Comparative study of Al – 5Ti – 1B and Al – 3La – 1.5B modifying ligatures

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The work was performed at the laboratory on semi-continuous casting unit (SCCU) of the Institute of Non-Ferrous Metals and Materials Science of SFU in order to carry out comparative studies of the modifying ability of Al – 5Ti – 1B ligature and a promising modifying ligature Al –3La – 1.5B. Casting and modification of experimental flat ingots was carried out according to the casting modes, which provide the parameters of the cast workpiece structure as close as possible to the parameters obtained in industrial conditions for ingots with a thickness of at least 300 mm. The paper presents the results of quantitative analysis of the microstructure of Al – 5Ti – 1B and Al – 3La – 1.5B ligatures. The paper shows that when 8079 aluminum alloy is modified with Al – 5Ti – 1B ligature rod at the rate of 0.5–1.5 kg/t metal, the average grain size decreases from 190 to 129 μm and there is a zone of columnar crystals, but at the rate of 2–2.5 kg/t metal, the grain size decreases to 100 and 90 μm, respectively, with the complete absence of columnar crystals zone. The paper shows the results of the experiment on modification with Al – 3La – 1.5B ligature at ligature bar consumption from 0.5–1.5 kg/t metal, where the average size in the obtained ingot samples was from 180 to 100 μm, but at modifier consumption from 2–2.5 kg/t metal the average grain size decreases to 99 and 86 μm, respectively. The paper shows that, according to the results of the experiments, the modifying ligature Al – 3La – 1.5B has better modifying ability in comparison with the ligature Al – 5Ti – 1B regardless of oxide film contamination and higher amount of non-metallic inclusions.

Key words: aluminum, aluminum alloys, modification, ligature, aluminum modification, titanium, lanthanum.

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Introduction

Aluminum and alloys based on it have unique characteristics that allow them to successfully compete with traditional construction materials and find application in various spheres [1]. Due to the fact that aluminum alloys are strong, corrosion-resistant, can be cut and welded, blanks made of them are widely used in many industries – shipbuilding, aircraft construction, car construction, etc. [2]. One of the promising areas in the production of aluminum alloys is casting for rolling production, which is not always ready to fulfill the ever-increasing quality and property requirements of customers. Therefore, the development of technical and technological solutions to ensure the required level of quality of aluminum alloy ingots for rolling production is undoubtedly an urgent scientific and technical problem [4].

At present, for grain refining in aluminum alloy ingots produced by semi-continuous casting, bar modifying ligatures based on Al – Ti – B and Al – Ti – C systems are mainly used, however, as production experience shows, it is not always possible to obtain a fine-grained and homogeneous structure throughout the ingot volume when using these ligatures [3–5]. The effect of modification with these ligatures is achieved due to the intermetallic phases contained in them, which allow to create additional crystallization centers — Al_3Ti and TiB_2 [6, 7]. Along with modifying particles Al_3Ti and Tib_2, rare earth elements and their compounds, e.g. Sc, Y, Re, In, Yb, etc., are also becoming popular due to their strong surface-active properties. One of such elements is La as the most economically accessible one [8–12].

The purpose of the work is to carry out comparative studies of the modifying ability of Al – 5Ti – 1B ligature and a promising modifying ligature Al – 3La – 1.5B.

Methodology of the experiment

Al – 5Ti – 1B rod ligature, produced by KBM Affilips (Netherlands) [13] and Al – 3La – 1.5B ligature rod produced by Fengyuen Al.Grain Refiner [14] were selected as the object of study.

An inverted metallographic microscope ZEISS Axio Observer (OM - optical microscopy) with the program complex of metallographic analysis Thixomet.PRO was used to study the microstructure of samples and the quality of modifying ligature.

