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Microdiffraction analysis of structure of silumin's high-velocity cellular crystallization

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Abstract. The submicro-nanocrystalline multiphase structure of high-velocity cellular crystallization has been formed in the surface layer of AK10M2N silumin by the irradiation with the intense pulsed electron beam. According to the morphological features the crystallization cells are divided into two types. The cells of the first type are formed by the alternating plates of silicon and aluminium of nanodimensional transverse sizes by the eutectic reaction. The cells of the second type are formed by the solid solution based on aluminium and contain the nanodimensional particles of the second phase. The cells of the first and the second type are separated by the interlayers of the second phase containing the silicon particles, aluminides and copper silicides. The obtained results enable to recommend the irradiation of silumin by the intense pulsed electron beam as the method resulting in the multiple dispersion of the structure facilitating the increase in wear resistance of the material.

1. Introduction

In the cast state the main structural components of silumin are the grains of solid solution based on aluminium and aluminum-silicon eutectic inside of which the inclusions of other phases [1–3] are practically always present. The important question from the practical point of view is how to get the formation of eutectic structure without α -phase dendrites with disperse inclusions of silicon and intermetallides without changing in the chemical composition of silumin (especially close to eutectic composition). The necessity of silumins with such a structure is connected with the prospects for their application in the frictional units when the wear resistance of a material increases due to the fine inclusions of silicon and the extension of temperature-load conditions of service expand [4, 5].

In the research [6] concerned with the analysis of structure and properties of AK10M2N hypoeutectic silumin it was shown that the irradiation of the material by the intense pulsed electron beam of submillisecond duration enabled to form the structure wherein the dimension of the second phase crystallites was less than 100 nm in the surface layer up to 100 nm thick. On the basis of the performed studies the conclusion has been drawn that pulsed electron beam processing is the



promising method that enables the multiple (up to nanodimensional state) refining of the phases hardening the material without changing in the chemical composition of the alloy.

The purpose of the present research is electron microscopic microdiffraction analysis, invoking the dark-field technique, of the surface layer structure of hypoeutectic silumin irradiated by the intense pulsed electron beam.

2. Material and method

AK10M2N (the USA analog – alloy 383.1) silumin is used as the test material of the following composition: 9.5–10.5 Si, 2.0–2.5 Cu, 0.8–1.2 Ni, 0.9–1.2 Mg, up to 0.6 Fe, up to 0.05 Mn, up to 0.05 Ti, up to 0.05 Pb, up to 0.06 Zn, up to 0.01 Sn, the balance Al, weight % [7] in the cast state. The samples had the form of the plates 20x20x10 mm in size. The irradiation of the plates' surface was done at the plant SOLO (IHCE SD RAS) [8] by the intense pulsed electron beam with the following parameters: the energy of accelerated electrons – 17 keV, the energy density of electron beam – $35 \text{ J}\cdot\text{cm}^{-2}$, the duration of electron beam pulse – 150 μs , the number of pulses – 3, the pulse repetition rate – 0.3 s^{-1} , the pressure of the residual gas (argon) in the working chamber of the plant – $2 \times 10^{-2} \text{ Pa}$. The elemental and phase composition, the morphology of phases and the defect substructure of the samples were studied by the methods of scanning (the device Philips SEM-515) and transmission electron diffraction (the device JEM 2100F) microscopy [9–12].

3. Results and discussion

The aluminium-silicon alloy under study (AK10M2N silumin) in the cast state is a multiphase material whose main structural component is the grains of the solid solution based on aluminium, the aluminium-silicon eutectic, the inclusions of the initial crystals of silicon and intermetallide phases of different morphology and dimensions (figure 1a, the lower part of the image).

