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Selected Aspects of Nitrogen Refinement of Silumin 226

T. Szymczak*, G. Gumienny, T. Pacyniak
Department of Materials Engineering and Production Systems, Lodz University of Technology,
Stefanowskiego 1/15 Street, 90-924 Łódź, Poland
*Corresponding author. E-mail address: tomasz.szymczak@p.lodz.pl

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Abstract

The work presents the results of the investigations of the effect of the nitrogen (N_2) refining time „τ_{raf}" and the gas output on the course of the crystallization process, the microstructure and the gassing degree of silumin 226 used for pressure casting. The refinement of the examined silumin was performed with the use of a device with a rotating head. The crystallization process was examined by way of thermal analysis and derivative analysis TDA. The performed examinations showed that the prolongation of the N_2 refining time causes a significant rise of the temperature of the crystallization end of the silumin, „tL", as well as a decrease of its gassing degree, „Z". An increase of the nitrogen output initially causes an increase of the temperature „tL" and a drop of the gassing degree „Z", which reach their maximal values with the output of 20 dm^3/min. Further increase of the output causes a decrease of the value „tL" and an increase of „Z". The examined technological factors of the refining process did not cause any significant changes in the microstructure of silumin 226.

Keywords: Theoretical basics of casting processes, Pressure casting, Refining, TDA method

1. Introduction

Refining is a procedure performed on a liquid alloy whose aim is to clean the latter of gases and solid impurities in the form of oxides, borides, nitrides or spinels, as well as harmful elements such as Li and Be. The silumin refining process involves the use of refiners in a solid or gas form. The solid silumin refiners include sodium fluoride and calcium, potassium and magnesium chlorides. In the gas refining process, we use inert gases such as: nitrogen, argon, chlorine or mixtures of the latter. Blowing of silumins with inert gases causes mainly the precipitation of hydrogen [1].

The high concentration of hydrogen in silumins is the reason for the presence of porosity in the casts, to which pressure casts are the most exposed. The high pressure working on the liquid silumin in the mould causes the precipitation of a large amount of hydrogen in the form of blisters, which remain in the microstructure of the alloy also after the end of the crystallization process. The application of a refining process for silumins assigned for pressure casting can significantly lower their gassing level and, in consequence, the amount of porosity in the cast [2].

In respect to the above, this work examined the effect of selected technological factors on the nitrogen (N_2) refining process with the use of a device with a rotating head on the crystallization process of silumin 226, as well as its microstructure and gassing degree. The examined technological parameters of the refining process were the following: the nitrogen blowing time and the volume output. Silumin 226, used for the tests, is widely used in pressure casting.

The examinations were performed under the production conditions of the Innovation and Implementation Enterprise Wifama-Prexer Ltd., Poland.

2. Test methodology

The range of the chemical compositions used in the examinations of silumin 226 is presented in Table 1. The silumin was melted in a gas-heated shaft furnace. After being poured into the ladle, the silumin was deslagged and carried into the head-rotating device, where it was refined with nitrogen. The following refining times were used: 1, 2, 3 and 4 min as well as the following values of the nitrogen volume output: 10, 15, 20, 25 and 30 dm^3/min. As the reference point for the evaluation of the effectiveness of the refining process, a test on non-refined silumin was also conducted. The refining process was followed by the examination of the crystallization process of the silumin by the TDA method as well as of the gassing degree. The TDA curves were recorded with the use of the thermocouple PtRh10-Pt placed in the tester TDA10m-PŁ. This method has been widely used in the examinations of the crystallization process of various metal alloys. At the Department of Materials Engineering and Production Systems of the Lodz University of Technology, this method is applied in the examinations of the alloys of: iron, magnesium, copper and cobalt [3-7].

Table 1.
Range of chemical compositions of silumin 226

Chemical composition, %							
Si	Cu	Zn	Fe	Mg	Mn	Ni	Al
9.27 ÷ 9.54	1.96 ÷ 2.32	0.84 ÷ 0.97	0.70 ÷ 0.87	0.30 ÷ 0.33	0.20 ÷ 0.23	0.06 ÷ 0.10	rest

Examinations of the microstructure of the casts from the TDA10m-PŁ tester were also performed with the use of the optical microscope Eclipse MA200 by Nikon. The microsections were etched with 4% HF solution.

