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Development of a Measurement and Control Method Enabling the Determination of the Gas Phase Content in a Liquid Metal or Alloy

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Abstract

This article presents various possibilities for testing the degree of gassed of liquid metals. This phenomenon poses a significant problem in the foundry industry, therefore, it is crucial to conduct extraction processes and develop a rapid and accurate method for assessing the gas phase content in the alloy. In addition to analysing methods already known and used in industry and laboratories, the authors present possible research directions using innovative ideas. To confirm hypotheses, they build a test rig and then conduct a series of tests that yield interesting results. The authors studied a lead alloy (Pb-Sn). The alloy was analyzed under the same conditions, subjecting it to moderate and then intense gassed. The results were compared with those of a reference test of a ungassed alloy. It was observed that the ungassed alloy showed a linear increase in the resistance of the liquid metal over the tested temperature range. With the gassed charge, there were either slight changes or the resistance value remained constant over the tested temperature range. The research method not only provides a quick answer but also allows for determining whether the degassing process can be interrupted or should be continued. The method is based on examining the micro-resistance of the system at a given temperature using measuring probes placed in the liquid alloy. By examining a reference sample and determining its reference system parameters, the user can quickly assess the charge in terms of the degree of gassed for each subsequent batch of material prepared in the industrial process.

Keywords: Gas, Metal, Measurement method, Microresistance

1. Introduction

The occurrence of gases in liquid metals is a known problem in the foundry industry [1]. The degree of gassed of liquid metal is directly related to Henry's law, which indicates that the solubility of gases in liquids is a function of pressure under isothermal conditions [2]. In this respect, there are known methods [3, 15 - 19] and patents [4] for measuring gas in liquid metal, such as the methods developed by Dardel or Straube and Pfeiffer [5]. However, all these achievements come down to the use of a gasometer and a hermetic chamber. Although these methods can be relatively fast, they require taking a test sample of the molten metal, and then performing a precise test in order to obtain a reliable result and final processing of the results. In each case, the process of preparing the raw material for casting purposes is prolonged.

For this reason, the authors decided to develop a research method that would provide a very short response regarding the degree of gassed of the metal charge, in the form of a message and an answer as to whether the charge should be degassed or not. This method should take a few seconds and be performed without the use of equipment or a vacuum chamber, allowing the material to be used to assess its suitability in the foundry process.

2. Research methodology

The Dardel method (metod of the first bubble) is based on the observation of the surface of the liquid metal, while the pressure of the melting atmosphere is constantly reduced. When the first gas bubbles appear, the measurement conditions are noted, and the system reaches a state close to saturation. Using the Sieverts relationship, it is possible to assess the gas content in the liquid metal at a given temperature. By reading the negative pressure at the moment of bubble release and using graphs defining the conditions of the equilibrium temperature and solubility of gases in the metal, it is possible to determine the volume of gas dissolved in a given amount of sample. The method requires the use of a vacuum furnace of an appropriate design equipped with a vacuum pump, a monometer and a temperature indicator. Although the method allows for fairly accurate measurements, for foundry purposes, where it is necessary to quickly obtain information on the degree of gassed of the charge, it will not be sufficiently convenient and effective. In addition, this method can only be used for a charge on the surface of which a compact oxide layer has formed. Without it, gases quickly evaporate from the metal, and the actual saturation of the bath is significantly reduced (**method 1**). Therefore, this method does not meet the authors' expectations regarding the rapid assessment of the degree of gassed of the liquid metal.

