An evolution of grain structure in polycrystals formed during HPHT sintering of nanostructured diamond powders made using dynamic synthesis

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Introduction

This work is aimed at studying structural transformations during sintering of diamond powder of dynamic synthesis (powder grade AD). The aim of the work was to establish the possibility of creating a monophase nanodispersed material based on the initial powder of dynamic synthesis diamond (grade AD, manufactured in Dzerzhinsk). For this, we studied the structural transformation during sintering of the specified powder and the mechanisms of the formation of the grain structure.

Methods

The preliminary preparation of the powder for pressing was carried out by granulating the powder at p = 5 GPa at room temperature. Experimental samples were obtained by pressing the specified powder at p = 7.7 GPa in the range of 1700-2100 °C.
The microstructural characteristics of the particles of the initial diamond powder and polycrystalline samples sintered on its basis were studied by transmission electron microscopy in combination with microdiffraction.
The microhardness of the obtained samples was determined with a load on the indenter of 9.8 N (weight 1 kg).

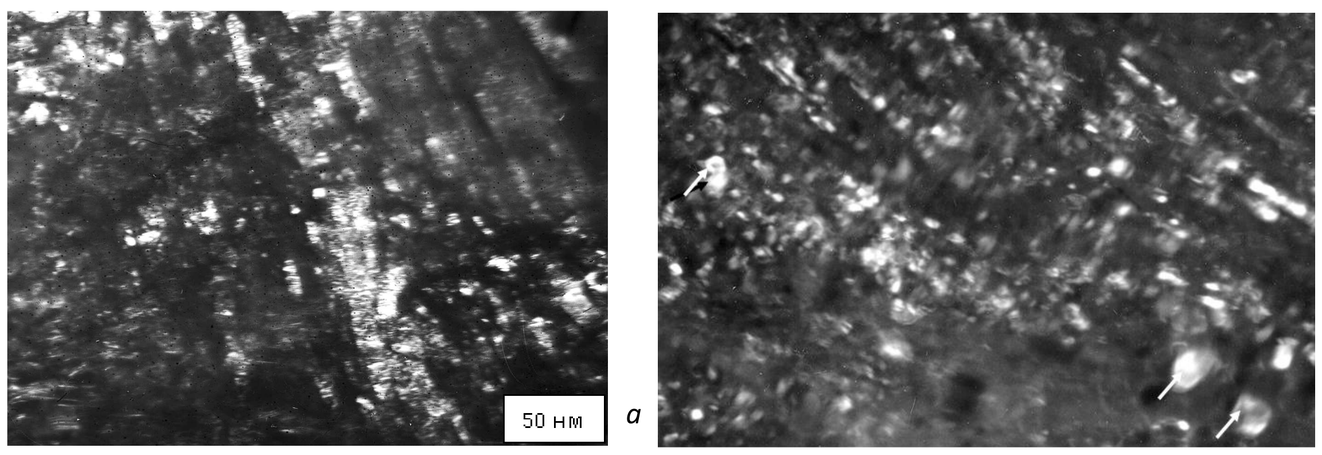

Fig. 1. Substructure of initial diamond particles (AD) with a high content of hexagonal (lonsdaleite) (a) and cubic (b) laths. The arrows indicate the associates of grains with internal interfaces.