3. Test results

Figure 1 shows the representative TDA curves of the silumin which underwent nitrogen refinement.

On the derivation curve, one can observe three thermal effects originating from the transformations taking place in the cooling and solidifying silumin. And so, the thermal effect described by points AB came from the crystallization of the primary dendrites of phase α. The thermal effect described by points BDEFH originated as a result of the crystallization of the eutectic mixture $\alpha + Al_9Fe_3Si_2 + \beta$. After the transformation ended, the remaining volume of the liquid crystallized in the form of the quaternary eutectic $\alpha + Al_2Cu + AlSiCuFeMgMnNi + \beta$. This can be seen on the differential curve in the form of the thermal effect HKL. The silumin crystallization ends at point L. An result of the above crystallization process is the microstructure of the discussed alloy presented in Figure 2 (a, b).

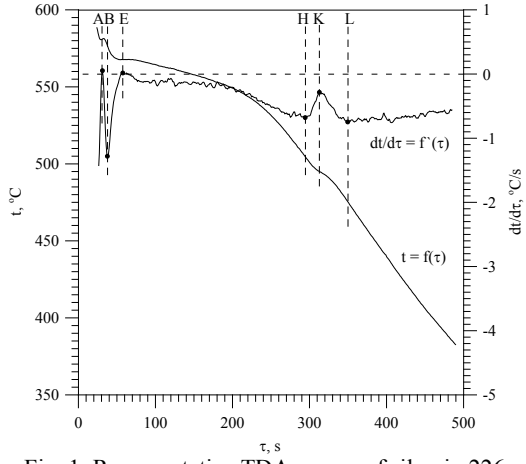
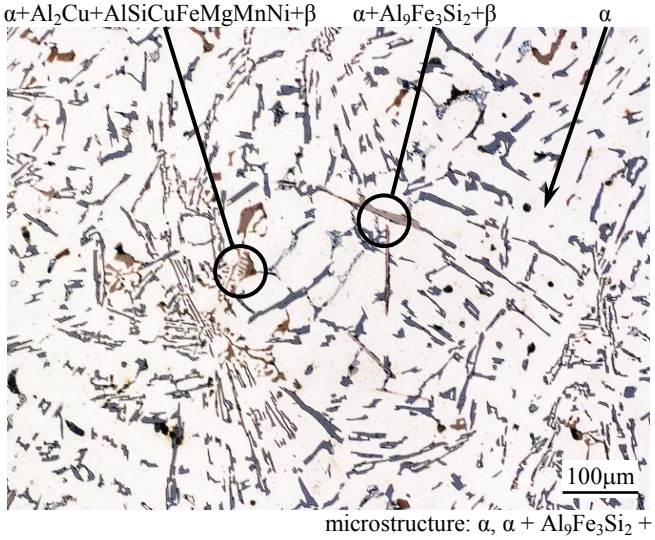
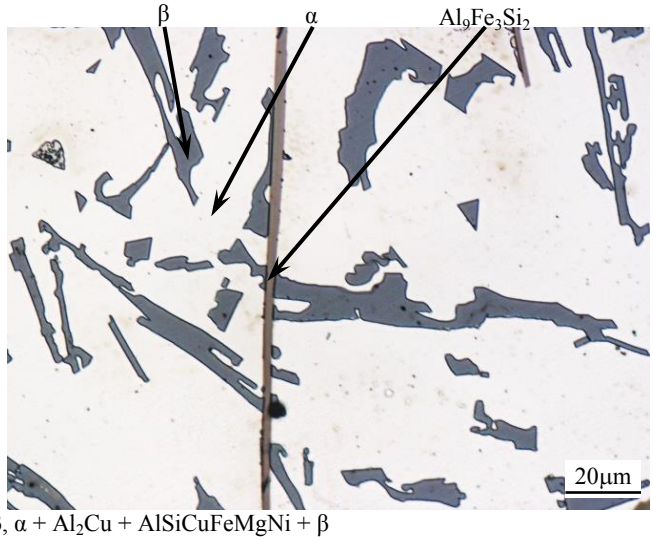


