X-ray microanalysis of silumin subjected to intense pulsed electron beam treatment

E A Petrikova, A D Teresov, O S Tolkachov and Yu F Ivanov
Institute of High Current Electronics SB RAS, Tomsk, 2/3 Akademichesky Ave., 634055, Russia

E-mail: yufi55@mail.ru

Abstract. Comparative studies of the distribution of alloying and impurity chemical elements in a cast aluminum and after irradiation with an intense pulsed electron beam have been carried out using X-ray microanalysis methods. It is shown that electron-beam processing of silumin is accompanied by the formation of a submicro–nanocrystalline structure of high-speed cellular crystallization. It was established that the substructure of the cells depends on the distance from the irradiation surface. In the layer near the surface, nanoscale globular particles are present in the cell volume; in the subsurface layer, the formation of cells of the plate-like eutectic of the Al-Si alloy was revealed.

1. Introduction
Al-Si alloys (silumin), due to their low specific weight, relatively high specific strength, good fluidity, belong to cast alloys that are widely used in aircraft and shipbuilding [1]. A clear disadvantage of silumin is the presence of coarse inclusions of the second phases, leading to a high brittleness of the material. One of the methods of radical transformation of the material structure is high-speed heat treatment based on the use of concentrated energy flows (electron and ion beams, plasma flows, laser beams, etc.) [2–10]. In [11–14], it was shown that the irradiation of silumin with an intense pulsed electron beam in the mode of melting of the surface layer up to 100 μm thick is accompanied by the formation of a submicro nanocrystalline multiphase structure with high tribological and mechanical properties.

The purpose of this work is to establish the patterns of distribution of alloying elements in the surface layer of silumin, subjected to irradiation by an intense pulsed electron beam.

2. Material and method
The silumin AK10M2N and AK12 [15] was used as a test material. Silumin was irradiated at the SOLO setup [16] with the following parameters: the energy of accelerated electrons is 17 keV; electron beam energy density 20–35 J·cm⁻²; irradiation pulse duration 150 μs; number of pulses 3; pulse repetition rate 0.3 s⁻¹. Irradiation was performed in argon plasma at a pressure of 0.02 Pa. The elemental composition and the state of the defective substructure of the samples of cast silumin and silumin after electron beam irradiation were studied by scanning (Philips SEM 515) and transmission electron microscopy methods (JEM 2100F) [17–20].
3. Results and discussion

The structure of silumin in the cast state is characterized by the presence of coarse silicon inclusions and intermetallic compounds of various shapes and sizes (figure 1a). Methods of X-ray microanalysis allow locally to carry out a study of the elemental composition of a particular chosen volume of the alloy [18]. The parts of silumin subjected to X-ray microanalysis in the cast state are shown in figure 1b. The results obtained are presented in table 1. It is clearly seen that the alloying elements of the alloy are distributed heterogeneously in the material, forming inclusions that differ in size, morphology and elemental composition.

![Figure 1](image1.png)

**Figure 1.** Electron-microscopy image of silumin brand AK12 structure in the cast state; the numbers in (b) denote the areas in which the X-ray microanalysis of the elemental composition of the material was performed. Scanning electron microscopy.

| Field | Element (the rest of Al, wt.%) |
|-------|------------------------------|
|       | Si  | Ni  | Cu  | Fe  | Mn  |
| 1     | 0.6 | 13.5| 13.3| 0.0 | 0.0 |
| 2     | 8.7 | 0.3 | 2.2 | 0.0 | 0.0 |
| 3     | 1.7 | 11.8| 14.0| 0.0 | 0.0 |
| 4     | 0.5 | 0.2 | 1.3 | 0.0 | 0.0 |
| 5     | 22.5| 1.1 | 1.6 | 1.2 | 0.0 |
| 6     | 1.1 | 14.8| 15.8| 0.5 | 0.0 |
| 7     | 2.3 | 17.2| 5.2 | 2.7 | 0.6 |

**Table 1.** Results of the X-ray microanalysis of the surface area of cast silumin, an electron microscopy image of that is shown in figure 1b.

Transmission electron microscopy methods (STEM method) allow to conduct X-ray microanalysis of the material using thin foils [17, 18]. An example of such an analysis (mapping method) is presented in figure 2. It is clearly visible that the micron-sized silicon crystal is surrounded by a shell of magnesium atoms. The elemental composition of the foil area is shown in figure 2, looks as follows (at.%): 74.2% Al, 23.6% Si, 1.4% O, 0.6% Mg, 0.2% Fe.

Figure 3 shows another example of the study of the alloying elements atoms distribution in a cast silumin, performed by X-ray microanalysis methods (STEM method). The elemental composition of the foil sections is shown in figure 3, is presented in table 2.
Figure 2. X-ray microanalysis of the silumin brand AK12 elemental composition in the cast state (STEM method); a – bright-field image; b, c – images obtained in the characteristic X-rays of the silicon atoms (b) and magnesium (c).