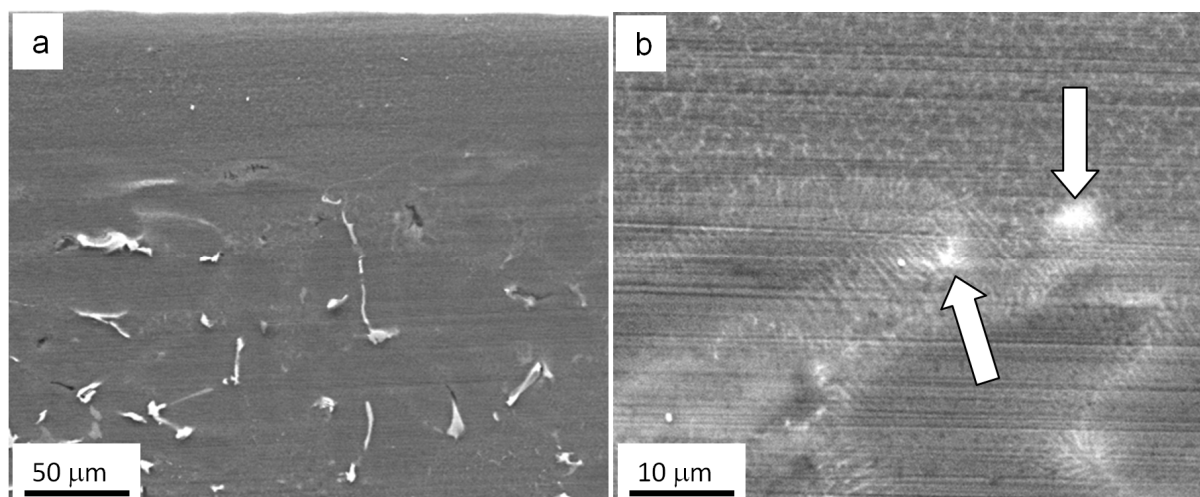


Figure 1. Structure of AK10M2N cast silumin subjected to irradiation by intense pulsed electron beam ($35 \text{ J}\cdot\text{cm}^{-2}$, 150 μs , 3 pulses). The arrows designate insoluble particles of the initial (cast) state. The transverse metallographic section. Scanning electron microscope.

The irradiation of the samples by the intense pulsed electron beam results in the melting of the surface layer up to 100 μm thick (figure 1a). The subsequent high-velocity crystallization is accompanied by the formation of the surface layer whose crystallites' dimension (the cells of high-velocity crystallization) varies within the limits of 1.5–3 μm (figure 1b). It is important to note that the layer is free from the inclusions of the cast state whose images are presented in figure 1a. The surface layer with the cellular crystallization structure transforms to the layer (further the transition layer)

containing incompletely dissolved inclusions of the second phase (figure 1b, the inclusions are designated by the arrows) along with the cells.

By the methods of the transmission electron microscopy it has been shown that the structure of silumin's surface layer irradiated by the intense pulsed electron beam is formed by the cells of high-velocity crystallization of two types (figure 2). To the first type belong the cells in whose volume the structure of the lamellar type (figure 2a) is observed. The transverse dimensions of the plates and the interlayers, separating them, vary within the limits of 30–50 nm. To the second type belong the cells in the volume of which the dislocation substructure (figure 2b) is observed. The dislocations are distributed chaotically, the scalar density of dislocations amounts to $2 \times 10^{10} \text{ cm}^{-2}$. The cells of the first and the second type are separated by the interlayers of the second phase. The transverse dimensions of the interlayers vary within the limits from 50 to 180 nm.

