

Microconstituents of the Modified Surface Layer of Austenitic Steel With Nanofibres of Aluminium Oxyhydroxide

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Abstract. In the paper the authors provide the results of experimental study of the effect caused by introduction of nanostructured fibres of aluminium oxyhydroxide into the surface layer of austenitic steel upon its microconstituents. The authors show that, due to introduction of given fibres dendrite size is reduced and equilibrium structure is formed.

1. Introduction

Under various methods of metal surface layer formation the crystalline structure of the given layer influenced by the conditions of liquid-solid transition is one of the factors determining the quality and the properties of this layer [1].

It is known that the process of nucleation can be spontaneous and induced [2]. In the build-up surface layer induced crystallization usually occurs at the fusion boundary where high-melting phases and microconstituents of the base metal may become nucleation centers [1]. In practice high-melting particles are deliberately introduced into the molten metal to increase the amount of induced nucleation centers which results in refinement of grains as the metal solidifies [1, 2].

Currently there are several promising ways used to modify surface layers with powders to obtain brand new properties of the surface: plasma, laser, ultrasonic, treatment with electric arc.

Electric arc was chosen as the means of surface activation as it is widely used in industry and allows introduction of nanodisperse particles after only slight modification of standard equipment.

The electric arc can be applied in three different ways depending on the place and method of application: when remelting the surface, when building-up the surface and for high-temperature treatment of the surface with application of consumable electrode [3, 4, 5].

The aim of the given research work was controlling the process of structure formation of the built-up layer produced from steel (chemical composition of the wire: 0.12% carbon, 18% chromium, 9% nickel; 1-15% titanium).



Relevance of the problem is determined by the fact that the structure directly conditions (specifies) the mechanical and service properties of the surface, i.e. insufficient service properties result in a larger amount of equipment failures and significant time and material losses caused by excessive repairing work [6, 7].

Thus, solution of the problem of properties improvement directly depends upon providing theoretical grounds and experimental verification of homogeneous fine-grained surface structure formation mechanism after introducing nanostructured powder modifiers into the surface layer.

2. Methods of research

To conduct the research built-up modified surface layers were produced. Two types of samples were used: №1 – modified with nanostructured aluminium oxyhydroxide fibers; №2 – without modification. For the purposes of the research the steel (chemical composition of the wire: 0.12% carbon, 18% chromium, 9% nickel; 1-15% titanium) samples were built-up by MIG-welding (welding conditions: current intensity 240-260 A; voltage 28-30 V) in argon atmosphere with electrode wire 12H18N9T 1.2 mm in diameter.

To study the microstructure sections were prepared. To prepare the sections the following methods were applied: mechanical grinding, mechanical polishing with diamond paste ASM 10/7 NVL and chemical etching (hydrochloric acid 75% HCl + concentrated nitric acid 25% HNO₃). The microsections were studied with application of optical metallography method using Neophot-21 microscope and digital camera Genius VileaCam for image recording.

In the given work we applied aluminium oxyhydroxide fibres AlO(OH) (Figure 1) of the following size: diameter of 5 nanometer, length 150 nanometer; specific surface area 150 m²/g. For AlO(OH) fiber synthesis aluminium conductor electric explosive-produced powders were applied with specific surface area 7.5 m²/g. Aluminium powders were made from aluminium wire "AM" Ø 0,35 mm. Then nanostructured Al powder was subjected to thermal hydrolysis to produce Al oxyhydroxide nanofibers (AlO(OH)). Aluminium electro-explosive powder was placed into distilled water which had been previously heated up to 60°C. The aluminium powder reacted with the water under the given temperature for 25-30 minutes. The color of the aqueous suspension changed from dark-grey to white. 10 minutes after the suspension changed its color the container with the reaction mass was taken out of the constant-temperature bath. After cooling to the room temperature suspension was filtered, the deposit was washed with several portions of distilled water (3x300 ml) until its reaction was neutral (pH 5.5-6). Then the powders were dried under 110-115°C. Microphotographs of aluminium oxyhydroxide were presented in Figure 1 [8, 9, 10, 11].

