

## STUDYING NANOPOWDER MODIFIERS (NPM) EFFECT ON STRUCTURE AND PROPERTIES STEELS

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Preliminary Note – Prethodno priopćenje

The article investigated the macrostructure and microstructure of cast metal. Studies have shown that the introduction of nanopowder modifiers leads to a significant modifying effect, which has a positive effect on the structure of cast metal and is manifested in the grinding of both cast grain and dendritic structure. It was found that when the concentration of NPM in the metal in the form of particles of titanium nitride is 0,035 %, the nitride particles are distributed fairly uniformly in the metal volume, the cast metal structure is highly dispersed, and the mechanical properties of the steel significantly increase after thermomechanical processing

*Keywords:* steel, modifier, titanium carbonitride, mechanical properties, structure.

### INTRODUCTION

Recently, the issue of the nanopowder modifiers (NPMs) effect on the structure and properties of structural steel grades deserves more and more close attention of steelmakers. It is known that nanosized particles in dispersion-hardened alloys have a very positive effect on structural characteristics, as well as on mechanical and operational properties [1, 2]. The experience of using such particles is known from the practice of hardening heat-resistant nickel alloys [3], gray cast irons [4], aluminum alloys [5].

There are also known positive examples of using rare earth metals and alloys based on them as modifiers of various alloys [6-8].

However, the problem that remains unresolved is related to the features of introducing particles into a molten steel melt, physical and chemical characteristics of the introduced particles, and their stability in liquid and solidifying metal in the conditions of unbalanced crystallization [9-12].

In this work, for the purpose of experimental approbation of nanopowder modifiers effect on the structure and properties of structural steel grades in the Laboratory conditions in the vacuum induction furnace 10 melts of structural steel.

The following basic chemical composition were carried out (Table 1).

It used nanopowder modifiers ranging in size from 40 to 100 nm with a refractory component in the form of  $TiC_{0,5}N_{0,5}$ , TiN,  $TiN+Y_2O_3$ , clad with medium-carbon steel and chromium in the form of final granular spheri-

Table 1 **Chemical composition**

Element	Concentration / %
C	0,16 – 0,22
Si	0,22 – 0,24
Mn	0,10 – 0,25
P	0,006 – 0,008
S	0,002 – 0,004
Cr	0,06 – 0,10
Ni	0,17 – 0,21
Cu	0,12 – 0,16
Fe	Rest

cal particles of 60 - 100 microns in size. The calculated concentrations of the main substance ( $TiC_{0,5}N_{0,5}$ , TiN,  $TiN+Y_2O_3$ ) in the melt ranged from 0,025 % to 0,8 %. Melting, aging, and casting of liquid metal were carried out in vacuum of up to  $2 \cdot 10^{-3}$  mm Hg. Nanopowder modifiers packed in aluminum foil were introduced directly into the melt through a lock device. The temperature of the metal upon modification ranged from 1 545 °C to 1 615 °C. After introducing nanopowder modifiers, the metal was intensely mixed using the electromagnetic field of the inductor within 1,5 - 2,0 minutes. The temperature of the metal in the crucible before casting was 25 - 30 °C higher than the temperature of the input nanopowder modifiers. Metal casting was carried out in cast-iron molds of the cylindrical and rectangular shape weighing up to 10 kg.

For the purpose of hot physical modeling plastic deformation processes, the obtained steel ingots were heated in the furnace before rolling to the temperature of 1 150 °C, the exposure time in the furnace was 30 minutes. Plastic deformation of the ingots was carried out at the laboratory rolling mill in either three or five passes, depending on the final thickness of the finished product. At the end of rolling, the samples were placed in the furnace for delayed cooling of the rolled strips,

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the space temperature in the furnace when loading the samples was 550 °C, and the cooling time of the samples in the furnace was 60 minutes.

Tensile tests were carried out at the room temperature in accordance with GOST 1497 - 84 on a Zwick/Roell Z100 tensile testing machine with a longitudinal deformation sensor. Impact strength tests were carried out at - 20 °C in accordance with GOST 9454-78 on a Zwick/Roell RKP 450 top with the maximum pendulum impact energy of 300 J.

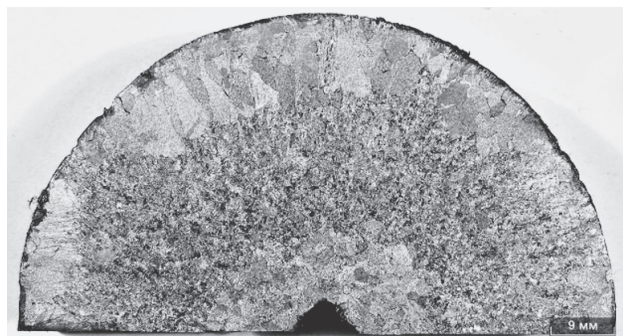
Metallographic studies to determine structural characteristics of metal samples, especially the distribution of nanometric particles in the metal matrix, were performed on previously prepared metallographic thin sections. Preparation of thin sections for studying was carried out using Buehler sample preparation equipment. The microstructure was studied using a Carl Zeiss Observer.D1m optical microscope with the magnification range 100-1000 times and the image analysis program Thixomet.PRO V3.0. The grain size was determined according to GOST 5639 - 82. To determine the chemical and phase composition of the structure components, we used a SUPRA 55V PWDS scanning electron microscope with an X-ray microanalysis accessory.

