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DEVELOPMENT OF QUALITY CONTROL AND STRUCTURE PARAMETERS DETERMINATION METHODS FOR LARGE SIZE PRODUCTS FROM SINTERED HARD ALLOYS WC-(Co+Ni+Cr) BASED ON ANALYSIS OF THE ULTRASONIC OSCILLATIONS SPREADING PARAMETERS

The object of research is hard alloys with a morphology of the carbide phase skeleton structure, in which particles contact with each other, and the gaps between them are filled with a binder phase. The mechanical and service characteristics of such materials depend on the degree of development of the skeleton structure.

One of the most problematic areas is the lack of non-destructive methods for determining the parameters of the structure. The introduction of such techniques will allow obtaining objective information on the structure of the material and using it to evaluate the quality of products. In the course of the study, the parameters of the scattering of elastic vibrations in inhomogeneous media were determined. The main hypothesis of the study is the assumption that the processes of energy dissipation occur both in the structural elements themselves (carbide grains and bond areas) and at their boundaries. Therefore, the evaluation of dissipation processes will allow obtaining a quantitative estimation of the alloys structure parameters, and will allow assessing the quality of the material. The following characteristics were chosen as the parameters characterizing the propagation of ultrasonic oscillations: the speed of the oscillations propagation, the scattering background level in relation to the amplitude of the bottom reflection, the oscillations attenuation coefficient. The parameters were determined and compared with the characteristics of the quality of the products and the parameters of the microstructure, which were determined by the methods of quantitative metallography and the statistical characteristics of the relationship between the parameters, were determined.

As a result, new quality control procedures for carbide products have been developed. The contiguity characteristics of the carbide skeleton of the sintered cemented carbide were determined by measuring the propagation speed of ultrasonic oscillations. The assessment of the level of porosity with a pore size of less than 1 mm was carried out according to the results of measuring the relative amplitude of the background scattering of ultrasonic oscillations.

The proposed methods are non-destructive and are carried out in one cycle with ultrasonic flaw detection, to which 100 % of the products are subjected. These techniques have been introduced in the production of carbide rolls by the method of controlled hot vacuum pressing. They have become an integral part of the quality control system for carbide rolls.

Keywords: *hard alloys, carbide grains, binding areas, skeleton structure, heterogeneous materials, ultrasonic oscillations, energy absorption, oscillations attenuation, porosity, particle conglomerates.*

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1. Introduction

Hard alloys are materials with a pronounced heterogeneity of structure. The process of wear of such materials depends on the degree of heterogeneity of the structure and specific operating conditions. The analysis of the main mechanisms of wear of hard alloys is given, in particular, in [1–3]. In [4–6] the influence of the processed material

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on the features of wear mechanisms is considered. This approach is legitimate when it comes to choosing material from existing ones. But when developing new materials, it is more important to study the influence of internal factors (structure parameters) of the material on the characteristics of wear, which will identify ways to improve the performance of the tool.

The most common approach is based on finding the relationship between the grain size of the carbide phase and the characteristics of the alloys. This takes into account both the influence of the initial particle size distribution of the mixture [7] and the influence of sintering conditions on the change in particle size distribution [8, 9].

However, it was found that in addition to the characteristics of the carbide phase, the properties of the alloy are greatly influenced by the parameters of the spatial distribution of carbide particles in the structure of the alloy [10, 11]. Hard alloys have a carbide phase sceleton morphology in which the particles contact with each other and the gaps between them are filled with a binding phase. Mechanical and service characteristics of such materials depend on the degree of the sceleton structure development. One of the most problematic places is the lack of non-destructive methods for determining the parameters of the structure. Therefore, the development of such techniques is important.

The main hypothesis of the study is the assumption that the processes of energy dissipation occur both in the structural elements (carbide grains and bonds) and at their boundaries. Therefore, the evaluation of dissipation processes will allow to obtain a quantitative measurement of the structure parameters of alloys, which will assess the quality of the material. The development of wear and destruction processes of materials at the macro level will be determined at the microstructural level by the processes of absorption and accumulation of energy as a result of external actions.

The object of research are hard alloys, which have a sceleton morphology of the carbide phase, i. e. the particles are in contact with each other, and the gaps between them are filled with a binding phase. Therefore, the processes of energy dissipation occur both in the structural elements (carbide grains and bonds) and at their boundaries.