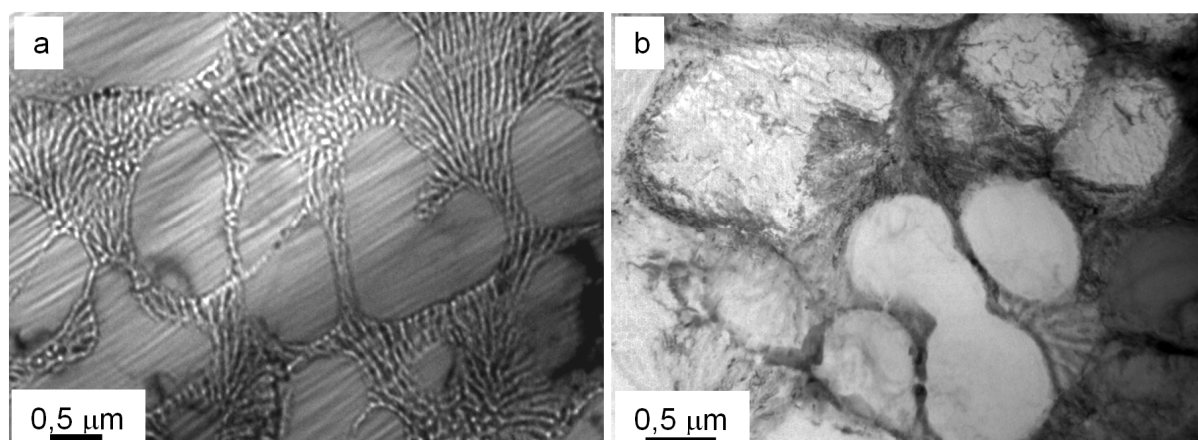


Figure 2. Electron microscopic image of surface layer structure of AK10M2N cast silumin subjected to irradiation by intense pulsed electron beam ($35 \text{ J} \cdot \text{cm}^{-2}$, $150 \mu\text{m}$, 3 pulses). Transmission electron diffraction microscopy.

The phase composition of the cellular high-velocity crystallization structure is studied by the methods of transmission electron diffraction microscopy when analyzing microelectron diffraction patterns and using the dark-field technique [13–15]. The results of the performed studies are shown in figure 3. The microdiffraction analysis of high-velocity crystallization cells has shown that independent of the cells' type their volume is formed by the solid solution based on aluminium (figure 3c). The plates located in the volume of the first type cells are silicon and aluminium (figures 3d–f). Thus, the first type cells are formed according to the eutectic reaction with the formation of nanodimensional lamellar eutectic. In the volume of the second type cells the particles of the second phase $\text{Cu}_{15}\text{Si}_4$ (figure 3f) are revealed by the methods of dark-field analysis. The particles have a round shape and the particles' sizes vary within the limits 10–15 nm.

The results of electron microscopic microdiffraction investigation into the structure of the transition layer are presented in figure 4. The analysis of microelectron diffraction pattern (figure 4b) shows that silicon (figure 4f) is the crystallite undissolved under the irradiation of the sample by the electron beam. The atoms of silicon form the interlayers located on boundaries of crystallization cells (figure 4 f) as well. The volume of crystallization cells is formed by the solid solution based on aluminium (figures 4cd). In the volume of crystallization cells the nanodimensional (5–8 nm) particles of Al_4Cu_9 are detected. The particles of the second phase Cu_5Si and Al_4Cu_9 are found in the form of the interlayers located along the boundaries of crystallization cells (figure 4f). The dimensions of the particles vary within the limits 100–120 nm.