Experiments on the evaluation of modifying ability were carried out in laboratory conditions of the Siberian Federal University at the semi-continuous casting unit (SCCU). The process flow diagram is presented in **Fig. 1**. Studies were carried out at identical technological parameters using aluminum alloy, the chemical composition of which corresponds to 8079 grade (**Table 1**).

Table 1
Chemical composition of 8079 alloy

Element	Si	Fe	Mn	Mg	B	Pb	Zn	Zr	Ti	Cu	Al
wt. %	0.06	1.04	0.02	0.004	0.0003	0.0009	0.008	0.0003	0.02	0.002	99.93

The alloy was produced by synthetic method in the induction furnace IAC-0.16 **(Fig. 2)** with charge material loading of 50 kg. per one melting. Aluminum grade A85, as well as alloying and modifying additives corresponding to GOST R 53777—2010 were used as the base of the alloy. The chemical composition of the alloy was analyzed on an optical emission spectrometer "Foundry master lab". During the experimental melts, the temperature of the molten metal in the induction furnace was 850 °C,

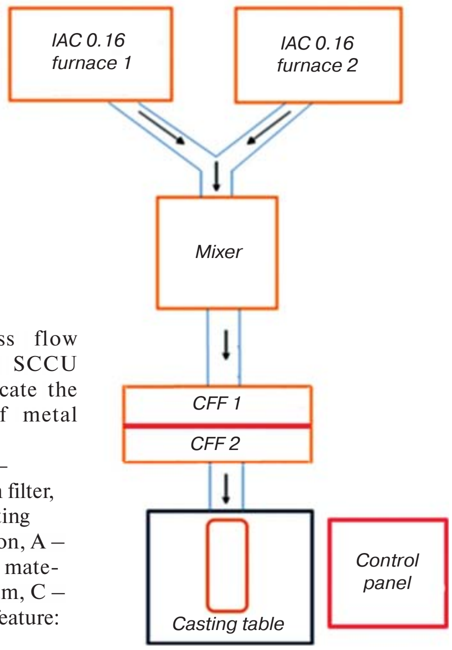

Fig. 1. The process flow diagram of SCCU (arrows indicate the direction of metal movement) (Note: CFF – ceramic foam filter, IAC: I – heating type: induction, A – main melting material: aluminum, C – basic design feature: crucible)

Fig. 2. Semi-continuous casting unit (*photo by authors*)

Table 2
Test matrix at the SCCU

Modifier	Ligature consumption, kg/t.					
Al – 5Ti – 1B	0	0.5	1	1.5	2	2.5
Al – 3La – 1.5B	0	0.5	1	1.5	2	2.5

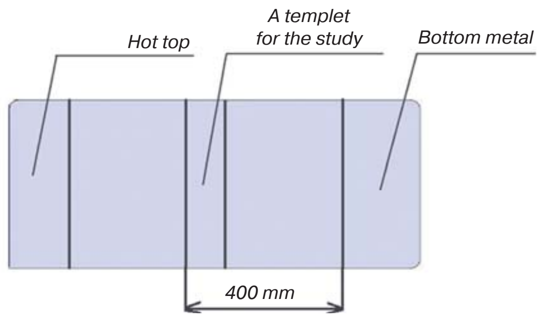

Fig. 3. Scheme of templet sampling for research

in the mixer 750 °C and 700-705 °C on the casting machine table. After reaching the required chemical composition, the melt was poured into a rotary mixer, where the addition of modifying ligature (**Fig. 2**) Al — 5Ti — 1B and Al — 3La — 1.5B with different concentration of modifier was carried out. Feeding of the prepared alloy into the crystallizer of the casting machine was carried out from the mixer, the speed of inclination of which automatically varies depending on the casting speed of the ingot and the level of molten metal in the crystallizer (**Fig. 2**).

Templates for grain size analysis were taken from a flat ingot with a cross section of 60×200 mm. at a distance of 400 mm from the bottom of the ingot **(Fig. 3)**. All obtained macrostructures were analyzed using a Carl Zeiss Stemi 2000C stereomicroscope.