The aim of research is to establish the relationship between the structure of hard alloys with the characteristics of the ultrasonic oscillattions spreading and mechanical properties. It permits to develop new methods of large-size carbide products quality control.

2. Methods of research

The method of ultrasonic flaw detection was used as the main one. Ultrasonic product quality control (USC) is widely used as a method of non-destructive flow control due to its high accuracy, informativeness, relative simplicity of the implementation procedure. With its help it is possible to control 100 % of the products. However, in technological practice, the main controlled parameter is the presence of reflections of ultrasonic (US) waves, which are interpreted as evidence of inhomogeneities and inconsistencies in the macrostructure of the material.

Given that the frequency range of ultrasound is 1–10 MHz, and the range of speeds of ultrasonic waves spreading is 1000–10000 m/s, the size of the detected defects may be at the level of 10^{-2}–10^{-3} m. Such defects are classified, as a rule, as macrodefects. To assess the microstructure of a material with a characteristic size of inhomogeneity less than 10^{-3} m, ultrasound techniques are usually not used. The study of the parameters of absorption and scattering of ultrasonic oscillations in determining the characteristics of the internal friction of materials has become widespread. However, these techniques are quite time consuming and allow evaluating the fine structure of the material (dislocation structure, the presence of impurities and atmospheres at the dislocations, and so on).

At the same time, the implementation of the processes of absorption, reflection and scattering of energy at the boundaries of inhomogeneous elements of the structure can affect the overall energy balance of the process of propagation of elastic oscillations.

Thus, the study of the relationship between the structural features of the material with the peculiarities of the propagation of elastic oscillations will increase the informativeness of the ultrasound procedure and obtain additional data concerning the structure of the material.

Carbide products, obtained by vacuum sintering, vacuum hot pressing, hot isostatic pressing were used as the object of study. The products were made of hard alloys based on tungsten carbide with a content of 70–90 % by weight. As a bond used cobalt, alloy 50 % cobalt+50 % nickel, alloy 45 % cobalt+45 % nickel+10 % chromium. The sizes of hard-alloy products were in the range: diameter – from 150 to 390 mm; product thickness – from 62 to 120 mm. These products were used as disk-type rolling rolls for high-speed wire rolling mills and as wear-resistant bandages for rolling rolls of rolling mills for the production of periodic reinforcing profiles [12]. The general view of disk rolls is given in Fig. 1, details of bandaged rolls and rolling rolls with bandages assembled are shown in Fig. 2.

Fig. 1. Carbide rolling disc-type rolls in the state of delivery to the consumer and prepared for operation with grooves

Fig. 2. Roller of small-grade rolling mill for rolling of a reinforcing profile with hard-alloy bandages (a bandage ring in a state of delivery, a bandage ring with the cuted grooves, a steel axis of a roll, the bandaged roll assembled)

As parameters that characterize the spreading of ultrasonic oscillations, the following characteristics were selected:

1. The speed of propagation of ultrasonic oscillations. It was measured using a direct combined emitter at a frequency of 5 MHz by fixing the first and second reflected impulses from the bottom of product with plane-parallel surfaces. The method of calibration of the calculated speed according to the known linear size of the product (propagation path length) was used. The total relative error of the device during measurements did not exceed 0.35 % of the measured value, which is in absolute units 22–24 m/s.

2. The scattering background level in relation to the amplitude of the bottom pulse. The presence of a digital signal-processing path in modern devices allows to obtain an objective assessment of the ratio of the amplitudes of different signals.

3. The attenuation coefficient of ultrasonic oscillations. Hard alloys have a low attenuation coefficient. In them, in addition to the first reflection from the bottom surface of the product, reflections of the second and higher orders can be observed. It was assumed that the amplitude of the attenuating reflections decreases exponentially and can be given by the formula:

$$A_n = A_o e^{-Q_n}, \quad (1)$$

where A_n is the amplitude of the pulse with the order of reflection n; A_o – preexponential coefficient; Q – attenuation coefficient; n is the order of reflection.

The amount of attenuation depends not only on the properties of the medium but also on the length of the signal propagation path as a characteristic of the material. Therefore, in addition to the eigenvalue of Q, Q_{giv} was defined as the ratio of Q to the thickness of the product h. This allows to compare the values of the attenuation factor for products of different thicknesses.