## EXPERIMENTAL STUDIES

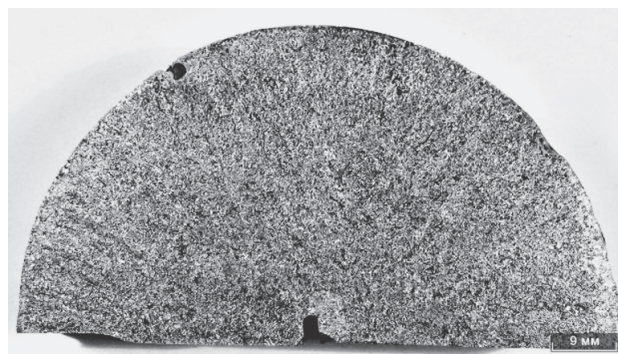
### Equipment and tools

When studying the effect of nanopowder modifiers on structural characteristics of structural steel grades, it was found that NPMs effectively affect the macro- and microstructure of metal. Figures 1 and 2 show the macrostructure of ingots without NPM and with NPM introduced. The macrostructure of the ingot obtained with introducing NPM (TiN) is characterized by a more dispersed and ground cast structure, which has a fairly uniform distribution of primary grains of the same size. Grinding the primary grain leads to the more uniform redistribution of impurities and segregating elements over the ingot cross - section, which will have a greater effect on the mechanical properties with increasing the size of the ingot.

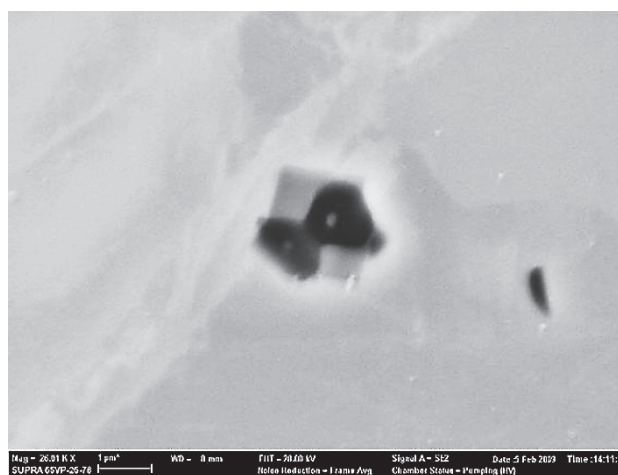
While the microstructure of ingots from the melts without NPM presents the fifth grade Widmanstätt fer-



**Figure 1** Macrostructure of the structural steel ingot without introducing NPM



**Figure 2** Macrostructure of the structural steel ingot after introducing NPM



**Figure 3** Titanium carbonitride inclusions that are formed on the corundum substrate

rite, the microstructure of ingots from the melts with introducing NPM has a fine-grained ferrite-pearlite structure with only a partially Widmanstätt character of the 1 – 1,5 size.

When analyzing the microstructure of the ingot with the introduced NPM, a large number of inclusions of titanium carbonitrides of various stoichiometric composition were found. As it is shown by studies performed on an electron microscope, the size of these particles grown on the oxide substrate from corundum is about 200 - 300 nm (Figure 3). These inclusions have an exogenous nature of formation and are formed in liquid steel as a result of recrystallization of the introduced NPMs through the liquid phase. Non-metallic inclusions are evenly distributed over the volume of the metal and can have a double effect on the grinding of the cast structure. On the one hand, they can be additional centers of crystallization, on the other hand, they can inhibit the growth of grains located at their boundaries.

## Discussion of the results

It is known from the results of previous studies that the range of particle sizes up to 0,01  $\mu\text{m}$  is the zone of the strengthening effect of inclusions of titanium carbonitrides [11]. That is, at sufficiently small sizes of inclusions and distances between them, these inclusions can

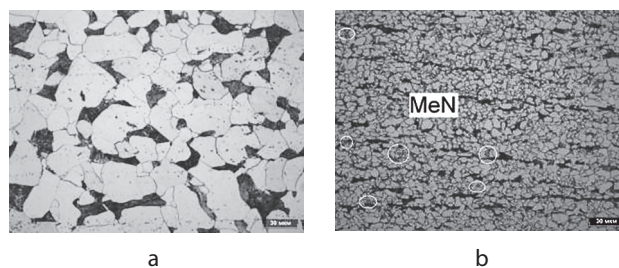
prevent the growth of dislocation rings upon deformation, reducing or leveling the development of microcracks. In addition, the distance between the particles depends mainly on the size of the particles, and not on their mass fraction in the melt.