Figure 3. Electron microscopic images of the structure of cast silumin brand AK12, obtained in the STEM analysis mode in the study of the elemental composition of the material.

Table 2. Results of X-ray microanalysis of characteristic sections of cast silumin foil, electron microscopic images of which are shown in figure 3.

| Field      | Si  | Ni  | Cu  | Fe  | Mg  | O   |
|------------|-----|-----|-----|-----|-----|-----|
| figure 3a  | 9.2 | 1.2 | 1.8 | 0.3 | 2.0 | 0.0 |
| figure 3b  | 0.4 | 0.0 | 0.0 | 0.1 | 1.0 | 1.2 |
| figure 3c  | 15.9| 0.0 | 0.8 | 0.0 | 0.6 | 0.8 |
| figure 3d  | 1.0 | 0.0 | 1.5 | 0.1 | 0.5 | 1.6 |

Thus, studies of silumin in the cast state, performed by X-ray microscopic analysis, revealed a high level of localization of alloying and impurity elements that form inclusions of the second phase of micron size, negatively affecting the plasticity properties of the material.

The irradiation of silumin with an intense pulsed electron beam in the surface layer melting mode is accompanied by the formation of a structure of high-speed cellular crystallization (figure 4a). The cell size varies from 250 to 600 nm and increases with increasing electron beam energy density. Crystallization cells are separated by layers of the second phase (figure 4a). The transverse dimensions of the interlayers do not exceed 100 nm. It was established that in the surface layer with a thickness of (10–20) μm, the cell volume is free from inclusions of the second phase, or contains nanosized...
(10–20 nm) round particles (figure 4a). At a greater distance from the irradiation surface, the formation of cells with a lamellar substructure is observed (figure 4b).

![Figure 4. STEM image (dark field) of the structure of silumin brand AK10M2N in the cast state, subjected to irradiation by an intense pulsed electron beam (25 J cm\(^{-2}\); 150 μs; 3 pulses).](image)

Study of the elemental composition of the cellular structure, which is formed upon irradiation of silumin by an intense pulsed electron beam have been conducted by X-ray microanalysis methods. The results of the analysis are given in table 3.

**Table 3.** Results of X-ray microanalysis of the cellular structure of silumin brand AK10M2N, irradiated with an intense pulsed electron beam (25 J cm\(^{-2}\); 150 μs; 3 pulses). Fitting Coefficient: 0.2129

| Element (keV) | Mass % | Counts | Error % | Atom % |
|--------------|--------|--------|---------|--------|
| Mg K         | 1.253  | 0.71   | 2140.4  | 0.44   | 0.81   |
| Al K         | 1.486  | 86.09  | 259994.77 | 0.0   | 89.41  |
| Si K         | 1.739  | 6.90   | 21650.79 | 0.05  | 6.88   |
| Ti K         | 4.508  | 0.23   | 535.54  | 1.99   | 0.13   |
| Mn K         | 5.894  | 0.03   | 59.06   | 20.87  | 0.01   |
| Fe K         | 6.398  | 0.48   | 956.06  | 1.18   | 0.24   |
| Ni K         | 7.471  | 1.26   | 2214.5  | 0.56   | 0.60   |
| Cu K         | 8.040  | 4.32   | 6702.6  | 0.2    | 1.9    |
| Total        | 100.0  |        |         | 100.0  |        |

It is clearly seen that the main element of the analyzed layer is aluminum; significantly lower amounts of alloying elements are present, the main of which are silicon, copper, nickel and magnesium. The distribution of these chemical elements in the structure of cellular crystallization was studied by mapping methods. It was found that the volume of crystallization cells is enriched with aluminum atoms and, in a substantially smaller quantity, with magnesium and copper atoms. The layers separating the crystallization cells are enriched mainly by silicon, nickel and copper atoms (figure 5).
Figure 5. X-ray diffraction analysis (mapping method) of the elemental composition of AK10M2N silumin surface layer irradiated with an intense pulsed electron beam (25 J·cm\(^{-2}\); 150 μs; 3 pulses); a – bright-field image; b, c – images obtained in the characteristic X-rays of silicon (b) and nickel (c) atoms.

Figure 6. X-ray diffraction analysis (mapping method) of the elemental composition of the surface layer of AK10M2N silumin irradiated with an intense pulsed electron beam (25 J·cm\(^{-2}\); 150 μs; 3 pulses); a – bright-field image; b,d – images obtained in the characteristic X-rays of the atoms of aluminum (b) and silicon (c) and copper (d).
The results of X-ray diffraction analysis of the structure of cellular crystallization with a lamellar substructure are shown in figure 6. Studies show that in this case the structure of Al-Si plate eutectics, containing alternating nanoscale (lateral dimensions 30–50 nm) plates of aluminum (figure 6b) and silicon (figure 6c) is formed. Along with nickel-enriched inclusions (figure 5c), inclusions enriched with copper atoms (figure 6d) may be present at the junctions of the boundaries and along the boundaries.