Using these techniques, a comparison of the characteristics of ultrasoic, defined in the standard quality control with the data of detailed studies. The analysis was performed on rolls made by the method of hot vacuum pressing (technology 1), controlled hot vacuum pressing (technology 2) and obtained by the method of vacuum sintering (technology 3).

All technologies were implemented in the conditions of Donix Company (Ukraine) on its own production base. To implement the technology 3, industrial sintering furnaces in a protective atmosphere of pre-formed semi-finished products were used. To implement technologies 1 and 2, Donix specialists developed a design and organized the production of equipment for the process of hot vacuum pressing, which allows to obtain products weighing up to 150 kg, up to 450 mm in diameter and up to 200 mm high.

The design of the hot vacuum pressing installation provides the possibility of automated change of the process parameters according to the set program. The temperature is regulated in the range of 100–1500 °C with an accuracy of ±3 °C. The pressure is regulated in the range of 0–5000 N/cm^2 with an accuracy of ±100 N/cm^2. The vacuum in the working chamber is maintained at a level not worse than 10^{-3} mm Hg. The accuracy of registration of the linear movement of the punch of the press is ±0.005 mm, which allows to control the process based on the control of the kinetics of shrinkage of the hard alloy during pressing. Necessary technical and economic parameters of the pressing process are achieved using durable three-dimensional resistance heater, reusable equipment with replaceable liners, closed cooling system of the working chamber, high-efficiency thyristor power regulator with equalization of power distribution to phases. The specially designed system of hot unloading of the pressed preparation increases efficiency of the equipment and allows operating installation in a continuous mode. The rational mechanical design of installation of hot pressing simplifies installation, repair, and routine works.

To simplify the analysis of the results, the quality of the macrostructure was evaluated on a conditional 5-point scale, where:

– score 5 corresponds to a homogeneous macrostructure;
– score 4 – structure, in which there are single weak reflections from defects;
– score 3 – structures with multiple weak reflections;
– score 2 – structures with large amplitude reflections on the background of multiple weak reflections;
– score 1 corresponds to the continuous defeat of the product by defects.

3. Research results and discussion

Analysis of the data set on the absolute values of the parameters of the ultrasonic wave's propagation and the scatter of their values showed that both the absolute values of the characteristics and the magnitude of the scatter of properties differ significantly in different manufacturing technologies. The difference between the rolls made by technologies 1 and 2 is much smaller (at the level of error of primary measurements) than between them and the rolls made by technology 3. However, it is noteworthy that the average values of the speed of ultrasonic waves are reduced, and the value the attenuation coefficient increases as the sintering conditions approach equilibrium. Since the chemical composition and particle size distribution of the raw material was the same for all three types of products, it was assumed that the difference in acoustic characteristics is due to the difference in the final structure of the material.

At the same time, the relationship between the characteristics of the spread of ultrasonis oscillations (USO) with a generalized assessment of the quality of the roll is not so clear (Table 1).

Table 1

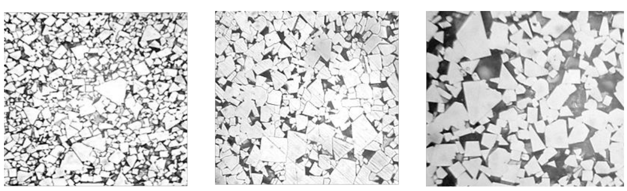
Ultrasonic propagation parameters in rolls with different macrostructure

Quality score	Technology 1		Technology 2		Technology 3	
	USO speed, m/s	Attenuation coefficient, mm^{-1}	USO speed, m/s	Attenuation coefficient, mm^{-1}	USO speed, m/s	Attenuation coefficient, mm^{-1}
5	6470	0.0176	6434	0.0190	6308	0.0241
4	6420	0.0175	6438	0.0196	6330	0.0246
3	6435	0.0194	–	–	6316	0.0248
2	6422	0.0199	–	–	6361	0.0253
1	6402	0.0195	–	–	–	–

As can be seen from Table 1, for the technology of hot pressing (technology 1) it is possible to talk about the presence of a certain relationship between the value of the attenuation coefficient and the quality of the product evaluated by the score. The higher the quality (higher the score), the lower the attenuation coefficient of the material, and the differences between quality rolls (score 5–4) and products of unsatisfactory quality (score 2–1) exceed the measurement error and are objective. At the same time, for rolls of type 3 such dependence also exists, but is expressed weaker that can be explained by inhomogeneity of sampling and big scatter of values of the investigated parameters for rolls with unsatisfactory quality. The value of the rate of propagation of USO is weakly correlated with quantitative assessment of quality.