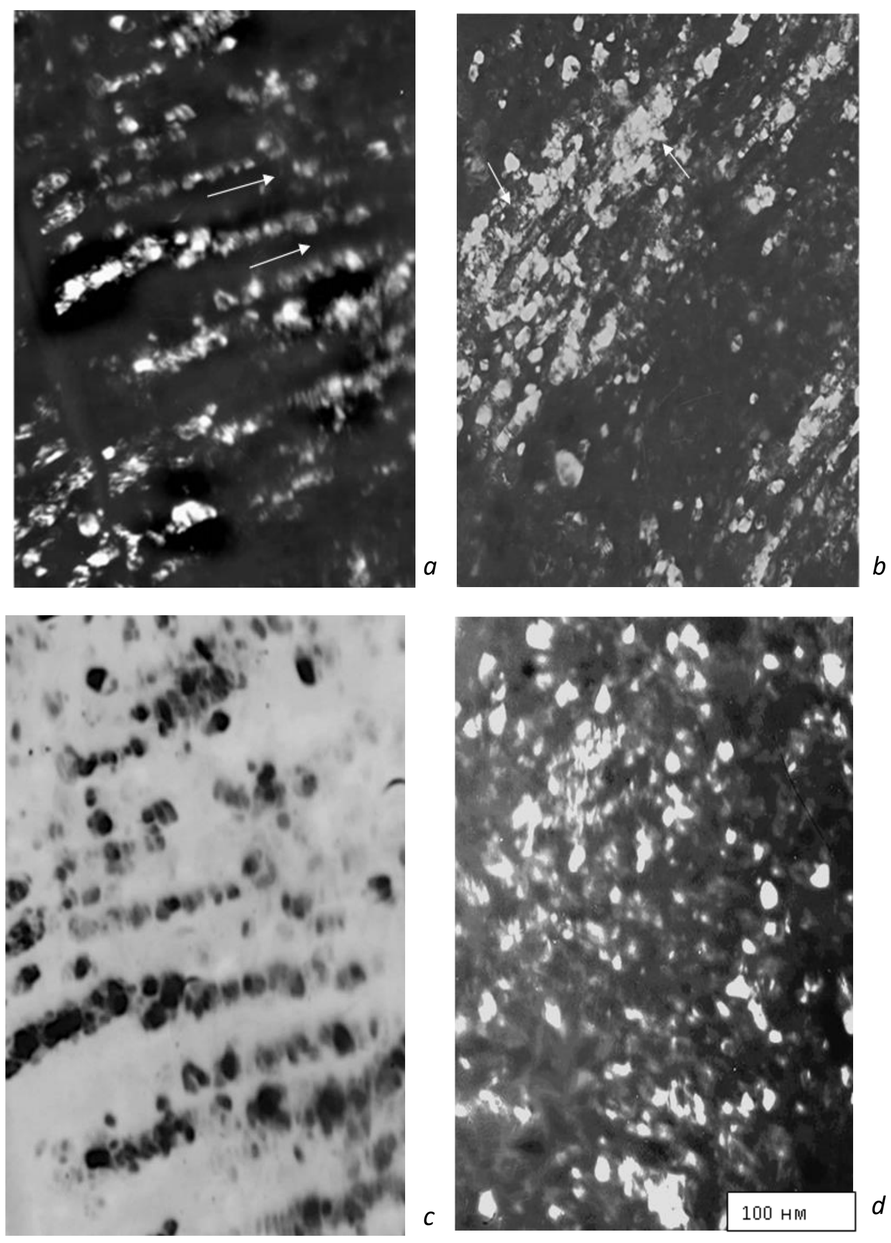

Fig. 2 Typical examples of the evolution of the lath substructure and grain structure in laths: predominant assembly of structural elements along the length of independent laths (a), combination of assembly in separate laths and between laths (b) and at the stage of predominant destruction of the lath substructure (c), granular structure of polycrystals obtained by sintering at T = 2100 °C - (d).

Results

The particles of the used diamond powder are in the form of plates. The size range of the developed surface of the plates is 1-5 microns. According to the results of microdiffraction studies, the bulk of the particles are practically monophase (cubic diamond). Two-phase (cubic diamond and hexagonal-lonsdaleite) particles are composed mainly of hexagonal diamond. The phases contained in these particles are crystallographically related to each other: $(111)_{а}П\ (001)_{Л}$
The particles have a four-level substructure: a particle (1), a package (2), consisting of laths (3). The laths of particles based on cubic diamond are composed of grains (4) with the main size range of 3-5 nm (these sizes correspond to the width of the laths). The two-phase particles contain batches of laths of three types in composition: lonsdaleite with basic stacking faults (a), twinned cubic diamond with different widths of twins (b), a combination of the indicated varieties of diamond and perfect diamond grains without twins (c). It should be noted that the width of the laths is different both in an individual particle and in different powder particles.

This also determines the difference in grain sizes in the particles. Monophase (cubic diamond) regions of particles also contain associates of grains, between which the interfaces are revealed. The size of the segregations reaches 30-40 nm. Such associates were observed for the first time in [3]. The data of [4] indicate that their formation can be explained by the development of self-organization in the system of nanodispersed grains already in the process of diamond synthesis at a high shock compression pressure. Figure 1.2 shows typical electron microscopic images of the substructure in the indicated types of particles.
During sintering of powders at high pressure, coarsening of grains and (or) their associates, contained in the laths of the initial particles or formed during sintering, takes place. According to the mechanism of the development of this process, the following stages can be distinguished: a) association (unification) of grains within the individual laths and between the laths (Fig. 3); b) the formation of thin boundaries between the grains in the associates; c) the appearance of the correct faceting or its elements of monolithic associates (new grains). Groups of such new grains are often formed along the laths.
The grain sizes over the sample volume are inhomogeneous, which is due (as already noted) to the different widths of the laths in the initial particles. This is most typical for samples obtained at sintering temperatures of 2000 °C. For these samples, the range of grain sizes is ≈ 10-50 nm. For the samples obtained at T = 2100 °C, there is a coarsening of grains and a homogenization of the range of their sizes (70-80 nm).
The table shows data on the microhardness of the obtained polycrystalline diamond samples. A slight decrease in the hardness of the studied samples (obtained at T = 2100 °C with an increase in the sintering time) can be explained by the appearance of microcracks during the preparation of the sample surface for hardness measurement.

№	Conditions for obtaining samples, when P=7,7 GPa		The value of hardness, GPa	The average value of the print size, μm
	T,⁰C	Endurance, c		
3	1700	60	18	28,174
4	2000	60	33	20,61
6	2100	60	33	20,784
7	2100	90	30	21,876
Load on the indenter – 9,8 H (weight -1 kg)				

Hardness calculation formula

$$HK = 0,102 \cdot \frac{F}{c \cdot d^2} = 1,451 \cdot \frac{F}{d^2},$$

Conclusion

1.For the first time, the features of the lath substructure in shock synthesis diamond, determined by the phase and structural state of the diamond, have been revealed.
2.It was established for the first time that the formation of a nano-grained structure during sintering of diamond powder of AD grade is determined by the presence of a lath substructure in the initial particles.
3.The sequence of structural transformations that determine the formation of the grain structure during sintering of the studied powders has been identified: a) the appearance of associates on the basis of lath structure elements (as individual grains); b) the latter are formed both within the rails and between the rails; c) the emergence of thin boundaries between grains in associates and the formation of elements of their correct faceting.
4.Comparison of the microhardness values of polycrystalline nanocrystalline diamond obtained in this work (table) and those given in [2] (Knopp hardness with a load of 4.98 N on the indenter is 110–130 GPa), indicate that the use of shock synthesis diamond powder allows to obtain a material with a sufficiently high microhardness when using moderate values of high pressure and temperature for sintering.

References

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