as pore nucleation sites and therefore, they have considerable influence in the gas porosity of the solid aluminium [1]. Gas porosity reduces strength in castings, consequently, hydrogen needs to be removed from cast alloy products. Depending on the application of the casting, the minimum acceptable content of hydrogen could differ. Common general target values are around <0.2 ppm [4].

Degassing is the most effective way to reduce the porosity during casting processes. Several types of degassing methods are currently in use. The most common methods in aluminium industry consist of blowing gases like argon, nitrogen or chlorine through the melt. Hydrogen is trapped by purging gas bubbles due to the low partial pressure of hydrogen in the formed bubbles. The bubbles escape from the melt and the gas is then removed. Rotary degassing is the method generally employed to blow gases. In this technique, gases are blown through a rotary impeller creating small bubbles and increasing the efficiency of the degassing process. Ultrasonic degassing is an alternative degassing method that revealed favourable results. The potential of this technology has been supported by several published studies, showing the effectiveness of the ultrasonic treatment in molten aluminium alloys [5–12]. Ultrasonic degassing uses ultrasonic vibrations to generate oscillating pressures in molten aluminium, producing cavitation phenomena and creating fine bubbles. The bubbles produced during cavitation could provide nuclei for hydrogen bubbles to merge and flow out of the melt [13]. Ultrasonic degassing is considered a promising technology due to its reduced dross formation, absence of brittle moving parts and energy efficiency [14].

In this work, nitrogen purging gas was blown through an ultrasonic probe, creating a fragmented and dispersed nitrogen micro-bubble stream due to the ultrasonic cavitation. This combined effect generates high capacity degassing system, as it has been demonstrated for the treatment of aluminium for twin roll casting [3]. In order to study the possible application of this technology in discontinuous casting processes, several studies were accomplished in this research: ultrasonic degasser performance, influence of degassing treatment parameters, effect of the oxides in the melt, dependence of the aluminium volume, and comparison of the ultrasonic degassing to a conventional rotary degassing.

A consistent method to gauge the hydrogen level in the melt is necessary to study the performance and effectiveness of the ultrasonic degassing. The RPT (Reduced Pressure Test) method is a common tool in foundries which allows the operator to qualitatively assess the hydrogen level of a molten aluminium batch, allowing corrective action to be followed [15]. Several works [16-17] studied the influence of oxides and bifilm index in the porosity formation. Bifilm index introduced by Dispinar and Campbell [18], is calculated totalling the maximum length of pores in RPT sample sections. In this work apparent density measurement by Archimedes´ Principle was preferred due to its simplicity and transferability to foundries. Comparing measured apparent density values, the dependence of the RPT method with several parameters as the relative humidity in the atmosphere, RPT temperature, and aluminium temperature was also studied.

2. Material and methods

RPT test consists of solidifying a sample of the melt under reduced pressure. This encourages pore formation, the pores expanding due to the lowered pressure and providing a much more porous sample than under atmospheric conditions of solidification [19]. The product, a cup-shaped specimen, permits the gas level to be evaluated by visual inspection or by apparent density measuring being the latter the assessment method employed in this work

In this work, RPT samples were obtained solidifying around 150 g AlSi10Mg aluminium alloy samples with reduced pressure (100 mbar). Table 1 shows the chemical analysis of the alloy used in this work and measured by spark spectrometry. RPT system´s dome was closed and vacuum pump was activated once the aluminium reached a temperature denominated as “RPT temperature”. The procedure to measure the RPT temperature consisted in the manual introduction of 1 mm diameter type K thermocouple and control the decreasing of the temperature until the objective temperature was reached. This was the moment when the vacuum pump was activated. The density of the solidified samples, apparent density or RPT density, was measured employing the Archimedes´ Principle, where the density is calculated after measuring dry weight and immersed weight of the samples. RPT densities measured during degassing tests explained in 3.2 and 3.3 sections were accomplished with the melt bath at 800°C. In those tests vacuum was applied when the aluminium sample cooled down to 650°C (RPT temperature). Same temperatures were applied to evaluate the effect of the humidity in the RPT test.

Table 1.
Chemical analysis of the AlSi10Mg aluminium alloy

Element	Si	Fe	Cu	Mn	Mg	Ti	Al
Con. [%]	10.24	0.111	0.001	0.65	0.274	0.048	bal.

