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Possibility of application of the computational intelligence in the production of superhard materials. Report 1

A new system to control DO138B model 630-ton and DO044 model 2500-ton presses (Russia) has been developed. The system not only controls the basic parameters of superhard materials synthesis and sintering, but also records a total of 11 parameters. The studies have been performed using high-pressure apparatuses of anvil with cavities. The features of behavior and the effect of individual parameters and their totalities on the superhard materials production have been discussed and the possibility to use these factors both for the process optimization and for the development of a future computational intelligence has been considered.

Keywords*: high pressure, superhard materials, synthesis, sintering, press, high-pressure apparatus, command.*

INTRODUCTION

Part of the information on the particularities of the processes of synthesis and sintering is given in [1–22]. The information is presented in publications related to industrial processes and scientific research of production of synthetic diamonds and other superhard materials at high pressures and high temperatures.

The explanation of this fact may be related to the “know-how” of each technology and the difficulties with logical analysis of the values of direct and indirect parameters, which influence the productivity of the process of obtaining superhard materials, despite the intense development of computer technology [1, 4–12].

It would be better to analyze it as thoroughly as possible from the phase diagrams of carbon in order to clarify the matter.

Chepurov et al. [13] compared the experimental data of nineteen researchers, who carried out synthesis of diamond in the Mn–Ni–C system. Theoretically, all values of pressure p and temperature T should be above the equilibrium line [16] of the phase diagram of carbon. However, comparing the data presented with the lines of equilibrium parameters [23–25] one concludes that thirty nine of the points of the parameters are below the line of Kennedy and Kennedy [24] (more reliable) and forty are above. This means that there is still not the correct starting point to aim at the exact values of key parameters of the synthesis. It is concluded that the irregularities of the diamond synthesis processes start here [26, 27].

As presented in [1, 3–13 and 18–21], the result of the synthesis process, particularly industrial, is explained by the fact that:

– the main p, T-parameters in the compression chamber of a high-pressure apparatus (HPA) are of difficult visual observation or direct control, i.e., it is impossible to measure p, T-parameters for all operations;

– it is visually impossible to evaluate the productivity and right quality of product of the synthesis (cluster) at the time of its obtaining;

– the processes of synthesis and its products significantly depend on quantitative and qualitative characteristics of other factors. These factors have not been definitively studied in relation to their influence on the production process.

It is concluded that the process of synthesis has an unstable character and depends on many phenomena related mainly to the solid pressure medium. Therefore, the productivity and quality of products vary from one operation to another. Some studies have attempted to explain the instability of the process [1, 3–5, 8, 9, 11, 28] noting:

– a change in pressure in the compression chamber of the HPA and the appearance of gradients of p, T parameters during the heating of the reactive cell and time of the operation;

– the discrepancy in the values of p, T parameters is related probably to the variation of the characteristics (density, weight, compressibility, etc.) and dimensions of the reactive cell components during a synthesis or sintering process;

– a change in the profile and status of the working surface of the HPA components from one operation to another;

– non-parallel surfaces of the press support blocks may appear, which causes the rapid decompression of the compressed medium followed by blow-out;

– electro-erosion can occur on the surfaces of components of HPA in the area of contact with the electric conductor graphite.

This is explained by the fact that the scientific community is not involved with real technology. It means that the community does not pay attention to problems related to automation of processes that use high pressures and temperatures. Although the world market already presents a flow of about 1000 tons of diamonds per year [22], we can deduce that the technology developed by the diamond-producing companies is using only small degree of the automation. However, some studies show separate data on the monitoring of synthesis and sintering parameters [1, 3–17, 21].

It was determined in [8] that under the action of the press, the heating of the reactive cell generated a significant increase in pressure compared with the value generated in the cold state in the compression chamber of the HPA. Later the same author [9] presented in detail the causes responsible for a sudden change in pressure during the synthesis. It is, mainly, the thermal expansion of reactive cells at the beginning of the process and the decrease in volume due to the graphite to diamond transformation.