Another method is the Straube–Pfeiffer method. In this method, a gasometer is used, in which a sample of the tested material is placed, and then a vacuum is created in a vacuum chamber in a very short time. During the experiment, changes in the meniscus of the liquid are observed. A large, convex meniscus of the sample indicates a high degree of metal gassed. On the other hand, a small convexity of the meniscus indicates a low degree of saturation. The above method differs from the Dardel method in that the measurement conditions in this case always take a constant value and are not set individually, as in the first case. It should be noted, however, that the optimal vacuum should be determined experimentally, because its erroneous values will affect the measurement errors. Based on literature data [6 - 9], specific vacuum values are assumed, e.g. for bronzes and cast irons 13.3 – 26.6 hPa, for light alloys 79.0 – 133.0 hPa, and for low-melting alloys 133.0 – 160.0 hPa. Although the above method allows for relatively quick obtaining of information on the degree of gassed of the liquid metal, it may be burdened with a significant error. This results not only from the need to observe the behavior of the meniscus of the liquid metal surface, but also from the need to determine specific vacuum values for given alloys. Therefore, the above method will not provide satisfactory evaluation results (**method 2**). The above two methods (method 1 and method 2) are well known and described in the literature, hence in this study we will focus on other research directions that can be used and tested.

In order to develop an effective measurement and control method, it is necessary to analyze the possible directions of research that should be undertaken in connection with the design of an appropriate laboratory stand. The first concept is the stand construction shown in the diagram below (Figure 1).

The principle of the station is based on the measurement of the charge density in a steel crucible (Fig. 1). The more gas phase the charge has, the lower its density will be. For this purpose, the isotope cesium 137 can be used. As a result of the decay of the isotope, beta and gamma radiation are emitted. The isotope must be placed in a steel ampoule made of acid-resistant steel, which blocks the emission of beta radiation. The ampoule must also be placed in a lead body surrounded by a steel jacket to block harmful gamma radiation. The body contains a so-called collimation gap, through which gamma radiation penetrates on the outside in a specific direction. The radiation passes through a crucible filled with a liquid charge and is attenuated by the entire system. It reaches the scintillator, where the energy of gamma radiation carried by photons is converted into micropulses of light. Then they pass to a photomultiplier, where they take on an electrical form in a cathode follower, and are then counted in a scintillation counter. A lower reading on the counter indicates greater attenuation of the medium through which the radiation passes, and at the same time, the presence of a smaller amount of the gas phase in composition.

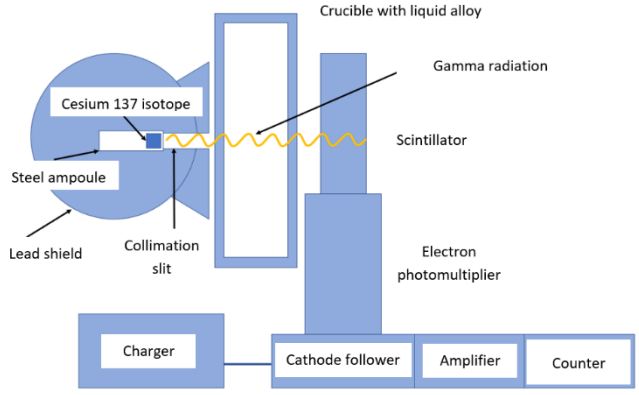

Fig. 1. Construction of a research station using the radioactive isotope of cesium

The above method, although promising, is associated with a number of difficulties related to the use of a radioactive element. Working with such material requires appropriate training of employees, proper preparation of the workplace, the use of a number of protective materials not only in the construction of the station itself, but also in the protection of the health and life of people nearby. In addition, the use of radioactive elements requires obtaining the consent of certain bodies. Therefore, the use of this method will have to be abandoned (**method 3**).

Another method to solve the above problem is to prepare the research stand as shown in the figure below (figure 2).