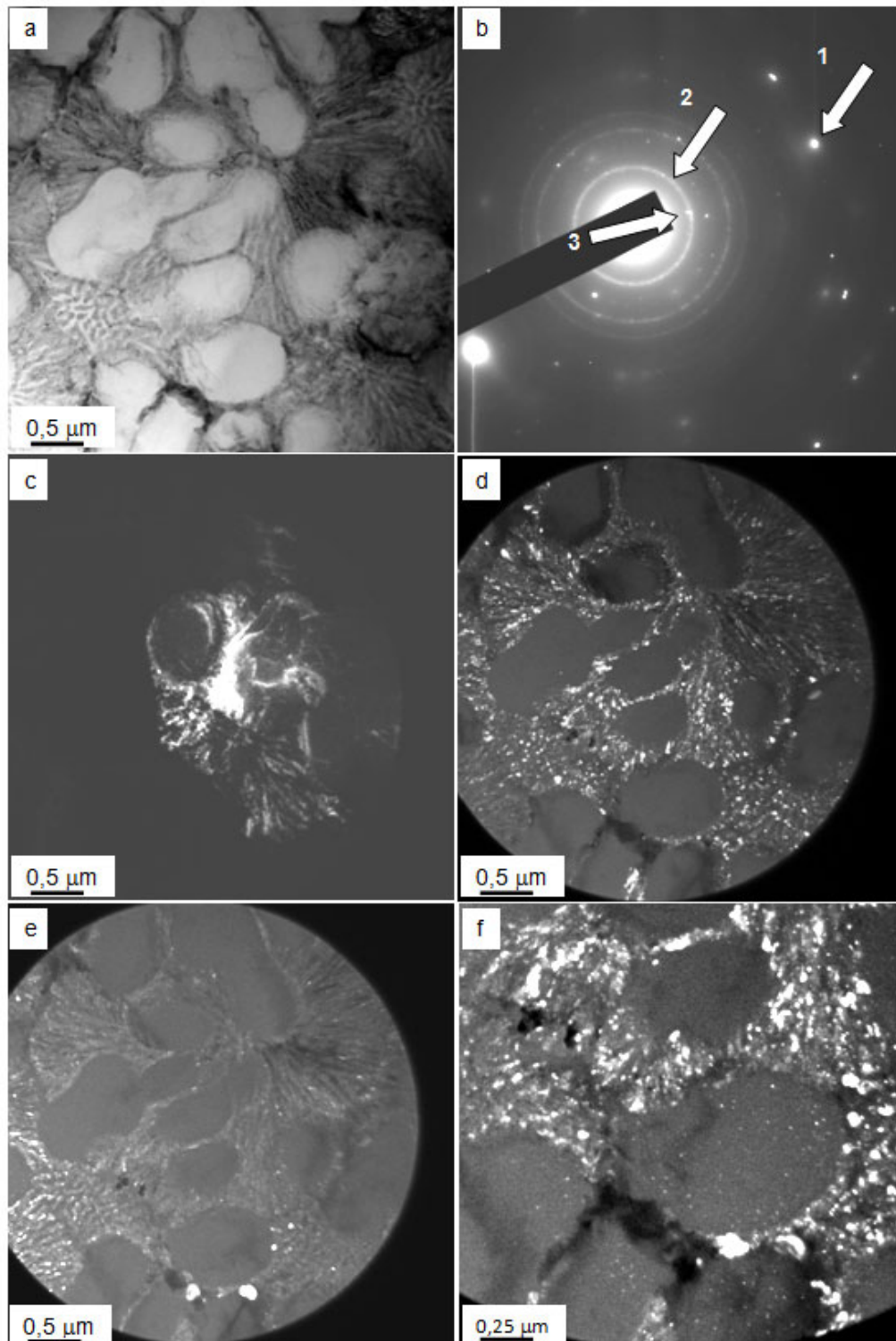


Figure 3. Electron microscopic image of surface layer structure of AK10M2N cast silumin subjected to irradiation by intense pulsed electron beam ($35 \text{ J}\cdot\text{cm}^{-2}$, $150 \mu\text{s}$, 3 pulses); a – bright field; b – microelectron diffraction pattern; c – f – dark fields obtained in reflections [111] Al (c), [111] Si (d), [111] Si + [220] $\text{Cu}_{15}\text{Si}_4$ (e, f). In (b) the arrows designate the reflections of dark-field formation 1 – c, 2 – d, 3 – e, f. Transmission electron diffraction microscopy.