There is a need to control also other parameters in addition to pressure and temperature. For example, according to [29], while maintaining the high pressure and high temperature, i.e., during synthesis, the distance between the anvils (critical thickness of the gasket) and parallelism between the working surfaces of them must be followed to decrease the instant depressurization in a way of a fast ejection of the material of the capsule followed by mechanical shock waves. Thus, the parameter “critical time” when controlled, may allow an increase in life of HPA and press [8, 10].

Therefore, the understanding of all the factors and their influence on the synthesis is a very difficult task and it seems impossible for automation. Some researchers have attempted to do this [30].

A basic technique for controlling and measuring pressure and temperature in the belt-type HPA is reported in [31]. The load pressure of the press is controlled using an auxiliary pump at low flow. The electrical power for heating is provided while monitoring the voltage. The current in the heater is regulated by the difference between the measured value in the controller input and the required temperature. It is possible to obtain diamond with cube–octahedral morphology and particle size between 0.1 mm and 0.7 mm in the synthesis performed with the use of this system. In this paper, technical details of the architecture, signs, type of controllers, control algorithms, and type of sensors are not provided. The indirect parameters such as temperature and coolant flow, temperature of HPA are not cited.

With relation to the process control of the synthesis parameters, diagrams of synthesis with their respective parameters of pressure and power versus time are proposed in [6, 7, 32, 33].

The diagrams show similar features, i.e. such as activating the heating after reaching the predetermined pressure and the heat deactivated before the pressure reduction. No information is submitted on possible methods and equipment for effective control of the appropriate parameters of diagrams and consideration of the negative factors.

The developed architecture of a computer system used to record the main parameters of the synthesis process was reported in [34]. According to the authors, the system has a feature that allows connecting it to various technological processes. This allows one to register the parameters of synthesis, i.e. the master cylinder pressure, components of HPA, current, power, voltage and temperature. The system was characterized as a universal system for storing data on synthesis and sintering. In this work the details of the software used for data acquisition and prerequisite knowledge needed to operate it were not opened.

Although some studies address various aspects, there are other causes that may influence the effectiveness of the processes of graphite into diamond transformation and sintering in industry. Therefore, to achieve the optimized synthesis and sintering, in addition to a storage system of the process data, it is necessary to use a programmable logic controller (PLC). This controller must be highly flexible programming, following, in preference, to IEC-61131 to change the logic operation of the process, considering all the possible parameters, and should seek the necessary data for the process optimization. In addition, the control system should give to the operator the means to change the parameters that indicate irregularity of the process. On the other hand, modern technology of computer science, which is based on the set theory and fuzzy logic [35], permits building based on joint analysis of the magnitudes of the parameters and expressions, the evaluation of indices of the product provided by linguistic data and intellectual model of the process of obtaining superhard materials. The theory using the CI (Computational Intelligence) presented in [36] did not propose the practical development and, especially, the technological parallel line to certain and rapid evaluation of the product by applying the purification and classification of diamonds.

It is attempted to develop and implement the primary fuzzy model for execution of the processes of synthesis of superhard materials and sintering of the composites to approach to the CI and to the optimization of the productive processes, considering the data in this study. This process uses the HPAs of the anvil type with a central cavity and toroid type with presses. A proposal should be developed for improving the functioning of commands of the existing presses, after analysis of the results obtained. The goal of this study is to find technical solutions for increasing the possibility of interpreting the values obtained as a basis for developing a system based on CI for this industry.

PROJECT TO MODERNIZE THE COMMAND OF PRESSES

The data on monitoring record systems of the process in industrial hydraulic presses of 2500-ton power model D0044 and 630-ton power model D0138B manufactured in 1993 by the Russian Rayzan' TyazhPressMash company [37] and installed in the North Fluminense State University (UENF), Brazil were not provided for scientific purposes.

At the project beginning the technological requirements were developed for a new automation system of the above presses, considering the accumulated experience and scientific-technological perspective. Thus, a modernization project is developed with SCADA (Supervisory Control and Data Acquisition Systems) that meets, among other resources, the scientific needs. Figure 1 illustrates an overview of the architecture of the SCADA system.