4. Conclusion
It has been shown by X-ray diffraction analysis methods that the alloying and impurity elements of cast silumin are in a localized state and form inclusions of micron sizes of different chemical composition. The irradiation of silumin with an intense pulsed electron beam makes it possible to form a submicron structure of high-speed crystallization of a cellular type in the surface layer up to 100 microns thick. Inclusions of the cast state of silumin are not detected in this surface layer. The formation of cells of two morphologically different types was revealed. Firstly, the cells, which volume is predominantly enriched with aluminum atoms, separated by layers of nanoscale thickness enriched with silicon, nickel, copper, and iron atoms. Secondly, the cells of the nanoscale lamellar eutectic of Al-Si system. The cells of this type are also separated by layers enriched with silicon, nickel, copper, and iron atoms. It is important to note that inclusions of the second phase (silicon and intermetallic compounds), which form the structure of the surface layer, are nanoscale and, undoubtedly, will contribute to plasticization of silumin.

Acknowledgements
The reported study was funded by RFBR and Tomsk region according to the research project No. 19-52-04009.

References
[1] Zolotarevsky V S and Belov N A 2005 Metal science of cast aluminum alloys (Moscow: MISiS) [in Russian]
[2] Pout J M, Foty G and Jacobson D K 1987 Modification and surface alloying with laser, ion and electron beams (Moscow: Mashinostroenie) [in Russian]
[3] Gribkov V A, Grigoriev F I, Kalin B A et al 2001 Promising radiation-beam technologies for metal processing (Moscow: Kruglyy stol) [in Russian]
[4] Shulov V A, Paikin A G, Novikov A S et al 2012 High-current pulsed electron beams for aircraft engine-building (Moscow: Artek) [in Russian]
[5] Kadyrzhanov K K, Komarov F F, Pogrebnyak A D et al 2005 Ion-beam and ion-plasma modification of materials (Moscow: Moscow State University Publishing House) [in Russian]
[6] Uglov V V, Cherenda N N, Anishchik V M, Astashinskiy V M and Kvasov N T 2013 Modification of materials by compression plasma flows (Minsk: BSU) [in Russian]
[7] Yakushin V L 2005 Metallo 212 [in Russian]
[8] Bagautdinov A Ya, Budovskikh E A, Ivanov Yu F and Gromov V E 2007 Physical bases of electroexplosive alloying of metals and alloys (Novokuznetsk: SibSIU Publishing House) [in Russian]
[9] Pogrebnyak A D and Tyurin Yu N 2005 Phys. Uspekhi 175 515 [in Russian]
[10] Rotshtein V, Ivanov Yu, and Markov A 2006 Materials surface processing by directed energy techniques ed Y. Paulau (Paris: Elsevier) chapter 6 p 205
[11] Laskovnev A P, Ivanov Yu F, Petrlikova E A et al 2013 Modification of the structure and properties of eutectic silumin by electron-ion-plasma treatment (Minsk: Belarus. Navuka) [in Russian]
[12] Koval N N and Gromov V E (eds) 2015 Modern trends in modifying the structure and properties of materials (Tomsk: Publishing House NTL) [in Russian]
[13] Koval N N and Ivanov Yu F (eds) 2016 Electron-ion-plasma modification of the surface of non-ferrous metals and alloys (Tomsk: NTL Publishing House) [in Russian]

[14] Zagulyaev D V, Konovalov S V, Gromov V E, Glezer A M, Ivanov Yu F and Sundeev R V 2018 Materials Letters 2209 377

[15] 2000 Aluminium foundry alloys. Technical specifications. State Standart 1583-93 (Minsk: Interstate Committee for Standardization, Metrology and Certification) [in Russian]

[16] Koval N N and Ivanov Yu F 2008 Izvestiya Vuzov. Fizika 5 60 [in Russian]

[17] Thomas G and Gorindzh M J 1983 Transmission electron microscopy of materials (Moscow: Nauka) [in Russian]

[18] Brandon D and Kaplan U 2004 Microstructure of materials. Methods of investigation and control (Moscow: Tekhnosfera) [in Russian]

[19] Utevskii L M 1973 Diffraction electron microscopy in physical metallurgy (Moscow: Metallurgia) [in Russian]

[20] Endrus K, Dyson D and Kyoyn S 1971 Electron diffraction patterns and their interpretation (Moscow: Mir) [in Russian]