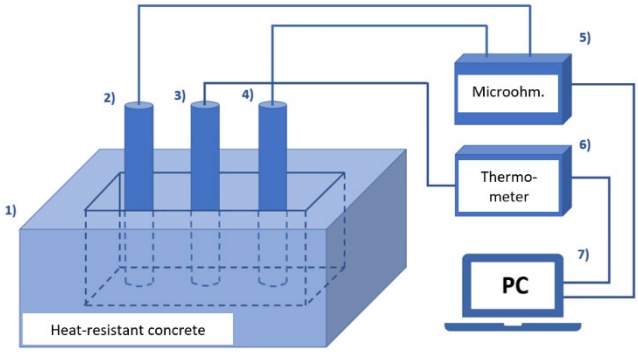

Fig. 2. Construction of a stand for testing the degree of metal gassed by recording the charge resistance

Based on the conducted studies [10, 11], it concluded that it is possible to determine the resistance of the medium, which varies depending on its chemical composition, metallic and non-metallic inclusions and the temperature of the system, and its determination can be performed with an accuracy of the order of μΩ.

The presented system consists of a crucible made of refractory concrete, which is placed in a chamber furnace together with the charge. The crucible must be made of a material that does not conduct electricity. After the charge is melted, measuring probes no. 2 and 4 are immersed in it, which are connected to an ohmmeter enabling the measurement of small resistances. Additionally, measuring probe no. 3 is placed in the crucible, which measures the temperature of the medium in real time. Both meters are connected to a computer (7), and data recording is started simultaneously by switching them on simultaneously. Based on the collected readings, it is possible to determine the change in resistance resulting not only from the change in the temperature of the system, but also to determine the occurrence of non-metallic inclusions in the metallic charge (**method 4**). This method is an interesting research alternative that meets the authors' expectations, and therefore can be analyzed in terms of its usefulness in foundry processes.

A completely different method of determining the degree of gassing in a system (theoretical method) can be the measurement of the charge using the Doppler effect. The Doppler effect uses an ultrasound beam, which can be used to measure the flow rate of contaminated or gassed liquids. Gas bubbles in the liquid cause the reflection of radiation emitted by the transducer, changing its frequency at the same time. A necessary condition for this method is the flow of the liquid through the measuring element, on which the ultrasonic flowmeter would be mounted. Meanwhile, the flow rate of clean and foamy liquid through the pipeline will change depending on the degree of gassing in the system. The proposed solution, although justified from a theoretical point of view, is not insignificant when it comes to the costs of implementing such a project. A flowmeter that meets its purpose (e.g. the DeltawaveC - P model) is quite expensive [12], and moreover, it does not guarantee a solution to the problem (**method 5**). The purely hypothetical nature of the method, as well as the high cost of building a test stand, preclude its practical application. This is particularly important because the method is used to measure the degree of gas dispersion in liquid pipelines, but has not yet been adapted to systems containing liquid metal.

It is also worth paying attention to the possible possibility of assessing the degree of alloy gassed using computed tomography. Since it is possible to detect porosity in ductile iron castings, one can try to assess the system in the liquid state in terms of the content of gas bubbles present in it [13]. However, when analyzing X-ray images of the obtained castings, there is a fear that we will not notice changes in the image of the gasified and non-gasified charge on the screen obtained from the computed tomography scanner. On the other hand, densitometric tests are used in medicine to determine bone density, which allows for the detection of osteoporosis at an early stage. Hence, by relating this fact to the degree of more or less gasified casting, one could expect to obtain an image that is more or less shaded, respectively. Thanks to this, it would be possible to determine the degree of charge gasssed even with a very high degree of gas dispersion. Moreover, the detection of large gas bubbles would be immediate, and determining the degree of material gassed should not be a major problem. However, this method does not allow for obtaining the result in a short time. Despite its high accuracy, the analysis takes a relatively long time, eliminating it from the group of potential methods allowing for the effective measurement of the gas phase in alloys in the liquid state (**method 6**). It's also worth noting that this method requires the object being tested to be at rest and not moving. However, the liquid metal can move within the crucible due to temperature changes and convective motion, which may further preclude this method from further consideration.