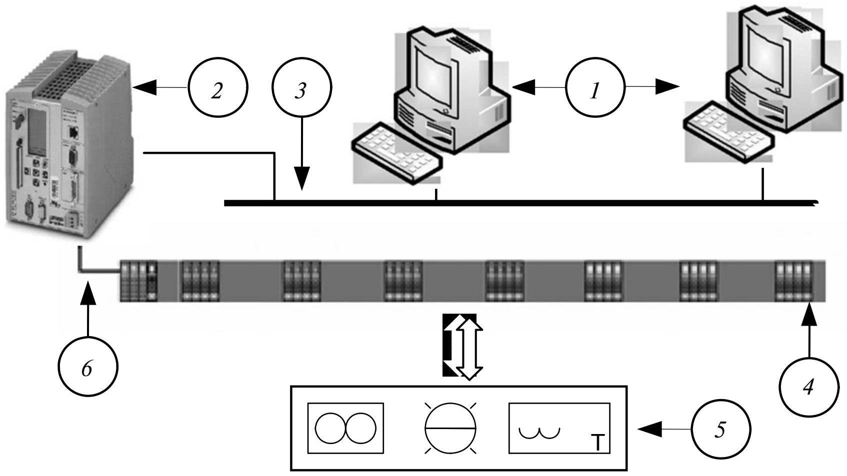

Fig. 1. SCADA System Architecture: computers with software of supervision (*1*), a PLC (*2*) networked Ethernet (*3*) with computers, input and output modules (*4*), the field instruments (*5*), a network (*6*).

The system consists of one or more computers with software of supervision, a PLC networked Ethernet with computers, input and output modules. They are connected to the PLC via network Interbus and receive and send signals to the field instruments.

This system allows performing an efficient and safe process control in the presses mentioned. For the main parameters measured and controlled, the following components are used:

– magnetic sensors to inform the position of moving parts of the press; Strain Gauge Transducer sensor in the hydraulic system to measure the pressure of main cylinder; conversion modules of electronic signals for reading of voltage and electric current; type K thermocouple sensors for measuring temperature of surface of HPA and type RTD (PT100) for input and output of cooling water; the flow sensor of cooling water; proximity sensor for measuring the critical height of the gasket, using laser sensor OMRON, Z4M-S100 model with signal conditioner model Z4M-W100 with a sensitivity of 8 μm;

– electronic input and output modules for reading various signals from the field and sending signals to the actuators in the field. Signals are divided into four types: AI (Analog Input), AO (Analog Output), DI (Digital input) and DO (Digital Output). These modules are connected with each other via communication network of shop floor called Interbus-S;

– central Processing Unit (CPU) from Phoenix Contact using the PcWorx programming software with IEC-61131 of the same manufacturer. The unit is installed remotely separated from the input and output (I/O) modules interconnected by an open network like “Fieldbus” type Interbus-S protocol. Logic and algorithms are developed to control the system, locks and interlocks, to improved safety of operators and equipment involved in the process.

A standard ISA PID algorithm, whose operation can be explained according to Fig. 2, is used to control the main variables, pressure and electrical current.

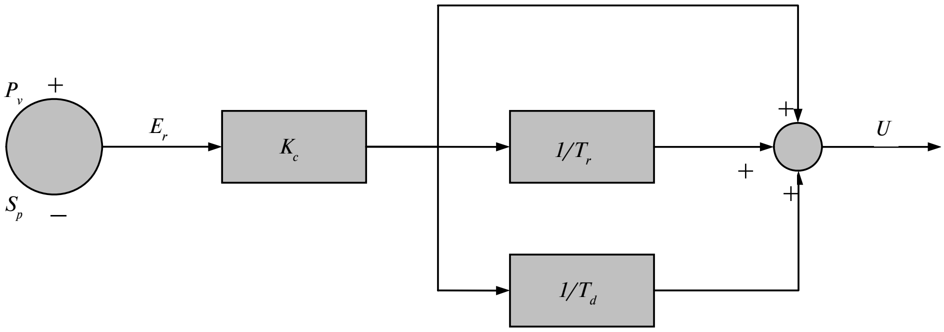

Fig. 2. Implemented PID controller using three controllers together.

