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Investigation of Macro- and Microstructure of Experimental Composite Materials Obtained by Disperse Hardening Method

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Abstract

Metal-based constructional materials will always be demanded by the companies of mechanical engineering, space and other industries. The most interesting is studying the methods, based on introducing micro-, ultra- and nanodisperse particles in liquid or hard-liquid melt, that is disperse hardening. Such methods of obtaining composite materials are known for a considerable period of time, though their application has been limited; they have been employed in different industries, but not widely. The article describes different methods of obtaining composite materials based on introducing the disperse particles; also a new original method is suggested, affording obtaining composite materials with predictable characteristics different in various areas of one and the same manufactured item. The article also describes the process, the experimental results, the findings of the investigations of the experimental material structure and properties, obtained by disperse hardening method. Disperse particles of wolfram and titanium carbides were introduced into solidifying melt while casting steel on the centrifugal casting machine. Such method of introduction allowed obtaining composite cylinder-shaped billets with different content of the introduced particles across the section (due to different densities of the introduced particles and of the hardened metal). The obtained samples were prepared and investigated for macro- and microstructure alteration across the section. The result of the study is represented by the data on the introduced particles distribution across the sections of the obtained samples.

Keywords: Centrifugal Casting, Composite Materials, Hard Alloys, Macro- and Microstructure, Methods of Introducing the Disperse Particles, Wolfram and Titanium Carbides

1. Introduction

Mechanical engineering, space and atomic industry progress stipulated developing new constructional materials, possessing high mechanical characteristics, particularly, the composite materials.

Composite material is obtained by means of introducing into the basic material a specific quantity of another material, which is added to obtain some specific qualities. Such additives can be represented by filaments, fibers, flakes, disperse particles of the harder material. Composite material can consist of two, three and more components. The dimensions of the hardening additive particles can vary within a wide range – from hundredths of a micrometer (for powder fillers) to several millimeters (when the fiber fillers are applied). By way of varying the volumetric content of additives, it is possible to obtain composite materials with the required values of hardness, heat-resistance, modulus of elasticity, abrasion resistance, and also to create the compositions with the required magnetic, dielectric, radiation absorption and other specific characteristics[1].

The hardening effect of disperse particles on the metal is realized due to creating the reinforcing framework, which prevents the material from destruction. The highest degree of hardening, at the same time preserving viscosity, plasticity and constructive strength, is achieved, when the dispersity of the particles is quite high, and when they are

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incoherent with the matrix. Usually the modulus of rigidity of the particles is twice as high as the modulus of rigidity of the matrix. The dislocations do not cut and do not deform the particles, but bypass the incoherent elements, increasing the level of stress. The longer the distance between the disperse particles, the better the hardening[2].

As of today, two classes of metal - based composite materials could be identified on the ground of the content of disperse particles in the metal matrix: composite materials obtained by powder metallurgy methods (hard alloys) and disperse hardened materials.

Methods of powder metallurgy afford obtaining heterophase (hard) alloys by mixing and pressing the powder of metal and that of oxides, carbides etc[3]. In the beginning of the 20th century, 1923, in Germany a patent was registered for the method of baking the wolfram carbide powder, using up to 10% of cobalt powder as a cementing binder. In later patents the content of cobalt was increased up to 20%.

For the first time the baked hard alloy for cutting tools was obtained on the basis of wolfram monocarbide and cobalt powders in 1923–1925 by Osram, a German Company, under the patent of a German engineer Schroetter[4]. In 1926 the industrial production of these alloys started at Krupp Company. The alloy named "vidia" (from German "wie Diamant" – "like a diamond") proved to be the material, which fitted perfectly for instruments of different kind: metal cutters, drill bits, plates for milling cutters, saws, core drills and reamers. Thanks to the new composite material it became possible to process steel and iron by cutting; before that time all parts were done either by forging or founding. As in the due course of time the high-speed steel did, this material brought about a revolution in mechanical engineering.