Ultrasonic degassing tests were performed with a SCR ultrasonic Ultra-D™ degasser running with 20kHz frequency. In order to avoid chemical wearing caused by molten aluminium Sialon stiff ceramic acoustic resonator or probe was used. The probe was submerged 50 mm depth in the melt.

Conventional degassing test were performed with a Foseco™ rotary degasser rotating at the nominal speed. High purity nitrogen with controlled flow rate was employed as a purging gas in both cases.

The degassing experiments were carried out in two steps. In the first step, small volume tests with only ultrasonic degasser were performed with the purpose of a better understanding of the technique, as well as to study the influence of the oxides in the degassing process. In each test performed in this step, 6kg of AlSi10Mg alloy were melted in a silicon carbide crucible (Ø150 mm x 260 mm) with an electric furnace. For the comparison between low and high oxide content melts, 100% ingot and 100% rejected parts were employed as feedstock respectively.

In the second step, 50 kg of the same alloy were melted in a medium volume electric furnace, employing also a silicon carbide crucible (Ø330 mm x 500 mm). The higher volume capacity of the furnace in this step, permitted to use both ultrasonic (Figure 1) and rotary degasser (Figure 2). In the previous step, the employment of rotary degasser was not possible because it requires more physical space than the ultrasonic degasser.

Fig. 1. Ultrasonic degasser

Fig. 2. Rotary degasser in the medium volume furnace

3. Results

Results obtained in the degassing experiments are presented and discussed below. Nevertheless, to have a solid baseline for the main research goal, principal parameters affecting the RPT degassing evaluation method were also studied.

3.1. RPT method

The dependence of the melt temperature on apparent densities obtained by the RPT method is understandable since the solubility of the hydrogen in molten aluminium increases with the temperature of the aluminium. Therefore, dissolved hydrogen content in equilibrium is higher for higher metal temperatures.

Previous RPT tests completed with constant metal bath temperatures, revealed high variation of obtained apparent density values. During the experiments, it was observed that in the RPT tests performed at high aluminium temperatures, the hydrogen dissolved in the aluminium came out of the melt before solidification due to the effect of the vacuum. This phenomenon is a degassing process itself, since the hydrogen contained in the aluminium goes out of the melt showing bubbles in the surface of the sample. Figure 3, shows a sample taken from the metal bath at 800ºC and exposed immediately to vacuum conditions.

Fig. 3. RPT sample inside the vacuum chamber with bubbling hydrogen on the surface of the aluminium taken from the metal bath at 800°C

After considering other variables that could affect the test, a new parameter denominated as “RPT temperature” was defined to analyse the scattering in the measurements of the preliminary tests. According to the deliberations, the measured apparent density depends on the metal bath temperature and just defined RPT temperature. Consequently, it is important to control the RPT temperature in order to obtain consistent measurements, and preferably perform the test below 700°C RPT temperature for this alloy, where aluminium bubbling is not expected. At higher temperature, the degassing effect of the vacuum during the RPT test can affect to the measuring purpose of the test, making it imprecise.

Once RPT temperature was taken into consideration, first test batch consisted of 16 measures performed with samples seized from 700ºC metal bath temperature and using different RPT temperatures between 580ºC and 660ºC. In the next steps, 10 measures for 750ºC, 5 measures for 775ºC, 10 measures for 800ºC, and 3 measures for 900ºC metal bath temperatures were taken for 600ºC, 625ºC, and 650ºC RPT temperatures. The results of this tests are shown in Figure 4 where apparent densities measured at different metal bath and RPT temperatures are displayed.

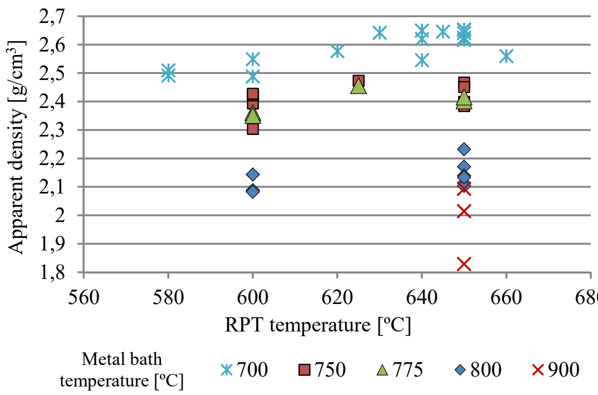

Fig. 4. Apparent density dependence of the RPT and furnace temperatures

Figure 4 results show lower densities for higher metal bath temperatures because hydrogen solubility in aluminium increases with metal bath temperature. Besides that, slightly higher densities can be appreciated as the RPT temperature is increased. This effect is attributed to the vacuum degassing process detected in previous RPT tests.