Fig. 1. Representative TDA curves of silumin 226 after nitrogen refinement

a)



b)



microstructure: α, $\alpha + Al_9Fe_3Si_2 + \beta$, $\alpha + Al_2Cu + AlSiCuFeMgNi + \beta$

Fig. 2 (a, b). Representative microstructure of silumin 226 after nitrogen refinement

From Fig. 2 we can infer that the microstructure of silumin 226 consists of the primary precipitations of phase α, the eutectic mixture α + $Al_9Fe_3Si_2$ + β and the eutectic α + Al_2Cu + AlSiCuFeMgMnNi + β. Both for the non-refined silumin and the one refined in the time of 1, 2, 3 and 4 minutes, one could see analogical thermal effects on the TDA curves. And so, their microstructures were also similar. The differences concerned the coordinates of the characteristic points describing the thermal effects in the case of particular transformations. Table 2 presents a compilation of the coordinates of the characteristic points on the TDA curves. The quantities tB, tH and tL denote the beginning and/or end temperature of the phase transformations, and tA and tK – the temperature at the moment of the most intense heat release at the time of the transformation. K = dt/dτ denotes the first derivative of temperature in reference to time and shows the rate of the temperature changes of the cooling and solidifying silumin.

From Table 2 we can infer that, for the refining time of 2, 3 and 4 min, no thermal effect was recorded coming from the crystallization of the primary precipitations of phase α, that is, point A did not exist. This is caused by the drop of the silumin's temperature at the time of refining, due to which the beginning of the TDA curve recording took place at a temperature which made it impossible to record either the liquidus temperature or the whole of the thermal effect coming from the crystallization of phase α.

From the data presented in Table 2 we can also conclude that the nitrogen refinement of the silumin significantly increases the temperature of its the crystallization end tL (of 11°C). This is caused by the elimination of impurities from the liquid alloy. Due to the fact that the impurities are pushed through the crystallization front into the depth of the liquid, they cause a decrease of the temperature of the crystallization end. That is why reducing their concentration results in an increase of the temperature of the crystallization end, tL. The remaining values of the temperature and the cooling rate did not undergo systematized changes.

The examinations of the effect of the nitrogen volume output used in the refining process on the course of the TDA curves and the microstructure of silumin 226 did not exhibit any differences in respect to the results obtained for different refining times. For all the examined nitrogen output values, the microstructure of the silumin is formed by the following phases: α, α + $Al_9Fe_3Si_2$ + β and α + Al_2Cu + AlSiCuFeMgNi + β, and on the TDA curves, we recorded the thermal effects corresponding to their crystallization: AB, BEH and HKL. The change in the nitrogen output caused differences in the coordinates of the characteristic points on the TDA curves of the examined silumin. They are presented in Table 3.

From the data shown in Table 3 we can infer that only the temperature of the crystallization end of the silumin, tL, undergoes systematized changes. The course of the changes of the temperature „tL" in the function of the nitrogen output is shown in Figure 3.

From the above we can conclude that the maximal value of the temperature „tL" occurs for the gas output of 20 dm^3/min. The decrease of the temperature „tL", despite the increase of the nitrogen flow at the time of the refining process to above 20 dm^3/min, was probably caused by the very intense mixing of the metal bath, as a result of which the contact area of the liquid alloy with the environment air significantly increased. This could result in an increase of the amount of the hydrogen dissolved in the silumin, despite the increase of the nitrogen output.

The gassing degree „Z" of the N_2 refined silumins was also examined, as well as gassing of the non-refined silumin was performed. The results of those tests are included in Tables 4 and 5.