Technological processes of manufacturing the existing grades of the powder hard alloys are mostly identical. But they can be principally different as regards the process conditions and the particular technological operations and techniques. The standard production process of the powder hard alloy includes a number of operations, the basic of which are as follows:

- obtaining powders of some kind of high-melting element and the carbides required for making the alloy;
- preparing the mixture of carbides with the powder of the high-melting element;
- pressing the shapes of this mixture followed by baking.

Quite often the standard process also includes finishing (diamond sharpening or processing) of the obtained baked pieces.[5-8]

Physical/mechanical and operational characteristics of the hard alloy depend predominantly on the chemical analysis, however, some alloy grades, having the same chemical analysis, are different in the carbide component grain sizes, which predetermines the difference in their physical/mechanical and operational characteristics. The characteristics of hard alloy grades are calculated in such a way, that the product mix could meet the requirements of the modern production process. Due to the fact that within the structure the high- heat carbides are present, the hard alloy tool possesses the high degree of hardness HRA 80-92 (HRC 73-76), heat-resistance (800…1000 °C), and therefore it can operate at the velocities several times exceeding the high-speed steel cutting velocity.[9, 10] It should be noted, that the hard alloys possess high indicators of wear-resistance. This results in the fact that now the active investigations are under way, involving the improvement of tribological characteristics of hard alloys by means of decreasing the dispersity of the powder down to nanodimensions[11-13].

However, possessing a number of advantages, the hard alloys are characterized by somewhat low strength (1000–1500 MPa), as well as by low indices of impact elasticity.

Even with the available wide range of the hard alloy grades their application is in different degree restricted due to the fact that the disperse phase is unevenly distributed in the matrix because of segregation formations, coagulation processes when sintering and pressing, and also because for their production the complex expensive equipment is required[14]. Moreover, the hard alloys are not practically feasible: due to their high hardness it is impossible to manufacture a one-shape tool (especially a big one), besides, they are not easily ground, with diamond tools only.

In view of the disadvantages, intrinsic to the hard alloys, the most interesting is such method of creating composite material, as disperse hardening. Disperse hardened materials are the metals or alloys, hardened by disperse particles of high-melt compounds, which do not solve and do not coagulate in the matrix (basis) of the alloy under high operating temperatures[15]. The finer the grains of the filler and the shorter the distances between them are, the harder the composite material is. Disperse particles of the filler make the material harder by maintaining resistance to dislocations movement under load, which impedes the plastic deformation[16]. High hardness is achieved, when the particles are distributed in the matrix either evenly or in a controllable way.

Four basic methods of introducing disperse particles in the metallic matrix could be identified:

- Introducing disperse powder in the ingot at the top-down casting stage.

In these experiments the metal was cast from one and the same ladle with a stopper into vertical chamotte moulds. To half of the ingots a mixture of powders SiO_2+C was fed, being introduced into the stream of the cast steel through a special chute, attached to the bottom of the ladle. Mechanical tests showed that the modified metal had improved hardness characteristics by 18% and plastic characteristics by 37-50%, the resistance to cracks and the impact elasticity improved 1.5-2 times[17].

This method of disperse hardening is sufficiently interesting and quite easy to realize, however, it should be noted, that when implementing this method there is no possibility to predict and to control the particle distribution.

- Introducing disperse particles at centrifugal electroslag casting

This technology means the process of electroslag melting of the electrode in a melting vessel, which accumulates liquid metal and slag in required amounts, and then taps them into a rotating mold[18]. The hardening particles are represented by synthetic fine disperse particles TiCN and Ti in the amount 0.3…0.5 % of the weight of the melt. The modifier was obtained by preliminary mixing the powder components and by pressing them cold in tablets with diameter of 25…30 mm and with thickness of 8…15 mm. The dimensions of the tablets were chosen taking into account the conditions of their solving in the modified melt within the period of 20…30 s. The obtained tablets were fed into the furnace 2 min before tapping.