In accordance with the purpose of the work, the number of experiments was determined, taking into account the controlled parameters. Test matrix is presented in **Table 2**.

Results and Discussion

The microstructure of the investigated modifying ligatures is presented in **Fig. 4**. The microstructure of Al — 5Ti — 1B ligature shows homogeneous distribution of intermetallides along the ingot cross-section (**Fig. 4, *a***). The average size of small uniformly distributed Al_3Ti particles reaches 45 μm, the size of large particles corresponds to 70 μm. The average size of TiB_2 particles that will be the center of crystal nucleation is 2.77 μm. Agglomerates of TiB_2 inclusions are drawn into lines, the size of which is in the range of 25—100 μm. (**Fig. 4, *a***). Titanium diborides, which are not bound in agglomerates, have sizes

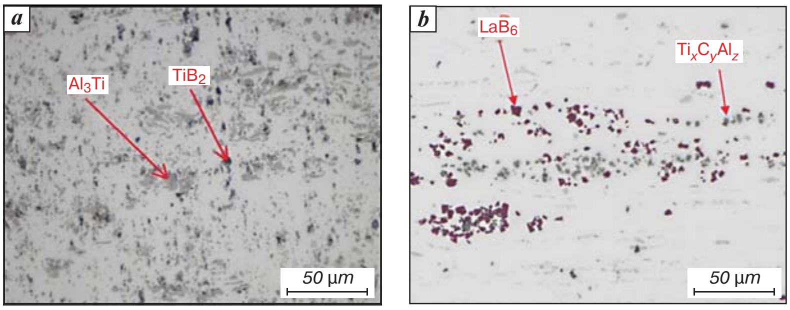

Fig. 4. Typical microstructure of investigated ligatures (magnification ×500): *a* – Al – 5Ti – 1B ligature rod, *b* – Al – 3La – 1.5B ligature rod

up to 5 µm. The average size of non-metallic inclusions in the ligature bar was 8 µm and the maximum size was 112 µm. The results of quantitative analysis of the microstructure of Al – 5Ti – 1B ligature rod are presented in **Table 3.**

On the microstructure of Al – 3La – 1.5B ligature rod **(Fig. 4, *b*)**, the LaB_6 phase has a pink color, inclusions of $Ti_xC_yAl_z$ impurity particles (with traces of Fe, La, V) of gray color have the form of polyhedrons. The average size of LaB_6 particles, which will be the center of crystal nucleation is 1.15 µm, $Ti_xC_yAl_z$ 0.63 µm. They are located mainly in association with each other, distributed unevenly along the cross-section in the form of individual small and enlarged crystals, as well as in the form of multiple coarse string clusters oriented along the deformation direction of the ligature bar. Oxide films are present in the ligature with a maximum size of 109 µm, with an average oxide size of 28 µm. The average size of non-metallic inclusions in Al – 3La – 1.5B ligature was 18 µm, with a maximum size of 354 µm. The results of quantitative analysis of Al – 3La – 1.5B rod ligature are presented in **Table 4.**

According to the results of the modifying ligatures microstructure study, it was found that in the Al – 5Ti – 1B

Table 3
Results of quantitative analysis of Al – 5Ti – 1B microstructure

Indicator	Unit of measurement	Analysis result
Al_3Ti	Max, µm	70
	average, µm	45
TiB_2	Max, µm	5.02
	average, µm	2.77
Size of particle clusters TiB_2	Max, µm	100
	average, µm	25
Non-metallic inclusions	Max, µm	112
	average, µm	8