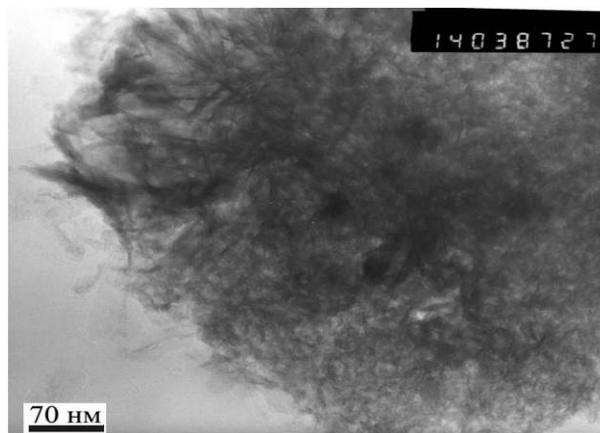


Figure 1 – Transmission electron microscopy: photograph of aluminium oxyhydroxide fiber

Aluminium electric explosive-produced powders on UDP-150 device have spherical form, the size of particles varies within (145-150) nanometer, specific surface area varies within (15-16) m²/g [8, 10, 12].

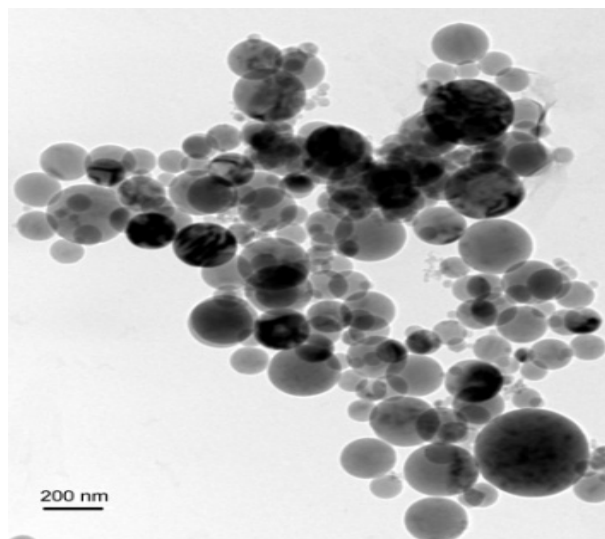


Figure 2 - Transmission electron microscopy of electric explosive-produced powders: aluminium

3. Results of the research and their discussion

Examination of the sections that had not been etched showed that the surface layers built-up with application of two different methods do not have macro- and microdefects.

The samples were studied according to the diagram presented in Figure 3.

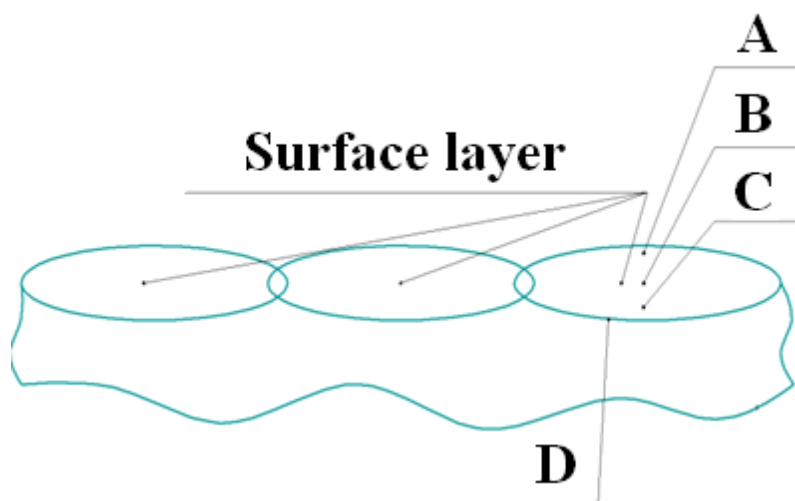


Figure 3 – Examination of the microstructure of the surface layer: A – upper underlayer, B – central underlayer, C – bottom underlayer, D – area of transition from the surface layer to the base one, E – base metal

Point D shows metal structure at the border of fusion and heat-affected zone (HAZ) (Fig. 4). Structure of heat-affected zone does not differ much from that of the base metal. The sample with aluminium oxide has smaller heat-affected zone which corresponds to less heat input and fast cooling.