It is worth mentioning another possibility of using the measurement method using a rotating 3D scanner. Assuming the presence of gas bubbles in the melted charge, it can be expected that the volume of the entire system will be larger than the system without the gas phase. The device could scan the volume of the crucible filled with the charge, informing the user about the volume that a given object would occupy. However, the imperfection of this method is the possibility of performing too imprecise measurements in a short time, as well as the high degree of dispersion of the gas phase, which could have a negligible or no effect on the change in the charge volume. Therefore, it was decided to omit this method in further considerations (**method 7**).

3. Research results

In order to test the gassed state of the liquid metal or alloy, it was decided to use the above-described "method 4" of testing the charge resistance, which is registered with the patent office [14]. Additionally, before the actual test, it was decided to test the temperature distribution of the solidifying alloy in the gasified and non-gasified state. The first series of tests used metal alloy LC90 containing 90% tin and 10% lead. It was decided to test the temperature of the system using the measuring station [15]. Under normal conditions, the temperature distribution of the solidifying material was as follows (figure 3).

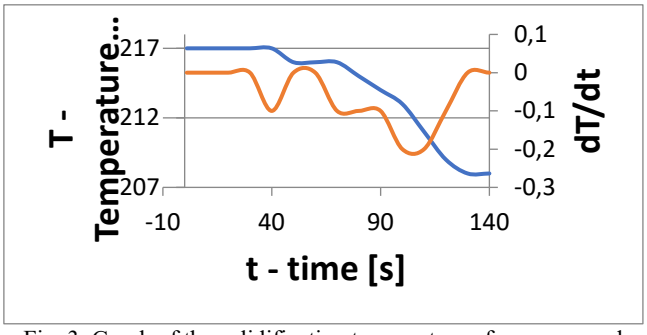

Fig. 3. Graph of the solidification temperature of a non-gassed alloy and the dT/dt

From the obtained graph, it can be read that the solidus - liquidus temperature was in the range of 211 - 217 degrees C. Then the same metal was melted again and then gasified. It turns out that the solidification temperature of the gasified alloy was in the range of 218 - 223 degrees Celsius, which would mean that its temperature increased by exactly 7 degrees.

Here it is necessary to indicate the method of metal gassing and its composition. The gas used for gassing the alloy was a mixture of gases with a composition similar to air (78% nitrogen, 17% oxygen, 4% carbon dioxide, 1% other gases). It was fed through a copper tube into the crucible with the alloy for at least 30 seconds. Feeding the gas into the crucible caused it to spatter slightly. As a result, it was not possible to obtain a material with a foam structure, but the macroscopic appearance of the solidified alloy differed slightly from the previous batch - a very fine granular needle structure could be observed, which could indicate the formation of metal oxides. According to the decrease in the thermodynamic potential G, one can expect the formation of a "suspension" of mainly tin oxides SnO in the alloy. As a result, this could lead to an increase in the solidification temperature of the alloy, especially since the reaction of oxide formation in the system is a strongly exothermic reaction. The occurrence of small differences in the solidification temperature of the gassed and not - gassed alloy does not seem to be a sufficiently effective method for determining the gas phase degree of the charge.

In the second phase of the study, it was decided to use the resistance test during the solidification system. For this purpose, the measurement system from the first experiment was rebuilt in such a way that it met the requirements of the diagram in Figure 4. The measurement system was equipped with two additional copper electrodes, galvanically isolated from the structure of the stand, as well as an electrode measuring the temperature of the alloy. In the first experiment, the resistance of the non-gassed alloy was constant over the entire measurement range and amounted to 0.5 Ω. In the second stage of the experiment, the alloy was subjected to gasification. At this point, small fluctuations in the changes in the charge resistance between 0.4 and 0.5 Ω could be observed. The test was performed with a digital meter UT-70B UNI-T, which allows for measuring resistance with an accuracy of up to 0.1 Ω. However, despite the high accuracy of the meter, it was not possible to record changes in resistance during the solidification of the alloy. The measurement results indicated the internal resistance of the system. For this purpose, it was necessary to use a device with higher accuracy. For this purpose, a low resistance meter was used as shown in the figure below, which has a measurement accuracy of up to 1 μΩ (figure 4).