Relative humidity in the atmosphere is another variable that should be taken into consideration. Humidity plays an important role in the amount of hydrogen dissolved in the melt, since equilibrium hydrogen concentration in aluminium is proportional to the partial pressure of hydrogen gas in the atmosphere [20]. In Figure 5, the relation between ambient humidity and apparent density is analysed. The results are significantly scattered. However, it can be noticed that the highest density values are obtained when relative humidity is below 55% and in general, average measured densities are lower for higher humidity atmospheres. Hence, atmosphere humidity should be considered for thoughtful work with RPT method, avoiding big humidity changes between comparative measures.

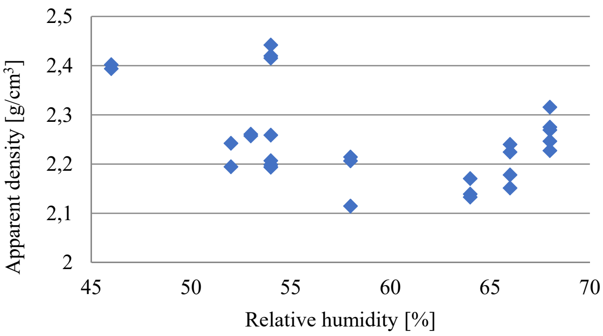

Fig. 5 Effect of the ambient humidity in the apparent densities measured with 800°C furnace temperature and 650°C RTP temperature

To minimize scattering of RPT measurements, 800°C furnace temperature and 650°C RPT temperature were set for later degassing tests. These working temperatures will provide adequate starting aluminium density values to analyse degassing parameters and processes. Relative humidity of the atmosphere was also measured in each test, avoiding extreme humidity or dryness conditions during the experimental work.

3.2. Ultrasonic degassing in small aluminium volume

With temperature settings fixed in the previous section, ultrasonic degassing tests were performed with different process parameters in 6 kg of aluminium alloy. Degassing steps were carried out with 2, 4 and 6 Nl/min purging gas flow rates and up to 10 minutes degassing treatment periods. The results in Figure 6 show that density increasing becomes very slow after 5 minutes long ultrasonic treatment. Besides that, achieved degassing levels are almost identical for the nitrogen flow rate ranges studied in this work. For 2 minutes degassing time, the highest density value is obtained with 6 Nl/min gas flow rate. However, the higher the flow rate, aluminium splashing on the melt surface was higher, most probably oxidizing the melt. It should be also noted that the initial density value difference between the 3 tests is maintained until 5 minutes of treatment time.

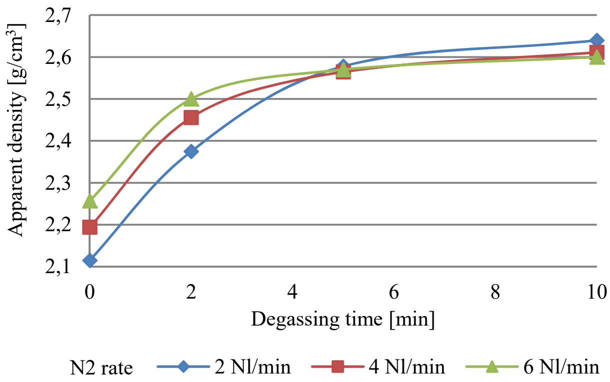

Fig. 6. Density measurements for different degassing times and purging gas flow

In the next experiments, the influence of the oxides in the degassing treatment was studied applying the same degassing parameters to low and higher oxide content melts. The terms low and higher oxide was used as qualitative description of the oxide amount estimation in the alloy, without any specific oxide quantity measurement. In this experiment, 2Nl/min N_2 purging gas flow rate was employed to avoid any addition of extra oxides to the melts due to splashing. The higher oxide content melt was obtained melting thin cross section scrap obtained by HPDC (high pressure die casting) process and the low oxide content melt was obtained melting new ingots. The results shown in Figure 7, revealed similar performance of the treatments for the oxidized and lower oxidized melts based on the parallel paths of the density/degassing curves. The offset between two curves (0,05 g/cm^3 average) is attributed to the different atmosphere humidity values measured during the tests (68% RH for the higher oxide content melt and 54 % RH for the low oxidized melt). Each point in the graph corresponds to one measurement.