Introducing the modifier 0.4 % (mass) in the metal alters the structure of the cast metal considerably. Also, the characteristics of this metal undergo changes. Particularly, the transcrystallization zones are eliminated in the round cast shapes, the sizes of dendrites become considerably smaller. Moreover, dendrites assume the preferable form throughout the volume of the solidified metal. In the structure of castings with introduced modifiers there is the ferrite-martensite matrix with compact carbides, which are mostly located in micro grains. The destruction of the samples at the impact is predominantly of transcrystalline character. The authors undertook no investigations of the mechanical characteristics.

This method has a considerable drawback: the hardening particles are fed into the furnace as pressed tablets. Therefore, it is impossible to predict how the disperse particles will be distributed throughout the volume of the metal. In addition, the interaction of particles with the melt "on the surface" of the "tablets" will occur for a considerably longer period of time than the interaction of the particles with the melt "inside" the "tablets". Even under condition, when no chemical interaction with the melt occurs, the particles will be affected by temperature, which will result in their partial dissociation.

- Introducing disperse particles in the metal at continuous casting

Prepared, rolled into wire, the particles were fed into the stream of steel while casting at the continuously casting machine by means of a portable wire-feeding machine; in one case the wire was fed into the mold, in another case it was fed into the tundish. The investigations of the obtained experimental metal showed that the density of the modified metal increased by 25-56 kg/m^3 (0.3-0.7 %). The mechanical tests on the disperse-hardened steel samples showed the improved mechanical characteristics:

- yield point, on average, by 7.2 %;
- ultimate breaking resistance – 1.0 %;
- ultimate stress limit – 1.7 %;
- tensile strain – 8.4 %;
- contraction ratio – 14.8 %.

Maximum improvement of the indicators was achieved at the basic substance concentration in metal of 0.015-0.025 %, which occurred when the powder wire was fed into the tundish of the casting machine[19]. This method of introduction does not secure a sufficiently even distribution of the introduced modifiers throughout the volume of the ingot both as a consequence of crystallization phenomena and as a result of segregation depending on the specific mass value. Due to this fact it is quite doubtful to assume that there is a possibility to predict the amount of particles to be introduced into the ingot.

That is, if the disadvantages of all existing methods of introducing disperse particles in the metal are properly analyzed, all of them could be expressed as the only one common disadvantage: it is impossible to control the process of the introduced particles distribution across the volume of the obtained samples, and thus, it is impossible to predict the mechanical characteristics of the obtained composite material.

2. Concept headings

The authors of this article assume that to solve the issue of the modifying particles predictable distribution throughout the entire volume of the casting the difference between density of the hardened metal and that of the introduced particles should used. This could be represented as follows: the particles, which density is higher than that of the hardened metal will move in the direction of the gravitational forces, and the particles which density is lower that that of the hardened metal will move in the direction of the melt surface. From this follows that in order to have the possibility to control the distribution of particles throughout the volume of the hardened metal, some forces should be applied to the dispersed particles. As such force the centrifugal force was chosen, affecting the solidifying melt and the introduced particles in centrifugal casting process. If the density of the high-melting disperse particles, introduced in the melt, is different from the density of the melt, then the force, affecting the particle, is not equalized by the centrifugal force and by the gravity force of their own. Consequently, the conditions occur for the particles' moving in this or that direction, i.e. to the inner or to the outer surface of the formed billet. When the particle gets in contact with the crystallization front it appears to be pressed by the melt to the crystallization front, and it has no longer the possibility to come to the surface, as it becomes captured by the growing dendrites[20].

Thus, the task of this study is carrying out the experiments to obtain composite material by introducing the particles with different density values, investigating the distribution of the disperse particles in composite material, investigating the possibility to distribute the particles across the sections of the obtained samples depending on their density.

As the charging materials for the experiments the soft iron of steel grade 10880 was used. The chemical analysis is shown in table 1.

Melting of the charge materials was implemented in the SELT-001-40/12-T melting furnace, at the generator maximum capacity operation. As the charged materials melted the remaining charge materials were added.