Table 2.
Compilation of the values of the quantities describing the TDA curves of silumin 226 – non-refined and after different nitrogen refining times

No.	Refining time, min	t, °C						dt/dτ, °C/s					
		tA	tB	tE	tH	tK	tL	KA	KB	KE	KH	KK	KL
1.	0	582	578	566	506	493	467	0.19	-0.92	0.10	0.66	-0.33	-0.80
2.	1	578	574	566	510	495	469	0.16	-0.92	0.09	-0.71	-0.27	-0.73
3.	2	–	569	561	496	490	471	–	-0.78	0.01	-0.67	-0.29	-0.75
4.	3	–	577	567	500	497	473	–	-0.90	0.06	-0.55	-0.28	-0.76
5.	4	–	573	564	508	494	478	–	-1.07	0.07	-0.62	-0.29	-0.72

Table 3.
Compilation of the values of the quantities describing the TDA curves of silumin 226 refined with the use of different values of the nitrogen volume output

No.	Gas output, dm^3/min	t, °C					dt/dτ, °C/s				
		tB	tE	tH	tK	tL	KB	KE	KH	KK	KL
1.	10	566	559	498	491	478	-1.08	0.20	-0.64	-0.28	-0.70
2.	15	576	567	509	500	484	-1.08	0.10	-0.66	-0.25	-0.75
3.	20	572	565	500	495	487	-0.78	0.09	-0.62	-0.24	-0.76
4.	25	569	566	506	499	479	-0.39	0.05	-0.61	-0.24	-0.74
5.	30	567	563	503	496	462	-0.51	0.08	-0.59	-0.25	-0.83

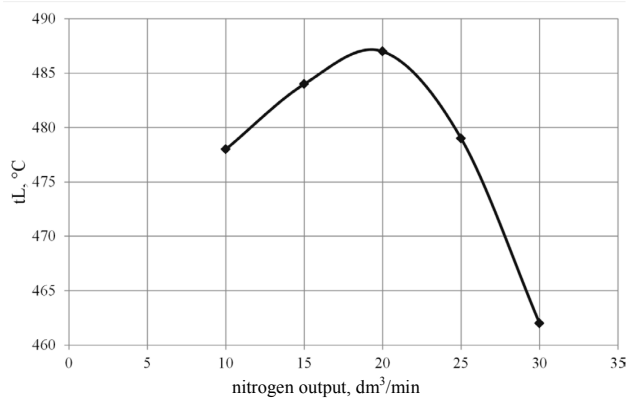


Fig. 3. Course of temperature changes „tL" versus nitrogen output

Table 4.
Gassing degree of silumin 226 – non-refined and after different nitrogen N_2 refining times

No.	τ_{ref}, min	Z, %
1.	0	1.79
2.	1	0.90
3.	2	0.89
4.	3	0.82
5.	4	0.05

Table 5.
Gassing degree of silumin 226 refined for different values of N_2 output

No.	N_2 output, dm^3/min	Z, %
1.	10	0.65
2.	15	0.60
3.	20	0.51
4.	25	0.67
5.	30	1.23

From the data presented in the tables we can infer a relatively high gassing degree of the silumin which did not undergo nitrogen refinement Z = 1.79%. The refining of the silumin for the time of 1, 2 and 3 minutes caused a decrease of the gassing degree down to the level of Z = 0.90÷0.82%. The lowest fraction of the gas in the silumin Z = 0.05% was obtained after refining for 4 min. The gassing degree of N_2-refined silumins with the use of different values of the gas output drops down from Z = 0.65% to Z = 0.51% together with the increase of the output within the range of 10÷20 dm^3/min. When the output is further increased to the value of 30 dm^3/min, the gassing degree increases to Z = 1.23%. The presented results correlate with the changes in the temperature value of the crystallization end of the examined silumin, „tL". The examinations of the effect of the N_2 refining time on the gassing degree „Z" were performed for a constant value of the output equaling 20 dm^3/min.

4. Conclusions

From the data included in this work we can draw the following conclusions:

- a prolongation of the N_2 refining time causes a significant increase of the temperature of the crystallization end of the silumin, „tL", as well as a decrease of the gassing degree, „Z",
- an increase of the nitrogen output initially causes an increase of the temperature „tL" and a decrease of the gassing degree „Z", which reach their maximal values with the output of 20 dm^3/min. Further increase of the output causes a decrease of the value of „tL" and an increase of „Z",
- a change of the examined technological factors of the N_2 refining process does not cause any significant changes in the microstructure of the examined silumin.

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