From the obtained results, it follows that in the investigated materials the value of the attenuation coefficient can be used to assess the quality of the structure of the material made using a particular technology.

The same parameter, as well as the speed of propagation of USO change significantly when changing the technology of a product manufacturing from the same raw materials. Therefore, studies of quantitative characteristics of the structure of materials obtained by different technologies were performed. Typical structures of the studied materials are shown in Fig. 3.



a *b* *c*

Fig. 3. Carbide structures with a bond content of 15 %, ×2000×0.5: *a* – Technology 1; *b* – Technology 2; *c* – Technology 3

As can be seen from Fig. 3, the structures of the material manufactured by different technologies differ both in grain size and in the shape of carbide particles and the degree of their contiguity and cohesion. It was found that the attenuation coefficient increases, and the rate of propagation of USO decreases with a decrease in such characteristics of the structure as contiguity, specific number of contacts, specific contact surface. Thus, it can be assumed that both the attenuation coefficient and the rate of propagation of USO in the structures of the studied type are structurally sensitive factors. To explain this relationship, let's use the following considerations.

It is obvious that in the case of forming a fully connected sceleton structure, in which the speed of the USO in the carbide phase forming the sceleton is higher than in the second phase, the speed V_{sum} will be equal to the speed V_{carb}. If the sceleton is imperfect, then the speed V_{sum} will decrease, because part of the oscillation path will propagate in the second phase (binder) with a lower USO speed. The most general characteristic of the continuity of the sceleton is the contiguity of the structure *Sm*, which is invariant to the shape of the sections of the phases forming the structure.

Therefore, let's introduce the assumption that there is a relationship:

$$V_{sum} = V_{carb} Sm + V_{bind}\left(1 - Sm\right). \quad (2)$$

Substituting the values of V_{carb} and V_{bind}, as well as the values of *Sm*, determined by the methods of [12], in (2) let's obtain the following values of V_{sum} for alloys made by different technologies:

– technology 1 – V_{sum}=6425 m/s;
– technology 2 – V_{sum}=6395 m/s;
– technology 3 – V_{sum}=6304 m/s.

The calculated values coincide quite well with those found experimentally. Thus, equation (2) allows to take into account the influence of the structure features on the speed of propagation of elastic oscillations. It also takes into account in implicit form and the composition of the material, because based on the ideas developed earlier, the magnitude of the contiguity of the structure *Sm* depends on the volume fraction of the phases.

From the obtained data it follows, that equation (2) can be used for experimental determination of the value of *Sm* in the process of ultrasonic control from the ratio:

$$Sm = \left(V_{sum} - V_{bind}\right) / \left(V_{carb} - V_{bind}\right). \quad (3)$$

Equation (3) allows to calculate the value of *Sm* based on other physical quantities than those used in the methods of quantitative metallography. Because this technique is simpler than the method of quantitative metallography, it can be recommended as an express method to assess the structure of the material.

The relationship between the attenuation coefficient and the structure parameters is more complex than for the USO speed, because it was previously shown that it depends on both the characteristics of the microstructure and the characteristics of the macrostructure. However, the order of magnitude of this characteristic change within each sample is less than the difference between the mean values between the samples. Therefore, the presence of a correlation between the average values of the attenuation coefficient and the quantitative characteristics of the microstructure was investigated.

The least squares method shows that the closest relationship when using a linear approximation exists between the value of the attenuation coefficient and the average number of particle contacts. The correlation coefficient r=0.998. For the dependence on the average number of contacts determined by the method [12], it is r=0.971, for the dependence on the contiguity r=0.976. When using a nonlinear approximation, the correlation coefficient may be higher, but the resulting dependencies are formal and cannot be interpreted from physical assumptions.

The results of statistical analysis are given in Table 2.

Thus, it is established that there is a correlation between the stereological characteristics of the structure and the parameters of USO propagation, which was used to develop methods for indirect and express determination of the characteristics of the microstructure during ultrasound control [12].