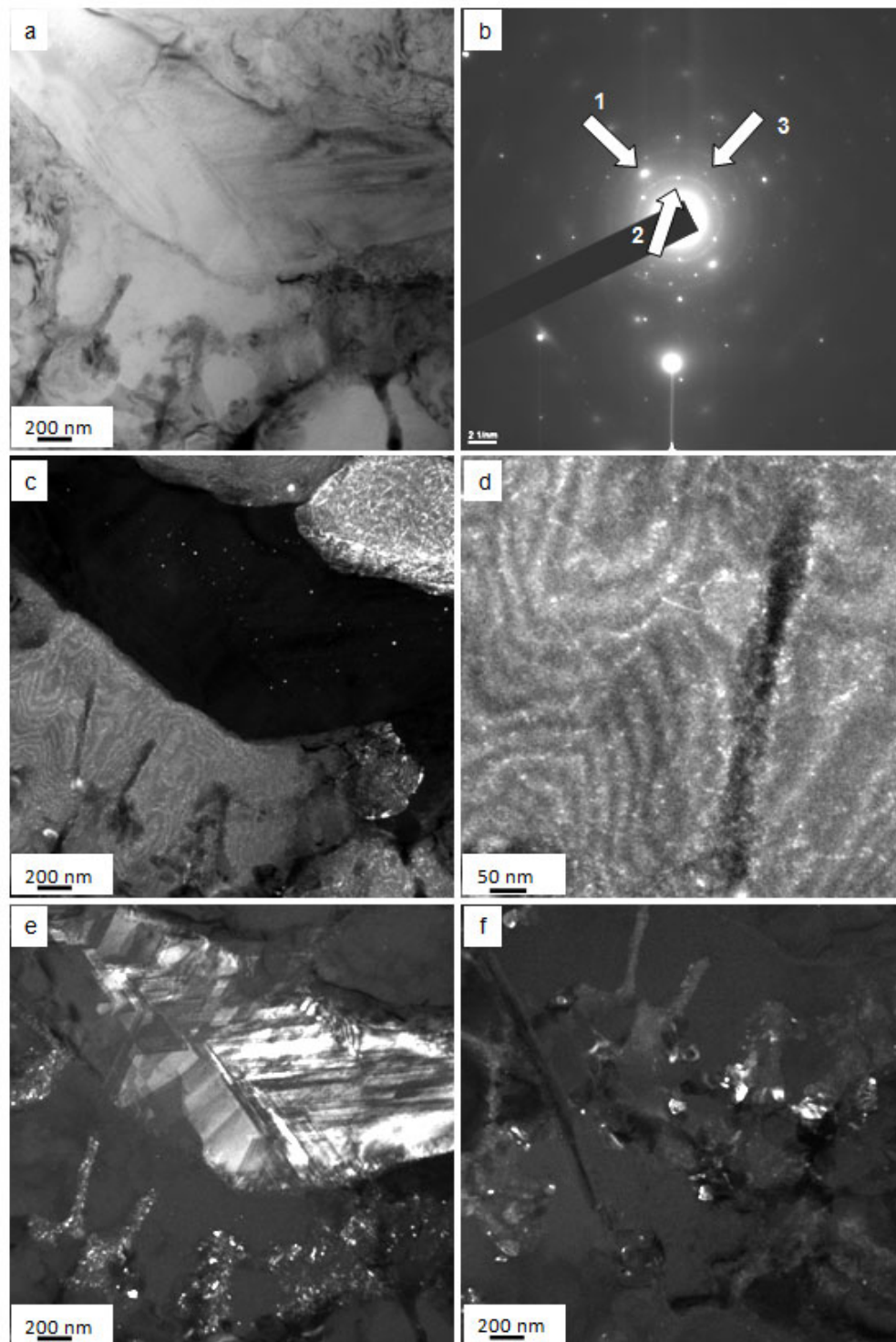


Figure 4. Electron microscopic image of the structure of AK10M2N cast silumin subjected to irradiation by intense pulsed electron beam ($35 \text{ J}\cdot\text{cm}^{-2}$, $150 \mu\text{s}$, 3 pulses) located at $\approx 80 \mu\text{m}$ distance from irradiation surface; a – bright field; b – microelectron diffraction pattern; c-f – dark fields obtained in reflections $[111] \text{ Al} + [111] \text{ Al}_4\text{Cu}_9$ (c, d), $[111] \text{ Si}$ (e), $[222] \text{ Al}_4\text{Cu}_9 + [211] \text{ Cu}_5\text{Si}$ (f). In (b) the arrows designate the reflections of dark field formation 1 – c, d, 2 – e, 3 – f. Transmission electron diffraction microscopy.

4. Conclusion

The investigations into the elemental and phase composition, the state of defect substructure of hypoeutectic silumin irradiated in vacuum by intense pulsed electron beam ($35 \text{ J}\cdot\text{cm}^{-2}$, $150 \text{ }\mu\text{s}$, 3 pulses) were performed by the methods of diffraction electron microscopy. The formation of the structure of high-velocity crystallization of submicron dimensions has been detected. According to the morphological feature it is suggested to divide the crystallization cells into two types. The first-type cells are formed according to the eutectic reaction and contain the alternating plates of silicon and aluminum of nanodimensional transverse dimensions. The second-type cells are formed by the solid solution based on aluminium and contain nanodimensional particles of the second phase. The cells of the first and the second type are separated by the second phase interlayers. The nanodimensional particles of silumin, aluminides and copper silicides have been revealed in the interlayers by the methods of microdiffraction analysis. The obtained results enable to recommend the irradiation of silumin by intense pulsed electron beam as the method resulting in the multiple dispersion of the structure (the formation of submicro-nanodimensional multiphase state) that should facilitate the increase in wear resistance of the material.

Acknowledgments

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