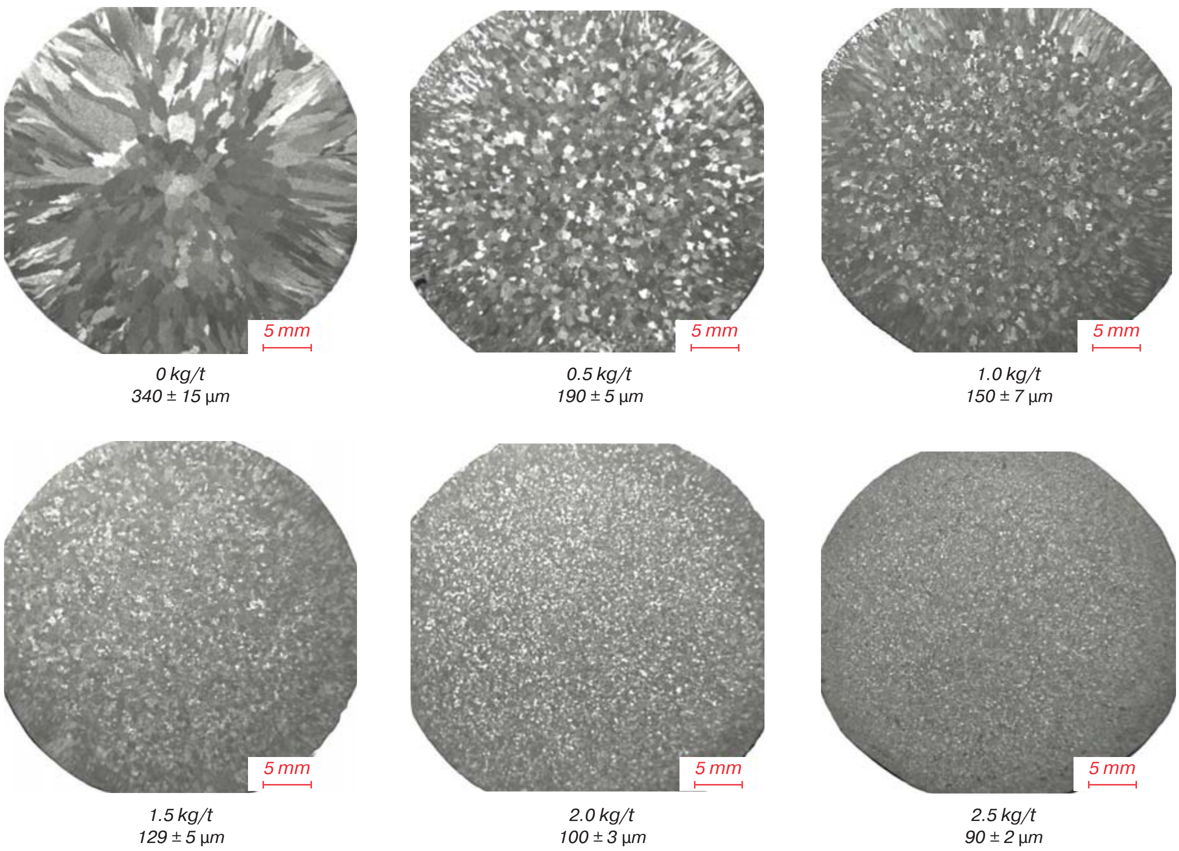

Fig. 5. Effect of modification by Al – 5Ti – 1B ligature with different concentration of modifier