Fig. 4. Photo of the microohm meter used for testing

The next test cycle consisted of three stages. The first involved melting the still charge and checking its resistance. The second stage involved remelting the same charge, intensively gassing it, and again checking its resistance within the same temperature ranges. The final stage involved remelting the already gassed charge, partially degassing it through thermal refining, and checking the resistance of the solidification system within the tested temperature range.

For the next test cycle, the LC30 alloy (30% tin Sn, 70% lead Pb) was prepared. First, the resistance of the non-gassed alloy was measured in the range from 170 to 200 ºC. The test results are summarized in the table below (Table 1) and presented in the graph (Figure 5). The graph also includes standard error bars. Data was processed in an Excel spreadsheet.

Table 1.

Measurement no.	Temperature [^{0}C]	Resistance [mΩ]
1	190	2.2
2	200	2.3
3	210	2.3
4	220	2.4
5	230	2.5
6	240	2.7
7	250	2.8
8	260	3.0
9	270	3.0
10	280	3.4

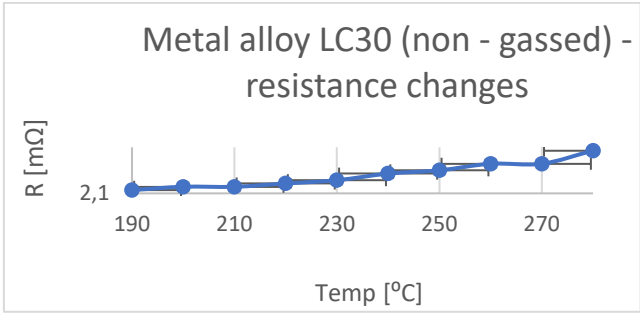

Fig.5. Resistance chart of non-gassed metal alloys with standard error bars

In the above case, a linear increase in the resistance of the liquid alloy can be observed in the range from 2.2 mΩ to 3.4 mΩ in the temperature range from 190 to 280 ºC. This is, of course, in accordance with the laws of physics, because we know that as the temperature decreases, the resistance of the metal decreases.
In the second case, the alloy was subjected to strong gasification. The system resistance was measured over a similar temperature range. The results are summarized in the table below (Table 2) and presented in the graph (Figure 6). The graph also includes standard error bars. Data was processed in an Excel spreadsheet.

Table 2.

Measurement number	Temperature [^{0}C]	Resistance [mΩ]
1	190	2.2
2	200	2.3
3	210	2.3
4	220	2.3
5	230	2.3
6	240	2.3
7	250	2.3
8	260	2.3
9	270	2.3
10	280	2.3
11	290	2.5
12	300	2.6
13	310	2.8

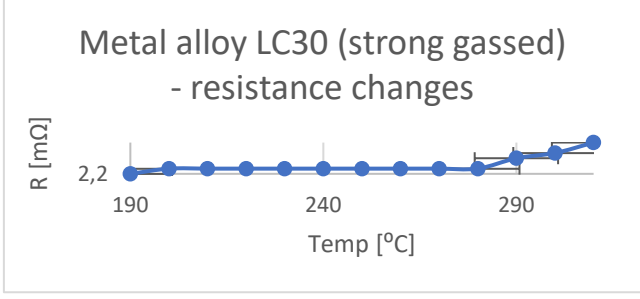

Fig. 6. Resistance graph of strong gassed metal alloys with standard error bars

In the above case, a strong drop in the system resistance can be observed. It is particularly noteworthy that the linear resistance value was maintained over a wide range of tested temperatures and amounted to 2.3 mΩ. Only at a higher temperature did the resistance increase to 2.8 mΩ.
Finally, the same alloy was melted. Since the system did not develop a dense oxide layer on its surface, gases could diffuse from the metal into the melting atmosphere, thus reducing the bath saturation. After melting, the system was again subjected to a quick bench test. The results are summarized in the table below (Table 3) and presented in the graph (Figure 7). The graph includes standard error bars too. Data was processed in an Excel spreadsheet.