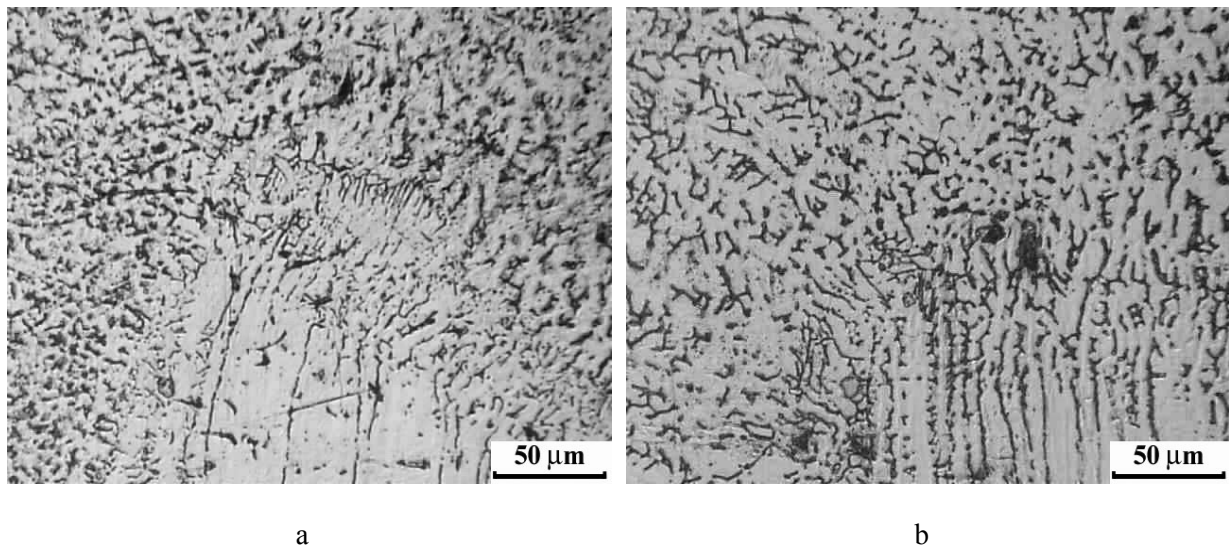


Figure 4 – Structure of the metal at the border between the fusion and heat-affected zone: a – sample №1 modified with nanostructured fibers of AlO(OH); b – sample №2 without the modifier

The grain structure of the heat-affected zone transits to surface layer structure with pronounced dendrites.

The first layer with polyhedral grain structure. In this underlayer polyhedral grains of austenite are observed together with dendrites. This underlayer is not well pronounced in sample №2 produced without the modifier (Fig. 5 b). Its thickness is 20% of the general thickness of surface layer.

The “grain” underlayer is better pronounced in sample № 1 modified with nanostructured fibers of aluminium oxyhydroxide (Fig. 5 a). Here the polyhedral morphology grains can be well seen as they alternate with nonoriented dendrites. The thickness of the considered underlayer is over 30% of the general thickness of the surface layer. The specific feature of the given underlayer is that the grains may contain short and strongly ramified dendrites.