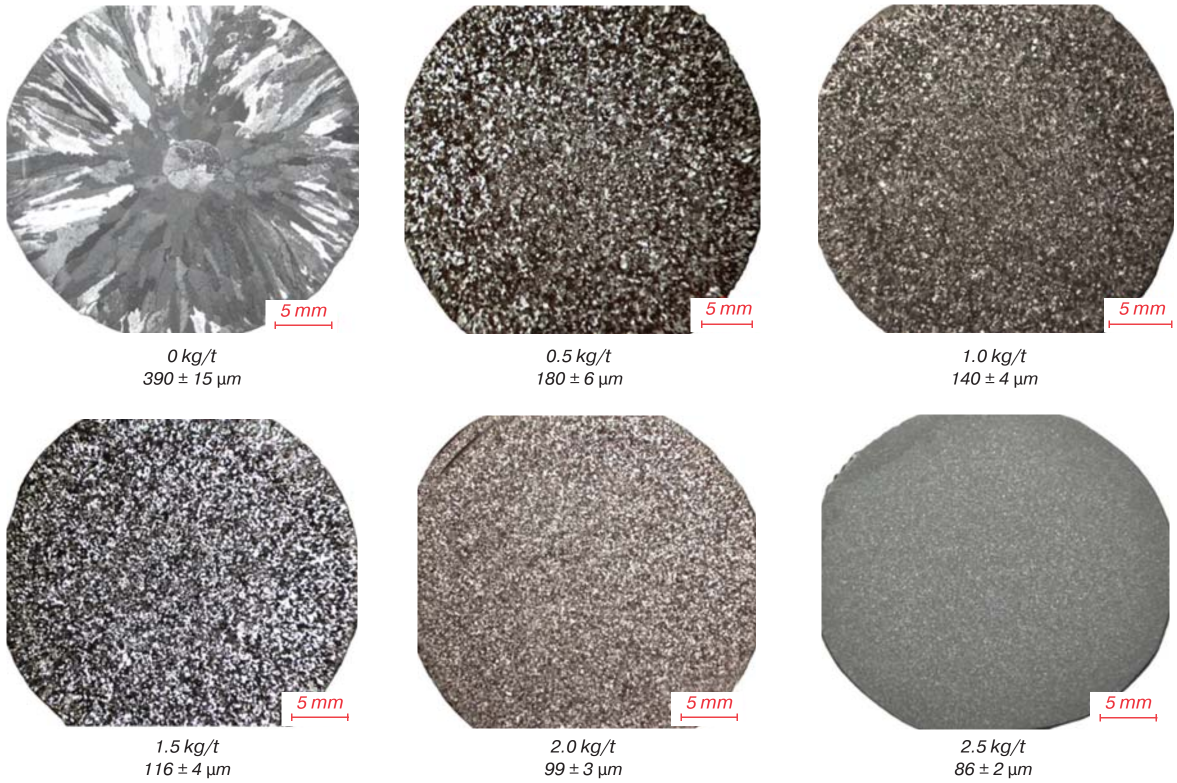

Fig. 6. Effect of modification with Al – 3La – 1.5B ligature with different modifier concentration

Table 4
Results of quantitative microstructure analysis of Al – 3La – 1.5B

Indicator	Unit of measurement	Analysis result
LaB_6	Max, µm	1.37
	average, µm	1.15
$Ti_xC_yAl_z$	Max, µm	0.75
	average, µm	0.63
Oxide films	Max, µm	109
	average, µm	28
Non-metallic inclusions	Max, µm	354
	average, µm	18

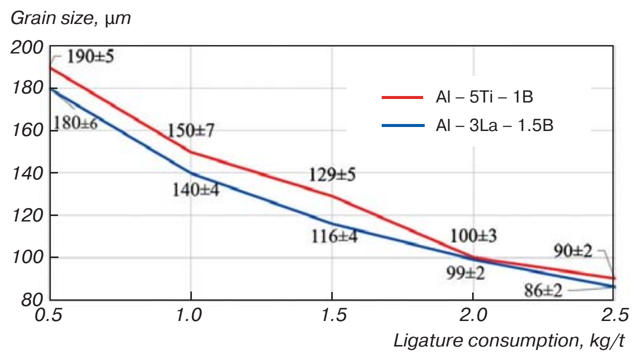

Fig. 7. Change in grain size as a function of modifier consumption

ligature rod refractory intermetallic particles of titanium boride (TiB_2) with melting point of 3230 °C have an average size of 2.77 µm, and in the Al – 3La – 1.5B ligature rod refractory particles are lanthanum borides (LaB_6) with melting point of 2740 °C and an average size of 1.15 µm. Since the average particle size of LaB_6 is smaller than the average particle size of TiB_2, it can be concluded that the promising modifying ligature Al – 3La – 1.5B has better modifying ability [15].