The results of this experiment show that the degree of assimilation of titanium increases with increasing the exposure time of the metal in the liquid state, as well as with increasing the temperature of modification. However, in some cases, NPM particles, falling on the surface of a liquid metal, are slagged due to the high content of sorbed gas and float. In addition, a certain amount of particles will in any case dissolve in the melt. If the particles did not dissolve, then the content of titanium and nitrogen in the metal after smelting would increase in a stoichiometric ratio compared to its content in idle smelting. The chemical analysis data once again confirm the hypothesis of instability of the introduced NPMs. A more thorough cladding of particles or changing the methods of their adding (for example, as part of a wire) can solve this problem.

According to the results of mechanical tests of finished products samples from (Table 2), it can be concluded that steel with NPM has higher values of tensile strength ( $R_m$ ) and yield strength ( $R_e$ ), impact strength (KCV) with almost the same relative elongation compared with samples without NPM. At the maximum concentration of NPMs in the metal (0,8 %), the yield strength increase is  $\sim 100$  MPa, the tensile strength  $\sim 80$  MPa, the elongation decreases by 0,6 %. It should be noted that the highest set of properties is observed for samples of finished steel, in which the concentration of NPM in the form of titanium nitrides clad with chromium is 0,035 %. These samples have a higher complex of properties than samples with the maximum concentration of NPM (tensile strength and yield strength are 15 – 20 MPa higher, impact toughness is 30 J/cm<sup>2</sup> higher, and relative elongation is reduced by 2 %).

Studying the structure of the samples of hot-rolled sheet shows that introducing NPM in steel smelting has a significant effect on changing the grain size in the deformed metal (Figure 4). So, the grain size of rolled samples without NPM corresponds to 8 points (the average grain diameter  $d_{cp} = 20$   $\mu$ m), and the grain size in samples after NPM introducing corresponds to 12 points (the average grain diameter is 5,4  $\mu$ m).

In addition, in the microstructure of steel samples with introduced nanopowder modifiers there are present



**Figure 4** Metal sample microstructure after plastic deformation: a – without NPM, b – after NPM introducing

separate particles of the correct geometric shape in the form of triangles and rhombuses of pale orange color, with then average size of 1 to 2 microns. The X-ray microanalysis allows identifying these particles as titanium nitride. However, the distribution of such TiN particles over the sample area is rather uneven, is characterized by forming various clusters.

## CONCLUSION

The results of studying the macrostructure and microstructure of cast metal show that introducing nanopowder modifiers leads to a significant modifying effect, which positively affects the structure of the cast metal and is manifested in the grinding of both cast grain and dendrite structure. In contrast to the microstructure of ingots from the melts without NPM that is a type of the fifth grade ferrite, the microstructure of ingots from the melts with NPM introduced has a fine-grained ferrite-pearlite structure.

However, it was established that the modifying complexes used in this work were not sufficiently monodisperse and compactly prepared, the degree of their assimilation turned out to be small. The introduced particles turned out to be insufficiently stable under real conditions of metallurgical smelting. When introduced into steel, they almost completely dissolve and recrystallize through the liquid phase. To ensure chemical reactivity of the nanoparticles, the introduced granules should not be small, which will ensure their reliable entry into the melt. On small granules there is a large amount of sorbed oxygen that forms an oxide film on the surface of the melt, blocking the granule.

In order to avoid sintering during heating, each particle should be clad with metal. The preferred option is the introduction of particles in the form of ligatures, for

**Table 2 Results of mechanical testing finished products samples**

Nanopowder type	Nanopowder concentration, mass / %	$R_e$ / MPa	$R_m$ / MPa	A / %	KCV <sup>20</sup> / J/cm <sup>2</sup>
Without nanopowder	0,00	380	505	32,2	100,5
TiN clad with chromium	0,025	423	554	30,6	114,1
TiN clad with chromium	0,035	498	600	27,8	149,0
TiN+Y <sub>2</sub> O <sub>3</sub> clad with chromium	0,035	432	561	29,0	103,5
TiC <sub>0,5</sub> N <sub>0,5</sub> clad with steel	0,40	466	583	30,5	115,2
TiC <sub>0,5</sub> N <sub>0,5</sub> clad with steel	0,80	483	583	30,0	117,4



example, in the form of a wire filler using a pinch roll. It has been established that when the concentration of NPM in the metal in the form of particles of titanium nitride is 0,035 %, nitride particles are distributed fairly uniformly in the metal volume, the cast metal structure is highly dispersed, and mechanical properties of steel after thermal-and-mechanical treatment are significantly increased.

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**Note:** Responsible for the English language is Natalya Drak, Karaganda, Kazakhstan