The resolution of the ultrasonic control method is determined by many factors, but the minimum size of the detected defect cannot exceed 1/4 of the ultrasonic wavelength. For most metallic materials in the frequency range used in the control, this value is 0.5–2 mm (i. e. about 10^{-3} m). That is, important structural elements of real materials (pores, non-metallic inclusions, clusters of particles of one of the phases, large areas of the binding phase, etc.) are not detected by traditional ultrasonic control. To detect them, it is necessary to use destructive metallographic methods on samples cutted from products. These methods are quite time consuming and do not allow 100 % product control. At the same time, the processes of scattering, absorption and reflection of the energy of elastic oscillations must occur in each case of interaction of the energy flow with areas of physical inhomogeneity. Therefore, in this section the problem of finding the parameter of propagation of elastic waves that responds to the presence or absence of defects in the material with a size of the order of 10^{-3}–10^{-4} m was solved.

A characteristic feature of the spread of USO in the study of hard alloy products is the presence of a certain scattering background. This is expressed in the appearance of chaotic noise on the indicator of the device. It is known from the literature that their appearance can be associated with their own noise of the amplifying path, as well as with the processes of reflection and scattering of USO on various inconsistencies of the environment. In this case, the amplitude of the background signal depends on the structure of the material, because with the same settings of the device, it varied from product to product and even within the same product in its different areas.

With a given gain and the use of a specific ultrasonic transducer, the amplitude of the background signal can be considered independent of the hardware settings. In this case, by statistical processing of the measurement results, it is possible to establish some average amplitude, which can be considered a characteristic of this type of material. For example, for rolls made by technologies 1 and 2 at a gain of 35 dB and the use of the converter PPP 111–5–003 (operating frequency 5 MHz), the average background amplitude is 0.3–0.4 large division of the scale of the flaw detector, and for rolls of technology 3 this amplitude is 0.8–1.2 divisions. Therefore, when detecting higher values of the scattering amplitude, it is possible to talk about the difference in the structure of the material.

Table 2

Statistical relationship of attenuation coefficient with quantitative characteristics of microstructure

Technology of manufacturing	1	2	3	Correlation coefficient
Volume fracture of carbides	0.735	0.749	0.742	0.090784
Contiguity of structure	0.544	0.526	0.473	–0.978
Average number of contacts per 1 carbide particle	1.56	1.53	1.34	–0.998
Average number of contacts per 1 carbide particle, calculated according [12]	1.46	1.41	1.27	–0.971
Specific surface area of the contacts between carbide particles (mm^2/mm^3)	2.4	1.47	0.79	–0.86677
Specific surface area of the contacts between carbide particles and binder (mm^2/mm^3)	4.039	2.64	1.76	–0.84543
Attenuation coefficient	0.0186	0.0192	0.0246	–

The reason for the background scattering can be the boundaries of crystallites, pores and non-metallic inclusions, particles of the second phase. However, in any case, their characteristic size L should be 0.25λ–1λ (where λ is the wavelength). For an operating frequency of 5 MHz and an average sound speed of 6300–6600 m/s, this size is 1.3–0.4 mm. As mentioned above, the particle size of the carbide phase in the studied alloys is in the range of 0.001–0.015 mm, which is much smaller than the specified range. However, the study of the microstructure of sintered alloys in some samples revealed conglomerates of carbide particles, the size of which approached the specified limits (Fig. 4). The formation of such areas is more typical fort the products, made by technology 1. At the same time, when using vacuum sintering technology 3 sometimes there is increased porosity (as large pores that can give localized reflections, and small scattered porosity with individual pore sizes less than 1 mm.