Table 3.

Measurement number	Temperature [^{0}C]	Resistance [mΩ]
1	170	2.78
2	180	2.81
3	190	2.90
4	200	3.01

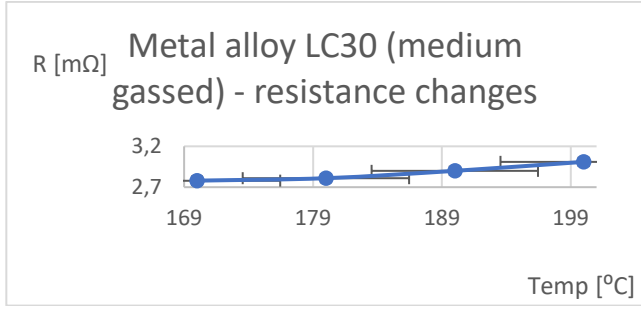

Fig.7. Resistance graph of medium gassed metal alloys with standard error bars

In this case, the resistance changes are less linear than in the previous situation, but it can be seen that the difference in the extremes of the function is not as large as in the case of the non-gassed alloy and ranges from 2.78 to 3.01 mΩ.

To complement the presented analysis, it was decided to investigate the degree of metal gasification using the proposed method for a different type of metal. In this case, a metal sample was selected that exhibited a lower chemical affinity for oxygen at the melting point, and therefore had a lower ability to form oxides but a higher ability to form gas bubbles, and also had a higher melting point. In the previous case, the alloy studied was LC30 containing approximately 30% Sn. In the next approach, a sample of pure lead (Pb) was examined. As in the previous case, the non-gased alloy was first examined, the results of which are summarized in the following table (Table 4) and presented in the graph (Figure 8). The graph includes standard error bars too. Data was processed in an Excel spreadsheet.

Table 4.

Measurement number	Temperature [^{0}C]	Resistance [mΩ]
1	330	1.99
2	340	2
3	350	2.02
4	360	2.04
5	370	2.06
6	380	2.07
7	390	2.09
8	400	2.11
9	410	2.11
10	420	2.13
11	430	2.14

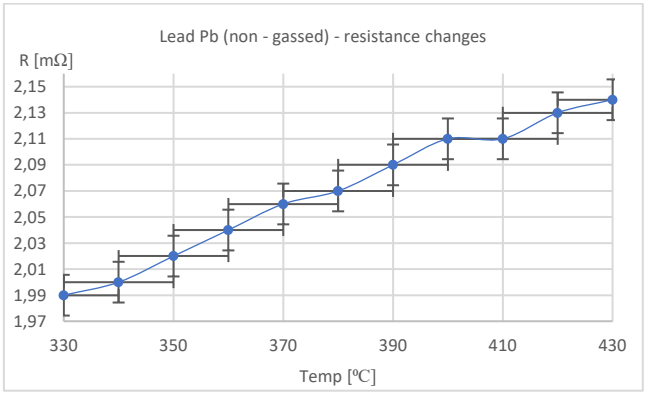

Fig.8. Resistance chart of non-gassed metal alloys with standard error bars

In the second test, the metal was melted and gassed under the same conditions as in the first test. The results are summarized in the table below (Table 5) and presented in the graph (Figure 9). In this case, a significant difference was also observed between the tested samples. As expected, the metal containing more gas bubbles had a significantly higher resistance than the pure metal without them.

Table 5.