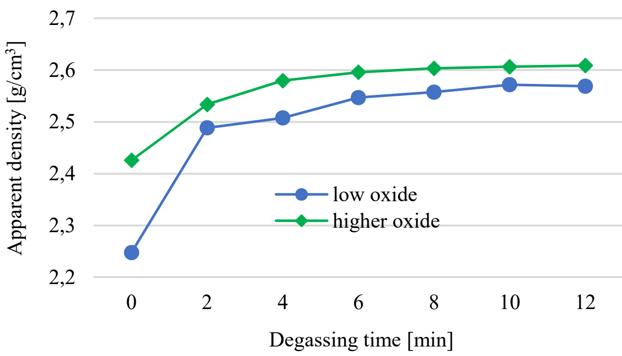

Fig. 7. Ultrasonic degassing (2Nl/min N2) comparison between low and higher oxidized melts

3.3. Effect of the aluminium volume

Degassing capacity of the ultrasonic degasser was studied and demonstrated in a small volume of aluminium alloy in the preceding step. However, the efficiency for this method is not clear for higher amount of metals. In this step, degassing was performed in 50 kg of aluminium alloy and compared to the previously performed with 6 kg of melt, using same metal bath and RPT temperatures, Figure 8.

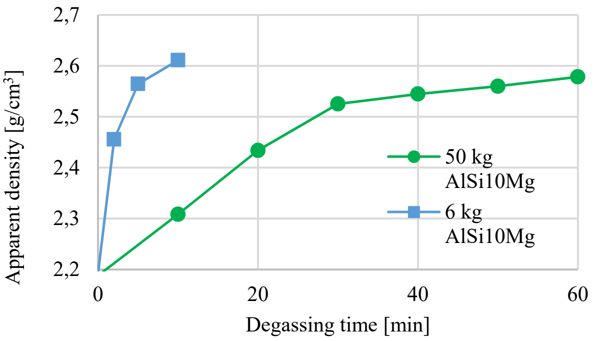

Fig. 8 Ultrasonic degassing for 6 kg and 50 kg of aluminium alloy employing 4 Nl/min purging gas

The results show the degassing rate decreasing for higher volume aluminium treatment. Comparing treatment time and obtained degassing level, it can be considered that the treatment time necessary to get certain degassing level is linearly proportional to the amount of aluminium in the furnace. For 6 kg alloy, 9 minute treatment was necessary in order to get 2.6 g/cm^3 apparent density (1.5 min/kg alloy). For 50kg, following the degassing trend, it would be necessary 73 minutes of degassing time to reach the same apparent density value (1.46 min/kg alloy).

3.4. Ultrasonic and rotary degassing comparison

In this stage, comparison of the ultrasonic degassing and conventional rotary degassing was accomplished in 50 kg of aluminium alloy. Degassing treatments were performed in similar steps for the two studied systems, with certain degassing time and purging gas flow (see in Table 2). Degassing time was increased every two steps due to usual efficiency decrease as the degassing of the aluminium is being effective. Gas flow rate also was increased from 5 to 8 Nl/min in the las 3 steps because similar reason. Notice that the minimum gas flow rate to keep liquid aluminium out of the orifices in the rotary impeller is 5 Nl/min. Thus, there was not any possibility to use same gas flow rates as in the small aluminium volume tests. Each degassing step shown in Table 2 was performed with the melt condition of the previous step, in other words, degassing was cumulative. After each step, RPT test was made to measure apparent density.

Table 2.
Degassing treatment parameters for the ultrasonic and rotary degassing comparison

Degassing time [min]	2	2	4	4	4	4	8
Gas flow [Nl/min]	5	5	5	5	8	8	8

To a better understanding of the performance of the two systems, degassing volumetric efficiency was defined. This efficiency is given in % per litre units and pretends to evaluate both the degassing itself and the purging gas consumption efficiency:

$$Efficiency\ \left[\%/_{l}\right] = \frac{\frac{D_f - D_i}{D - D_i} \times 100}{V_{N_2}}$$

Di, is the apparent density of the melt before the degassing step; Df, is the apparent density after the treatment; D, is the theoretical density of the alloy (2.67 g/cm^3 for AlSi10Mg); and VN2, is the volume of the purging gas employed in the treatment step (flow rate x time).