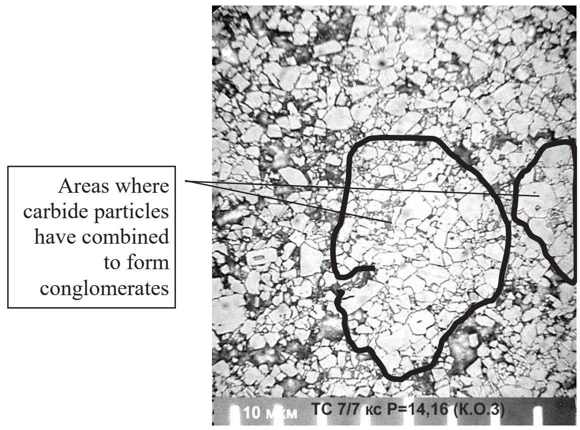


Fig. 4. Conglomerates in the structure of the sintered alloy with 15 % bond

As follows from the ideas considered earlier, the difference in the acoustic resistance of pores and conglomerates is very significant, and the reflection of energy at the boundary «material – pore» is much more intensive than at the boundary of conglomerates. To test this assumption, studies of the integrated background intensity of products manufactured according to different technological schemes and studies of the structure of products with areas of high background were fulfilled.

In order to establish the relationship between the levels of the background, which is detected during ultrasonic control, a study was fulfilled on the rolls, which have exhausted the full working life and were decommissioned. Measurements were performed on rolls of size ∅170×92×62; ∅215×120×72; ∅215×120×80; ∅215×120×85 mm. The test results showed that on rolls made by hot isostatic pressing technology, the background level is 1–1.4 divisions of the scale of the device display. On rolls made by the technology of hot vacuum pressing and controlled hot vacuum pressing – 0.4–0.9 divisions. The background level on the rolls made by vacuum sintering technology is 1–1.8 divisions.

Evaluation of the influence of the background level on the performance of finished products was performed by analyzing the performance of rolls with a known level of background in the conditions of finishing and pre-finishing groups of stands of rolling mills.

The analysis showed that the background level is lower than 1.8 divisions, does not affect the performance of the roll (the rate of operation, grinding). At the level of 2–2.2 divisions on the working calibers of rolling rolls,

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pores and shells visible to the naked eye are revealed during planned grinding (Fig. 5).

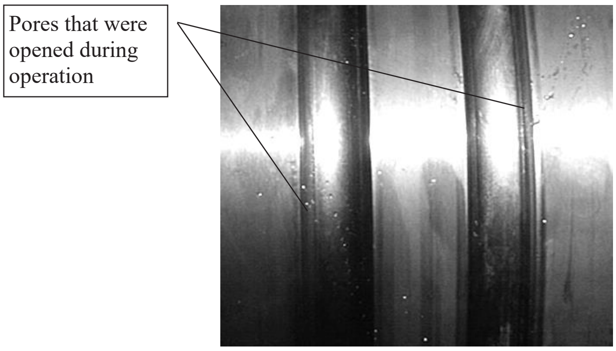

Fig. 5. The porosity of the roll ∅170×∅92×62 mm with a high level of ultrasound background

Thus, the results of research have shown that the main factor, influencing the amplitude of the background, is the porosity of the material.

Quantitative parameters, obtained in this work, refer to specific products made of sintered hard alloys of the WC-(Co+Ni+Cr) system with a binder phase content in the range of 10–30 % by weight. The products were obtained by hot vacuum pressing. Measurements of parameters were performed with the introduction of ultrasonic oscillations between plane-parallel end surfaces of the product. The amplitude of the background was determined in conventional units when normalizing the amplitude of the bottom pulse on 100 % of the vertical scale of the device display.

Of course, if the production technology, chemical composition, characteristics, or shape of the products will be different, the quantitative values of the amplitude of the scattering background may be different. Specific values of the background amplitude must be determined by accumulating statistics.

At the same time, the principle of determining the contiguity of the sceleton structure is based on the analysis of physical phenomena occurring during the propagation of ultrasonic vibrations and the proposed method of determining the contiguity of the sceleton structure can be applied to a wide range of materials with sceleton structure.

4. Conclusions

As a result of the fulfilled researches the following new quality control procedures for hard-alloy products were developed:

1. Estimation of the characteristics of the contiguity of the carbide sceleton carcass of the sintered hard alloy by measuring the speed of propagation of ultrasonic oscillations.
2. Estimation of the porosity level with a pore size less than 1 mm based on the results of measuring the relative amplitude of the background scattering of ultrasonic oscillations.

The proposed techniques are non-destructive and are carried out in one cycle with the fulfillment of ultrasonic flaw detection, which is exposed to 100 % of the products. These techniques are implemented in the production of carbide rolls by the method of controlled hot vacuum pressing. They have become an integral part of the quality control system of carbide rolls.

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