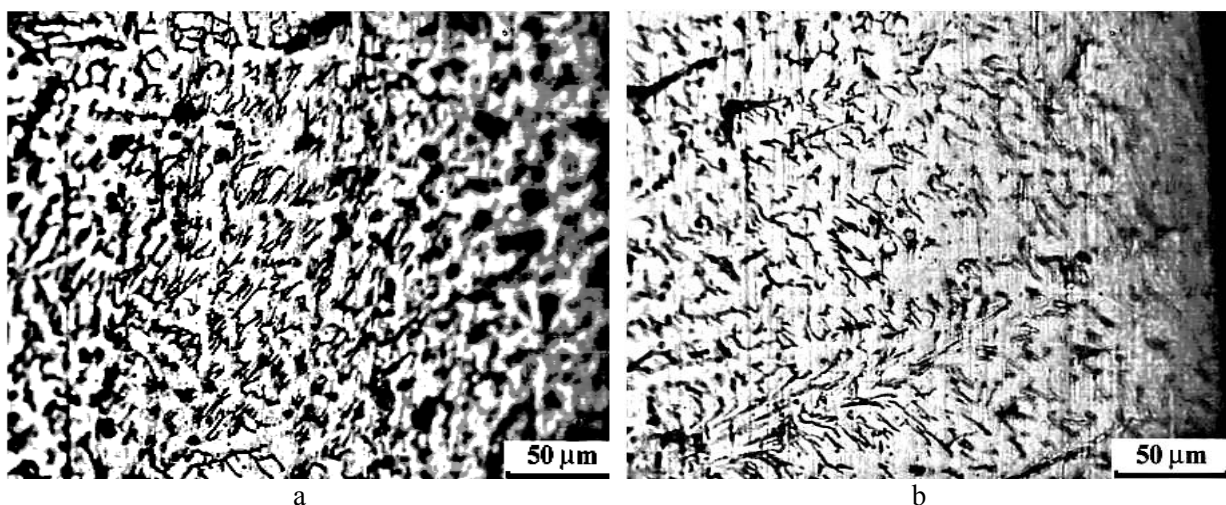


Figure 5 – Microstructure of the underlayer of polyhedral grains: a – sample №1 modified with nanostructured fibers of AlO(OH); b – sample №2 without the modifier

The microstructure of the next underlayer is characterized by relatively short, smaller, strongly ramified nonoriented dendrites. In sample №1 modified with nanostructured fibers of aluminium oxyhydroxide the given underlayer is more pronounced (Fig. 6 a).

The dendrites in the given layer are relatively small as their axes do not coincide to the heat flow direction and they stop growing.

Thickness of the given underlayer in sample № 1 modified with nanostructured fibers of aluminium oxyhydroxide is 34% of general one. The same underlayer in sample № 2 without the modifier is less pronounced. In sample № 2 without the modifier relatively short strongly ramified nonoriented dendrites form a continuous pattern (Fig. 6, b) and in sample № 1 modified with nanostructured fibers of aluminium oxyhydroxide we can observe areas of free surface where, at the same time, grain boundaries can be seen. Thickness of weekly oriented dendrites layer in sample № 2 is 30% of general surface.