The results of the density measured in each step and the calculation of the volumetric degassing efficiency are shown in Figure 9. According to the measures, both systems have similar performance, getting slightly higher densities with the rotary degasser. As expected, the volumetric efficiency decreases as the density gets closer to the ideal density (2,67 g/cm^3). Once this value was about to be reached, the efficiency became negative or close to zero. The efficiency also decreases when the flow rate of the nitrogen is increased. In most of the steps, the efficiency of the rotary degasser is higher than the ultrasonic one.

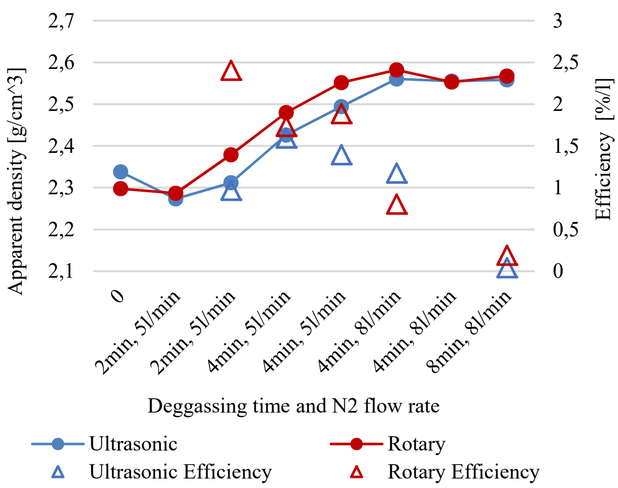

Fig. 9. Effect of the degassing time and purging gas flow rate for both ultrasonic and rotary degassers

4. Conclusions

In this work, RPT sample analysis was not performed visually examining cross cutting sections but measuring apparent density using Archimedes´ Principle method. Bi-film index was not calculated in this work, as it is not usual quality assessment method in foundries. However, in future works, especially in those aimed to establish the relation between oxide content of the melt and hydrogen porosity, this index should be calculated and correlated to other quality indicators.

The experiments carried to study the RPT method demonstrated that temperature control of the aluminium sample when vacuum is applied during the solidification, is necessary to obtain consistent results. The considerable influence of the atmosphere humidity in the RPT measurements, recommends considering or at least keeping humidity value records and avoid density measure comparisons with measures performed in different humidity conditions (more than 10% RH difference).

In the ultrasonic-gas blowing combined degasification method, purging gas flow rate has minor effect in the hydrogen removal. Increasing gas flow rate, degassing could be accelerated, but the gas usage efficiency decreases. Efficiency loss shown in the last four steps (Figure 9) was caused both by the increasing of the gas flow rate and the lower dissolved hydrogen in the melt in the last steps. The oxide content in the melt does not seem to have considerable influence in the degasification process by this technique.

Necessary degassing time is proportional to the aluminium mass to be treated. The results obtained in this work suggest that about 1-2 min/kg degassing rate could be considered to achieve a density of 95% (from 2,2 g/cm^3 to 2,6 g/cm^3), employing the ultrasonic-gas blowing combined degasification method, with a 1500W ultrasonic degasser and 4Nl/min of nitrogen purging gas.

According to this study, rotary degasser has higher volumetric efficiency than the 1500W ultrasonic degasser. Degassing rates obtained in this study are similar for both technologies, therefore, the required time to reach optimum hydrogen level has not been improved with the ultrasonic degasser.

Degassing time is a major aspect for the discontinuous casting processes, especially in those where degassing is performed in the transfer ladle without any heat input to the aluminium. To consider ultrasonic degasser as a real alternative to conventional methods in discontinuous casting processes, more powerful ultrasonic degassers, or simultaneously working ultrasonic degasser arrays should be tested and compared to the rotary degasser.

Acknowledgements

This work has been done within the AL-STRUC project supported by the ELKARTEK program of the Basque Government under Grant KK-2017/00020.

References

[1] Robles-Hernandez, F.C., Ramirez, J.M.H. & Mackay, R. (2017). *Al-Si Alloys: Applications in the Automotive and Aerospace Industries*. Springer.