Upon melting, before casting the metal was deoxidized with Al, and then it was charged into the centrifugal casting machine (Figure 1a). While casting to obtain experimental samples No. 2 and 3 in the stream of the molten metal the dispersed particles were fed (Table 2) (Figure 1b). When obtaining the samples, before casting, carbon (C) and titanium sponge (FeTi) were fed on the surface of metal, to ensure wettability of the introduced particles and to secure their interaction with metal.

The obtained samples underwent cutting to perform the investigation of the macro- and microstructure (Figure 2). For cutting the samples from the obtained material, according to the given schemes, the Ergonomic 320.250 DGH contour band saw was used. At this machine-tool the overheating of the processed material is prevented to preserve the structure; good quality is achieved and precision of cutting is secured.

Table 1. Chemical analysis of steel grade 10880, %

C	Si	Mn	S	P	Cu
not more than 0.035	not more than 0.3	not more than 0.3	not more than 0.03	not more than 0.02	not more than 0.3

Figure 1a. General view of the centrifugal casting machine (front view; the unit is opened for maintenance and repair).

Table 2. Content of the introduced particles in the samples

Sample No.	Mass of the introduced carbides, % of the billet weigh	
	WC (density 15,6 (g/cm^3))	TiC (density 4,93 (g/cm^3))
1	0	0
2	0.5	0.5
3	1	0

Figure 1b. Feeding the disperse particles at obtaining samples No.2 and No.3.

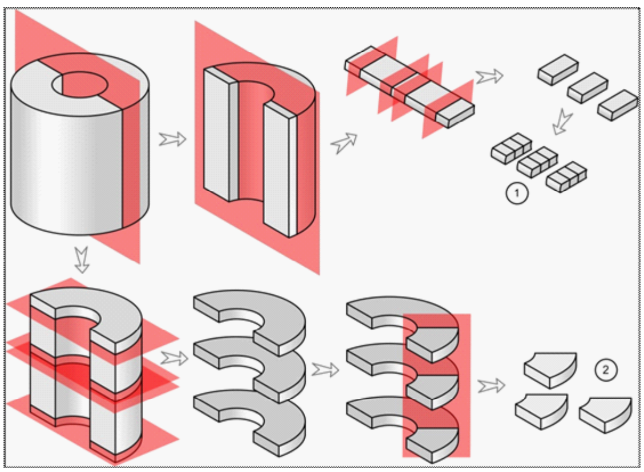


Figure 2. Schematic of cutting off the samples for the investigations: 1 – samples for microstructure investigation; 2 – samples for macrostructure and hardness alteration investigation.

The samples for investigating macro- and microstructure of the obtained samples were prepared as follows:

Inserting the samples in the tar with the help of Simplimet 1000 automated fitting press manufactured by BUEHLER;

Grinding and polishing at the EcoMet 250 / 300 grinding machine tool, equipped with the AutoMet 250 / 300semi-automated head.

To estimate the alteration of the length of the crystalline areas, the samples of the obtained metal were pickled, and then were scanned with Epson scanner at high resolution. The investigation of microstructure was implemented with the help of the C. Zeizz Observer.D1m inverted microscope equipped with the Thixsomet.PRO image analyzing complex; also the JEOL JSM – 6460 LV electronic microscope was applied.

3. Discussion

As a result of the experiment three cylinder-shaped samples of the composite material with different introduced carbide content were obtained. The surfaces of all samples were of satisfactory quality; no surface defects (delamination, cracks) were present.

The investigations of the cast metal macrostructure showed that in sample No. 1, after the initial solidification area, at the wall of the mold, the dendrite structure is clearly observed, and is located perpendicularly to the heat withdrawal direction (Figure 3a). In the samples with the introduced carbides the dendrite area is not found at all, which is stipulated by higher velocity of metal solidification. Sample No.2 with the introduced wolfram and titanium particles has non-equilibrium structure: on both sides (the outer and the inner sides) a finer structure, as compared to that in the middle of the billet, is clearly observed (Figure 3b). Sample No.3 containing only particles of wolfram carbide possesses more unified structure over all the section area (Figure 3c).