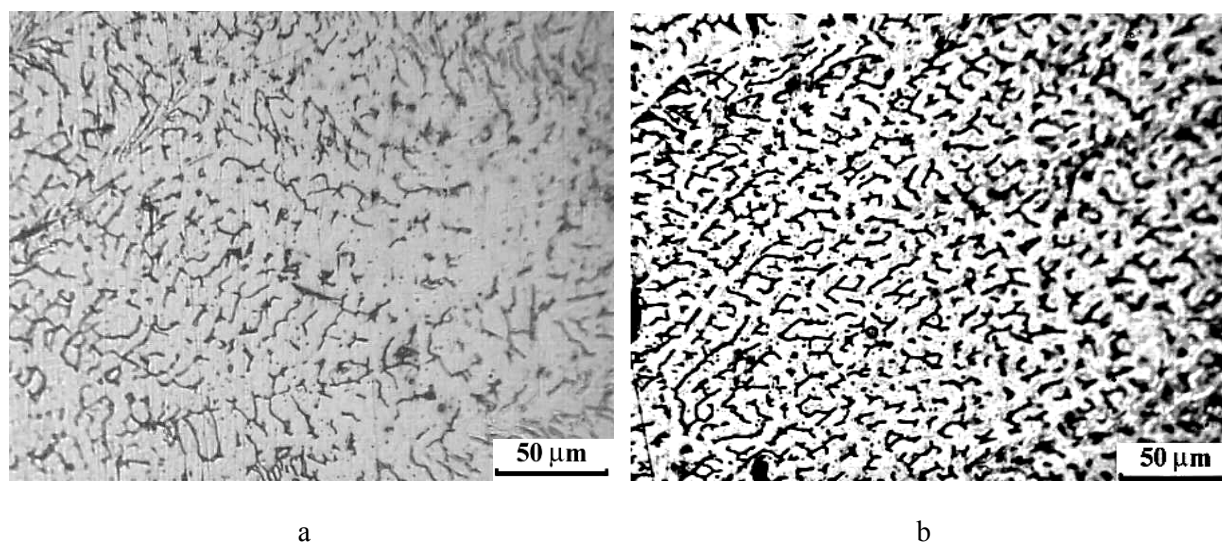


Figure 6 – The microstructure of the underlayer of nonoriented dendrites:
a – sample № 1 modified with nanostructured fibers of $AlO(OH)$;
b – sample № 2 without the modifier

The microstructure of the following underlayer is made up of oriented long dendrites (Fig. 7 a, b)

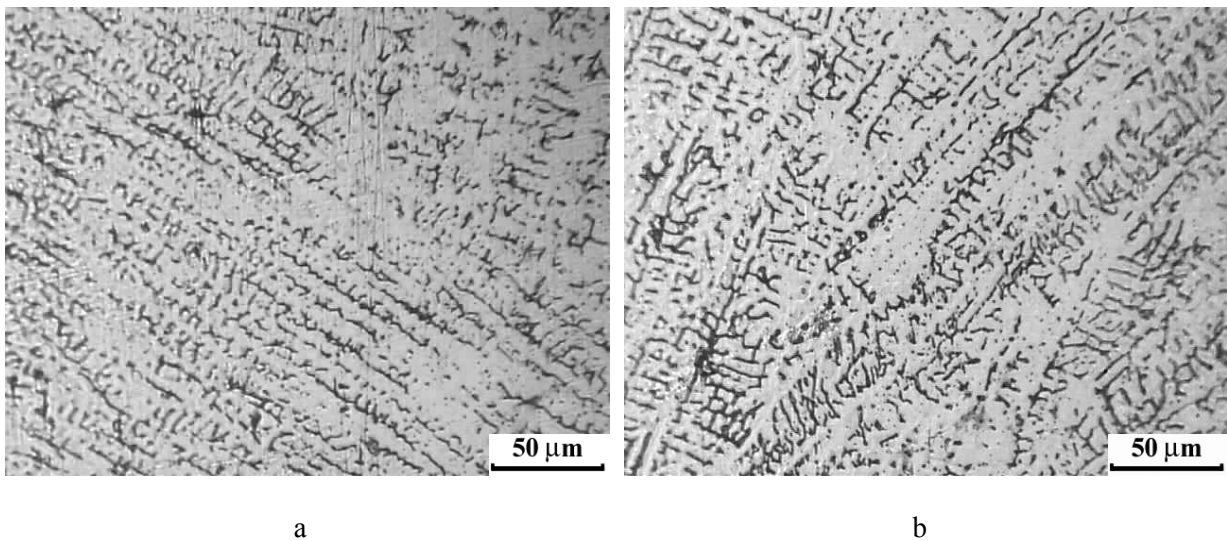


Figure 7 – The microstructure of the underlayer of oriented dendrites: a – sample № 1 modified with nanostructured fibers of $AlO(OH)$; b – sample № 2 without the modifier

Their orientation axis is normal towards the fusion border and is oriented along thermal current direction from the surface layer to the base metal. Strongly ramified thick dendrites are observed in sample № 2 without modifier (Fig. 7 b). Thickness of dendrites is 1.5-2 micrometer and their width (distance between the ends of opposite branches) is 20-25 micrometer. Thickness of dendrites in sample № 1 modified with nanostructured fibers of aluminium oxyhydroxide 0.8-1 micrometer and their width (distance between the ends of opposite branches) is 7-10 micrometer.