To confirm these results, comparative studies of the modifying ability of Al – 5Ti – 1B ligature (**Fig. 5**) and a promising modifying ligature Al – 3La – 1.5B (**Fig. 6**) have been carried out.

The results of the study of modifying ability of Al – 5Ti – 1B ligature (**Fig. 5**) at consumption of ligature rod from 0.5 kg/t to 1.5 kg/t shows that the sample structure is represented by predominantly equiaxed uniformly distributed grains with average grain sizes from 190 to 129 microns, which are surrounded by a zone of columnar crystals at the edges. At modifier consumption from 2 kg/t to 2.5 kg/t the highest modification efficiency is achieved and the absence of columnar crystals zone with uniform distribution of grains with average grain size from 100 to 90 microns is observed on the whole investigated surface, respectively. The results of research of modifying ability of Al – 3La – 1.5B ligature (**Fig. 6**) at consumption of ligature rod from 0.5 kg/t to 1.5 kg/t shows that the structure of the sample is represented mainly by equiaxed evenly distributed grains of metal, with no zone of columnar crystals and with grain sizes from 180 to 100 microns, respectively. At modifier consumption from 2 kg/t to 2.5 kg/t, very high modification efficiency is achieved and no columnar crystal zone is observed over the entire surface under study with uniform distribution of grains with average grain size from 99 to 86 µm, respectively.

The results of comparative studies of modifying ability of Al – 5Ti – 1B ligature and promising modifying ligature Al – 3La – 1.5B are shown in the graph (**Fig. 7**), the abscissa axis shows the ligature consumption in kilograms per ton, the ordinate axis shows the average grain size in micrometers.

Conclusion

According to the results of the study of modifying ligatures, it was found that in the Al – 5Ti – 1B ligature rod refractory intermetallic particles of titanium boride (TiB_2) have an average size of 2.77 µm, and in the Al – 3La – 1.5B ligature rod refractory particles of lanthanum boride (LaB_6) have an average size of 1.15 µm, which is 2.5 times smaller.

In the course of experiments it was found that at the consumption of ligature rod Al-5Ti-1B from 0.5–1.5 kg/t macrostructure of the obtained samples is represented mainly by equiaxed uniformly distributed grains with average grain size from 190 to 129 microns, respectively, with a zone of columnar crystals at the edges. At the consumption of this modifier from 2–2.5 kg/t the highest efficiency is achieved and the average grain size is from 100 to 90 microns, respectively.

Macrostructure of samples modified with Al – 3La – 1.5B ligature at ligature bar consumption from 0.5–1.5 kg/t is represented by equiaxed uniformly distributed metal grains with average grain size from 180 to 100 microns. At modifier consumption from 2–2.5 kg/t, very high modification efficiency is achieved with average grain size from 99 to 86 microns, respectively.

According to the experimental results, it can be concluded that the modifying ligature Al – 3La – 1.5B, regardless of contamination by oxide films and a higher number of non-metallic inclusions, has a better modifying ability at a consumption of 0.5–1.5 kg/t, which is confirmed by experiments conducted in the laboratory conditions of the Siberian Federal University at the semi-continuous casting unit (SCCU). Thus, the use of modifying ligature Al – 3La – 1.5B in comparison with ligature Al – 5Ti – 1B allows to obtain ingots with lower metal grain size. These studies were conducted on a laboratory unit and the tasks of the research team did not include finding out the economic effect of the implemented development, but the authors assume that the implementation of the research results will potentially increase the competitiveness of finished products. Since research on this subject is still ongoing, it will be possible to discuss economic efficiency when the proposed technology is implemented at a specific production facility.