Investigations of the cast metal with optical microscope show that in sample No.1 the particles of iron and silicon oxides are observed, which is explained by the fact that

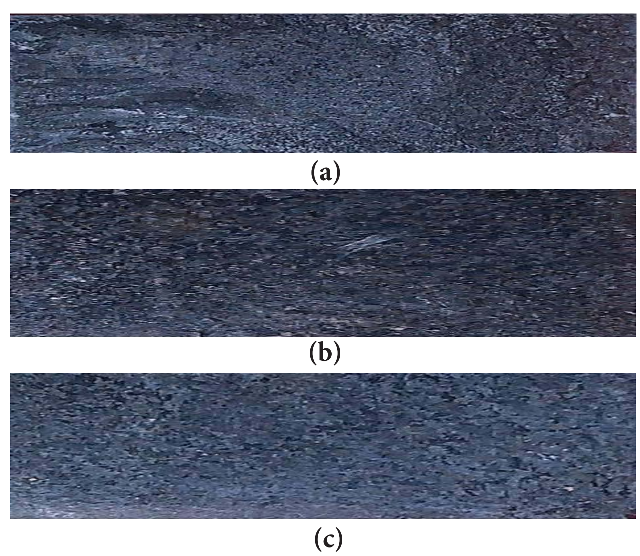
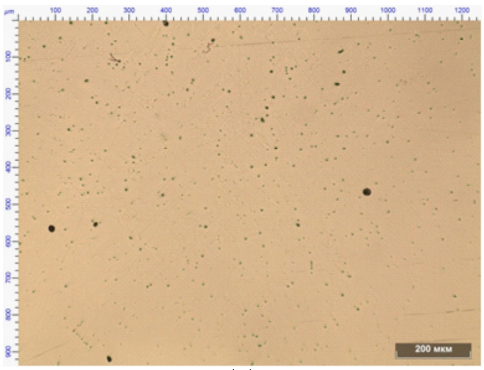


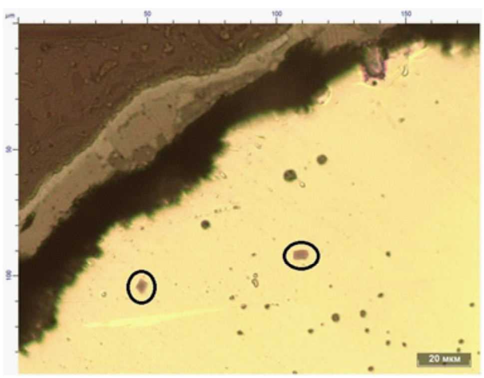
Figure 3. Investigating macrostructures of the obtained samples: a – sample No.1, (×6); b – sample No.2, (×6); c – sample No.3, (×6).

the metal was oxidized during casting and the oxides were formed (Figure 4a). Investigation of the microstructure of sample No.2 shows that at the outer edge of the sample there are clearly distinguishable separate grains of titanium carbonitride (Figure 4b). At the inner side of sample No.2 there are quite evenly located aggregated groups of inclusions, consisting of titanium carbide and carbonitride (Figure 4c). The presence in the microstructure of both carbides and carbonitrides is explained by the reaction of the introduced particles with nitrogen present in the air or with the nitrogen solved in the metal.