Orderly orientation of long dendrite axes is broken right before the fusion border and one more underlayer of weakly oriented dendrites is formed (Fig. 8).

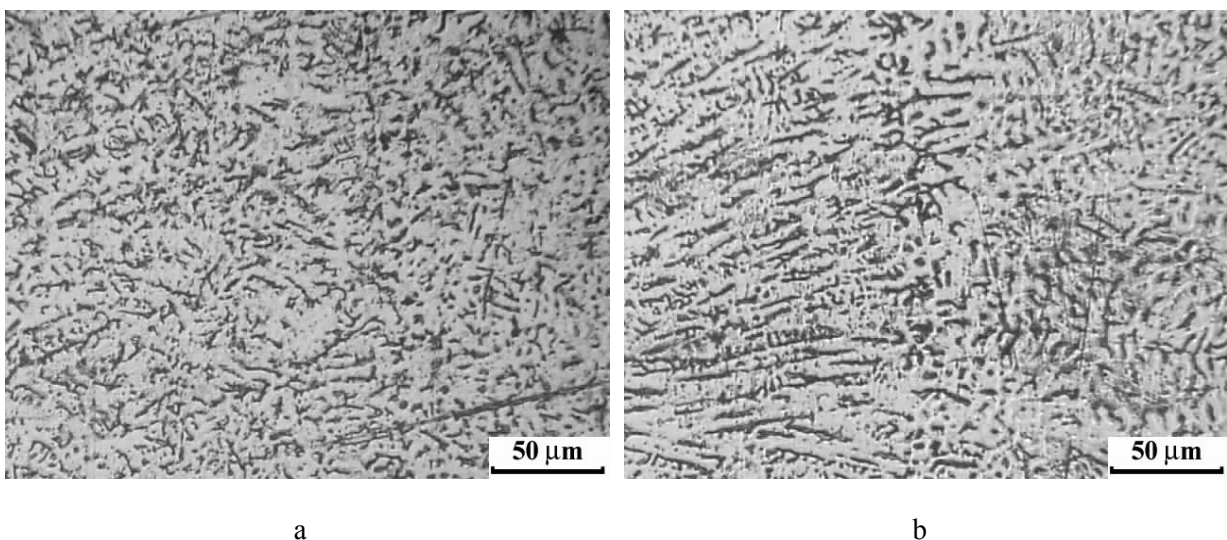
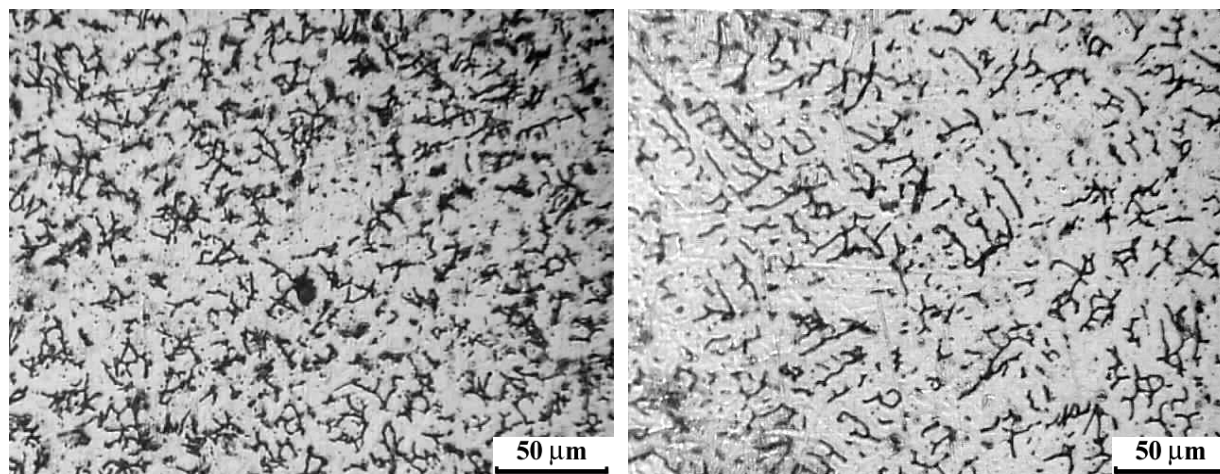