Measurement number	Temperature [^{0}C]	Resistance [mΩ]
1	330	3.02
2	340	3
3	350	3
4	360	2.99
5	370	2.98
6	380	2.98
7	390	3.01
8	400	3.07
9	410	3.13
10	420	3.42
11	430	4.14

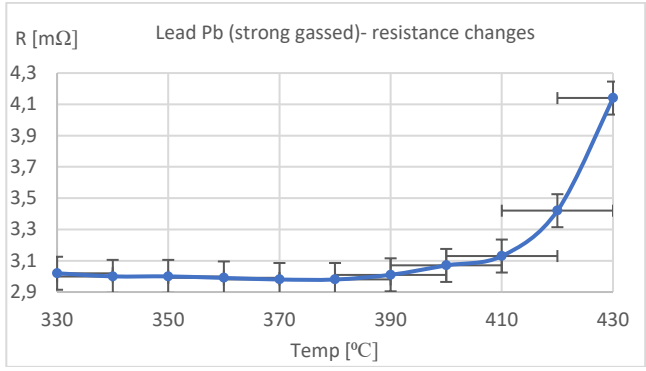

Fig.9. Resistance graph of strong gassed metal alloys with standard error bars

4. Conclusions

Based on the conducted research and analyses, it can be stated that the proposed method of testing changes in the resistance of the medium for assessing the share of the gas phase in a liquid metal or alloy is a very good and fast analytical method. The research methodology related to the temperature analysis of the system has a significant disadvantage, despite the differences in the liquidus temperature of the gasified and non-gasified alloy, is the additional time needed for analytical processing of the obtained data, which does not allow for reading the exact result in a direct and fast reading. Despite the visible difference in temperature values, the user is not able to use the observed relationship in practice. This is related not only to the time needed to process the data, but also to the data observed only at the stage of alloy solidification and its liquidus temperature. The lack of possibility of analysis above this temperature does not provide specific information to the interested person.

In the case of the metal alloys resistance test, however, it is possible to immediately read the resistance value of the system over time by comparing it with the known resistance of the non-gassed system and, on this basis, determine the degree of gassed of the charge at least at a level sufficient to fill the casting mold. After taking a sample, the measurement is carried out in a very short time (a few seconds), the data does not need to be processed in any way, and the user receives a very specific quantitative indicator. Knowing the type of alloy and the optimal resistance values of the charge at a given temperature, it is possible to obtain information about the need to carry out refining procedures of the system, including the need to degas the metal bath. At the end of the test, it was decided to make test castings from an alloy with a medium degree of gasification. The casts made were satisfactory and had no macroscopic defects in their structure. However, their internal structure was significantly different, and the casting itself was much more brittle than castings made from the non-gassed alloy. The resistance measurement results confirm the observed effect, which may contribute to further research into determining the optimal values for selected alloys.

Common alloys used in foundry applications include silumins, brasses, bronzes, and even steels. The above method can be applied to the facility processing the selected alloy and making the castings. The above measurement method must be adapted to each alloy

selected. First, a reference measurement is taken of an alloy well-prepared for the casting process. Then, during the process, batch test measurements can be performed to assess the gassed level in the system and the correctness of degassing.

A reference measurement is a key step in determining the degree of charge gassed. The microresistance of any alloy can vary significantly, not only due to the charge's chemical composition, the presence of non-metallic inclusions and impurities, but also due to environmental conditions and the type of electrodes used. Therefore, a test measurement of a well-prepared and degassed alloy, ready for the casting process, serves as a starting point and reference for subsequent tests. Subsequent tests should be conducted under similar conditions, ensuring the greatest possible similarity in the chemical composition of the charge and electrodes (length, diameter, and chemical composition – copper electrodes were used in the tests). Only tests prepared in this way will yield the desired results and answer the question of whether further refining and degassing of the alloy is necessary. It is also important to remember that the measurement should be performed at the same current intensity, which in the presented tests was 1 A.

It's also worth noting that this is a proof-of-concept method, which confirms the feasibility of the presented idea by creating a simplified experimental model for conducting research. According to the presentation, the research was tested on lead alloys, but it is assumed that it can be applied to other casting alloys as well.

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