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References

1. Partyko E. G. Research and Improvement of the Degassing Process in the Blank Casting of Aluminum and Its Alloys: Diss. ... Candidate of Technical Sciences. Krasnoyarsk, 2023. 164 p.

2. Sidorov A. Yu. Improving the Technology of Production of Large-Sized Flat Ingots from Aluminum Alloys of the 5xxx Series to Reduce Porosity: Diss. ... Candidate of Technical Sciences. Krasnoyarsk, 2021. 120 p.

3. Rozhin A. V. Improving the Processes of Alloying and Modifying Aluminum Alloys: Abstract of the Diss... Candidate of Technical Sciences. Eekaterinburg, 2013. 23 p.

4. Wang Y., Fang C. M., Zhou L., Hashimoto T., Zhou X., Ramasse Q. M., Fan Z. Mechanism for Zr Poisoning of Al –Ti – B Based Grain Refiners. *Acta Materialia*. 2019. Vol. 164. pp. 428–439.

5. Kostin I. V. Research and Improvement of the Modification Process of Flat Ingots Made of Aluminum Alloys of the 5xxx Series: Diss. ... Candidate of Technical Sciences. Krasnoyarsk, 2018. 160 p.

6. Napalkov V. I., Baranov V. N., Frolov V. F. Aluminum Ligatures: Structure and Purpose. Krasnoyarsk: SFU, 2019. 175 p.

7. Stepanenko N. A., Kulikov B. P., Kosovich A. A., Belyaev S. V., Konstantinov I. L., Partyko E. G., Yuriev P. O. A Comparative Analysis of Technologies for Producing Al – 3Ti – 1B Modifying Master Alloy. *Metallurgist*. 2023. Vol. 67. pp. 1166–1177.

8. Li D., Cui C., Wang X., Wang Q., Chen C., Liu S. Microstructure Evolution and Enhanced Mechanical Properties of Eutectic Al – Si Die Cast Alloy by Combined Alloying Mg and La. *Materials & Design*. 2016. Vol. 90. pp. 820–828.

9. Qiu C., Miao S., Li X., Xia X., Ding J., Wang Y., Zhao W. Synergistic Effect of Sr and La on the Microstructure and Mechanical Properties of A356.2 Alloy. *Materials & Design*. 2017. Vol. 114. pp. 563–571.

10. Jing L., Pan Y., Lu T., Li C., Pi J., Sheng N. Application of Al – 2La – 1B Grain Refiner to Al – 10Si – 0.3Mg Casting Alloy. *Journal of Materials Engineering and Performance*. 2018. Vol. 27. pp. 2838–2843.

11. Kosovich A. A., Bogdanova T. A., Partyko E. G., Stepanenko N. A. Prospects for Using La-Containing Materials in Al-Alloys for the Automotive Industry. *Youth and Science: Current Problems of Fundamental and Applied Research. Materials of the VI All-Russian National Scientific Conference of Young Scientists. In 3 parts. Pt. 1.* Komsomolsk-on-Amur, 2023. pp. 137–140.

12. Hosseinifar M., Malakhov D. V. Effect of Ce and La on Microstructure and Properties of a 6xxx Series Type Aluminum Alloy. *Journal of Materials Science*. 2008. Vol. 43. pp. 7157–7164.

13. Aluminium-Titanium-Boron (AlTiB). URL: https://www.kbmaffilips.com/aluminium-based/aluminium-titanium-boron/ (Accessed Date: 21.06.2024).

14. AlLa3B1.5 Special Grain Refiner For Al-Si Casting Alloys. URL: https://www.fy-altic.com/aluminum-lanthanum-boron/alla3b1-5-special-grain-refiner-for-al-si.html (Accessed Date: 21.06.2024).

15. Napalkov V. I., Makhov S. V., Pozdnyakov A. V. Modification of Aluminum Alloys. Moscow: Izdatelskiy Dom MISiS, 2017. 348 p.

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