[2] ASM International. (2018). *ASM Handbook Volume 15: Casting*. ASM International.

[3] Kaufman, J.G. & Rooy, E.L. (2014). *Aluminum alloy castings: properties, processes, and applications*. ASM International.

[4] Lumley, R. (2010). *Fundamentals of aluminium metallurgy: production, processing and applications*. Elsevier Science.

[5] Smith, D.D. & Britt, K.O. (2016). Theory and practical application of ultrasonic degassing at JW aluminum. *Light Metals 2016*. Springer. 803-808.

[6] Alba-Baena, N., Pabel, T., Villa-Sierra, N. & Eskin, D.G. (2013). Effect of ultrasonic melt treatment on degassing and structure of aluminium alloys. *Materials Science Forum*. 765, 271-275. DOI: 10.4028/www.scientific.net/MSF.765.271.

[7] Eskin, G.I. (1995). Cavitation mechanism of ultrasonic melt degassing. *Ultrasonics Sonochemistry*. 2(2), S137-S141. DOI: 10.1016/1350-4177(95)00020-7.

[8] Puga, H., Barbosa, J., Azevedo, T., Ribeiro, S. & Alves, J. (2016). Low pressure sand casting of ultrasonically degassed AlSi7Mg0. 3 alloy: Modelling and experimental validation of mould filling. *Materials & Design*. 94, 384-391. DOI: 10.1016/j.matdes.2016.01.059.

[9] Rundquist, V., Manchiraju, K. & Qingyou, H. (2015). Ultrasonic Degasing and Processing of Aluminum Part II. *Light Metals 2015*. Springer. 943-948.

[10] Puga, H., Barbosa, J., Seabra, E., Ribeiro, S. & Prokic, M. (2009). The influence of processing parameters on the ultrasonic degassing of molten AlSi9Cu3 aluminium alloy. *Materials Letters*. 63(9-10), 806-808. DOI: 10.1016/j.matlet.2009.01.009.

[11] Haghayeghi, R., Heydari, A. & Kapranos, P. (2015). The effect of ultrasonic vibrations prior to high pressure die-casting of AA7075. *Materials Letters*. 153, 175-178. DOI: 10.1016/j.matlet.2015.04.034.

[12] Pabel, T., Petkov, T., da Silva, M., Ruzsinszki, R., Planta, X., Tort, J. & Eskin, D. (2015). The effect of ultrasonic degassing on the quality and properties of components produced by low and high pressure die casting. *International Foundry Research*. 03, 28-33.

[13] Meek, T.T., Han, Q. & Xu, H. (2006). *Degassing of aluminum alloys using ultrasonic vibration*. United States: Department of Energy. Office of Energy Efficiency and Renewable Energy.

[14] Eskin, D.G. (2017). Overview of ultrasonic degassing development. *Light Metals 2017*. Springer. 1437-1443.

[15] DJurdjevic, M.B., Odanovic, Z. & Pavlovic-Krstic, J. (2010). Melt quality control at aluminum casting plants. *Metallurgical and Materials Engineering*. 16(1), 63-76.

[16] Uludağ, M., Çetin, R., Dişpinar, D. et al. (2018). On the Interpretation of Melt Quality Assessment of A356 Aluminum Alloy by the Reduced Pressure Test: The Bifilm Index and Its Physical Meaning. *International Journal of Metalcasting*. 12, 853-860. DOI: 10.1007/s40962-018-0217-4.

[17] Riestra, M., Bjurenstedt, A., Bogdanoff, T. et al. (2017). Complexities in the Assessment of Melt Quality. *International Journal of Metalcasting*. 12, 441-448. DOI: 10.1007/s40962-017-0179-y.

[18] Dispinar, D. & Campbell, J. (2004). Critical assessment of reduced pressure test. Part 1: Porosity phenomena.

International Journal of Cast Metals Research. 17(5), 280-286. DOI: 10.1179/136404604225020696.
[19] Samuel, A.M. & Samuel, F.H. (1993). The reduced pressure test as a measuring. *Metallurgical Transactions A.* 24(8), 1857-1868. DOI: 10.1007/BF02657860.
[20] Totten, G.E. & MacKenzie, D.S. (2003). *Handbook of Aluminum: Vol. 1: Physical Metallurgy and Processes.* CRC press.