Figure 8 – Microstructure of the underlayer of weakly oriented dendrites:
A – sample № 1 modified with nanostructured fibers of $AlO(OH)$;
B – sample № 2 without the modifier

The given underlayer is weakest pronounced in sample № 2 № without the modifier (Fig. 8 b). Sample № 1 modified with nanostructured fibers of aluminium oxyhydroxide (Fig. 8 a) the border between oriented and nonoriented dendrites is about 25 micrometer thick.

The microstructure of the underlayer can be estimated as intermediate between areas A and B. In sample № 1 modified with nanostructured fibres of aluminium oxyhydroxide a clearly pronounced "grain" layer is observed again (Fig. 9 a).



a b
Figure 9 – Microstructure of the underlayer of nonoriented dendrites:
A – sample № 1 modified with nanostructured fibers of AlO(OH);
B – sample № 2 without the modifier

The situation in sample № 2 without the modifier is different (Fig. (b) as the structure of the underlayer is closest to the microstructure in B in terms of morphology. Here we mainly observe chaotically situated nonoriented dendrites with insignificant amount of polyhedral morphology grains. In sample № 1 modified with nanostructured fibres of aluminium oxyhydroxide the underlayer of grain structure was ≈ 4 mm and in sample № 2 without the modifier the layer of nonoriented dendrites was ≈ 3 mm.

All studied samples are rather perfect both in terms of their structure and quality of the surface layer. Application of nanostructured fibers of aluminium oxyhydroxide allows regulating the structure of the surface layer. It was established that the surface layer has stratified structure determined by changing the heat removal conditions from the surface inside the molten pool. Heat removal is weak near the free surface that is why crystallization results in forming polyhedral grain, dendrites do not have time to get formed. Most clearly this process is formed in the sample modified with nanostructured fibers of aluminium oxyhydroxide. In the rest part of surface layer typical dendrite crystallization took place. Approximately half of surface layer volume is taken by oriented dendrites. In the sample modified with nanostructured fibers of aluminium oxyhydroxide it is less than a half and in the sample produced without the modifier it exceeds half of surface layer volume. There is also the underlayer of nonoriented dendrites. This underlayer is especially wide in the sample modified with nanostructured fibers of aluminium oxyhydroxide. According to generally accepted view the more dendrite structure reveals itself, the larger are dendrites the worse are the service characteristics of the given layer [13, 14, 15]. From this point of view the surface layer produced without the modifier is worse than that of the sample modified with nanostructured fibers of aluminium oxyhydroxide. The microstructure of the surface layer modified with nanostructured fibres of aluminium oxyhydroxide is fine-grained and homogenous.

4. Conclusion

1. It has been established that application of nanostructured powders allows controlling the microconstituents of the surface layer.

2. Most clearly the layer of polyhedral grains is observed in the sample modified with nanostructured fibres of AlO(OH). The layer of oriented dendrites in the sample modified with nanostructured fibres of AlO(OH) occupies less than half of the built-up surface layer in terms of thickness. The layer of nonoriented dendrites is the widest in the sample modified with nanostructured fibres of AlO(OH).

3. In terms of dendrite size more equilibrium structure is produced when modifying the surface layer with nanostructured fibres of AlO(OH).

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