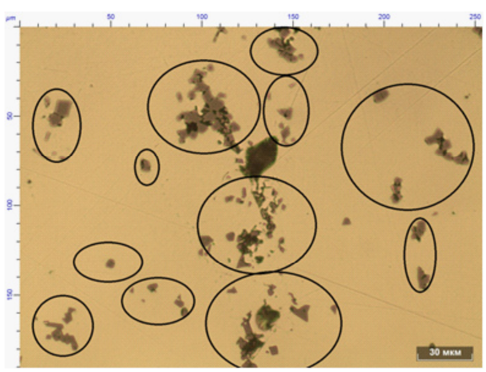
Investigations of the outer edges of samples No.2 and No.3 with optical microscope show, that in these areas there are aggregations of fine inclusions, determining the morphology of which is impossible due to the zoom limitations of the microscope. To determine the morphology of these particles the investigations are carried out at the electronic microscope, and they show that these inclusions are the introduced wolfram carbides (Figure 5a). The occurrence of wolfram carbide locations at the outer edge of samples No.2 and No.3 is not even, which is explained by different amount of the introduced particles (Figure 5b). The particle size analysis shows that the introduced carbide became considerably smaller: the initial size at introduction was 4-9 micrometers, and in the samples the size of the particles is 1 micrometer and less.



(a)

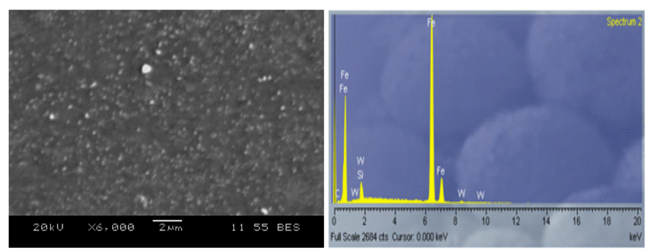


(b)

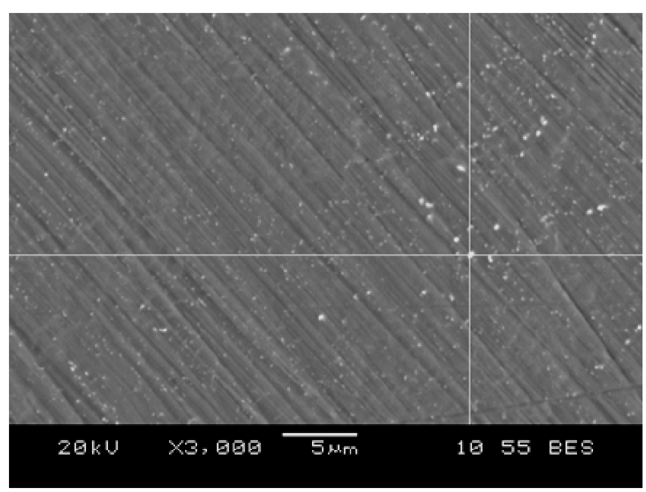


(c)

Figure 4. Investigations of the microstructures of the samples, implemented with the optical microscope: **a** – oxide inclusions in sample No.1, (×1250); **b** – titanium carbonitride at the exterior of sample No.2, (×1250); **c** – aggregation groups of titanium carbide and carbonitride, located at the inner side of sample No.2, (×650).



(a)



(b)

Figure 5. Wolfram carbide at the outer edge: **a** – sample No.3, (×6000); **b** – sample No.2, (×3000).

4. Conclusion

Thus, on accomplishing the study a conclusion can be made that the distribution of particles takes place according to the suggested method: the particles with the density lower than that of the metal are located at the inner side of a centrifugally cast billet, and those with a higher density are located at the outer side. It was experimentally discovered that increasing the amount of the introduced disperse particles of wolfram carbide results in their increased concentration in the surface layers. It was illustrated that the introduced particles come into reaction with the solidifying melt: titanium carbides, as a result of their interaction are partially transformed in carbonitride, and the size of wolfram carbides becomes smaller.

A specific feature of the suggested method of obtaining composite materials is that it is universal as regards its application by the enterprises specialized in manufacturing metal products, parts for machinery and mechanisms. This is stipulated by the fact that it can be relatively easily integrated in the existing technological operations. The variety of the disperse particles, of their physical/chemical and mechanical characteristics, together with the possibility to vary the velocity of the horizontal mold rotation, create the prerequisites for obtaining new disperse-hardened materials with such characteristics which are required for each particular application.

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