The performance of the PID controller is shown mathematically in equations

$$U = K_c\left(E_r + \frac{1}{T_r}\int E_r dt + T_d\frac{dE_r}{dt}\right); \tag{1}$$

$$E_r = p_v - p_h, \tag{2}$$

where U is the controller output; K_c is the controller gain; T_r is the reset time; T_d is the derivative time; E_r is the error, difference between the values of the process variable p_v and the desired value p_h.

The value of the parameterized hydraulic pressure p_h is subtracted from the measured actual pressure p_v. This value is used to calculate the output correction to eliminate the difference between the values measured and parameterized. Moreover, proportional, integral and derivative calculations were performed to remove deviations of regime. However, the output of this controller operates in a final control element type on–off (pressure multiplier of the press or pump) making that the performance and consequent record of pressure measurement have variations of up to 1 % in the form of saw tooth. The PID cannot eliminate the phenomena of inertia of performance of pump, multiplier, and master cylinder. For this reason, in the future it would be better to complement a layout of the hydraulic press with auxiliary pump with a controlled flow.

The record apparently shows the electric current in a constant value compared with the desired value. Meanwhile, a second PID controller operates in seeking to maintain control of the electric current value as in the chosen diagram. Consequently, the PID controller must act to keep the electricity on the values set in advance. However, there may be deviations of up to 0.7 %, because the controller acts after the existence of the deviation.

The man–machine interface (MMI) of the monitoring system is developed using Elipse SCADA communication with driver OPC Client/Server communication driver and Windows® XP operating system. This tool interfaces are developed for operation in the manual and semi-automatic systems; diagnosis of communication/CPU; real trend chart; historical trend graph; recording data files in a standardized format; parameterization of the five diagrams of basic parameters; alarm history; diagnosis of the system operation; calibration of the displacement; temperature calibration; pressure calibration; matrix of causes and effects; among others. One can have access to the monitoring screens through the PC-AT. On each screen, features have been implemented, such as explanatory texts, emergency button, indication of states of the process and system, selected diagram already parameterized, instant alerts, trend graphs, connected user, alarms, among others.

The developed algorithms allow a flexible controlling that can perform any maintenance curve p_2 and T depending on the diagram of synthesis or sintering chosen. Algorithms for calculating the rates of changes of pressure, voltage and current are developed to identify and stop the process, in cases of rapid ejection in the form of explosion, short circuit, and electro-erosion not controlled; also, to evaluate the irregularity of the processes that can give the opportunity to create CI.

Thus, this system allows recording direct parameters like hydraulic pressure p_1, current I, power W, voltage V of the synthesis or sintering process as well as indirect ones like critical height h_c, flow of Coolant Q, electrical resistance R, cooling water temperatures T, during the time of synthesis it is also possible to measure the temperature of parts of the HPA. It is also possible to keep the parameters with adequate precision with the ability to change them during the process.

The evaluation of the new automation system was performed from tests under the real conditions of processing using for each press the computer itself in order to highlight the direct and indirect parameters that can be used in future CI.

EXPERIMENTAL

Based on research conducted at the UENF, the obtained results should be analyzed and the necessary parameters for development of CI indicated.

Processing of synthetic diamond

The high-pressure synthesis at 4.3 to 5.1 GPa was performed in an anvil type HPA with central cavity diameter of 55 mm [38], using the 2500-ton press. Figure 3 shows the layout of HPA with a fitted capsule (a) and deformed capsule (b), showing the formation of gasket h_c.

The calibration of the temperature and pressure generated in the apparatus is made using the methodology presented in [11]. This methodology uses this system

$$p_2 = f\,(p_1);$$

$$T = f(I \text{ or } W),$$

where p_1 is the pressure in the master cylinder; p_2 is the pressure generated in the compression chamber of HPA; T is the temperature in the reaction cell; I is the current and W is the electric power for heating. Moreover, the curve of elastic deformation of the whole "apparatus–support blocks" during loading of the press is defined to determine the real critical thickness of the gasket.

The arrangement of the reactive cell presents a homogeneous mixture of graphite powder and chips of the 42 % Ni–58 % Mn alloy by weight in proportion 1:1.

All components of the synthesis, including the shell, are prepared and separated with a dimensional uncertainty of ±0.5 %.

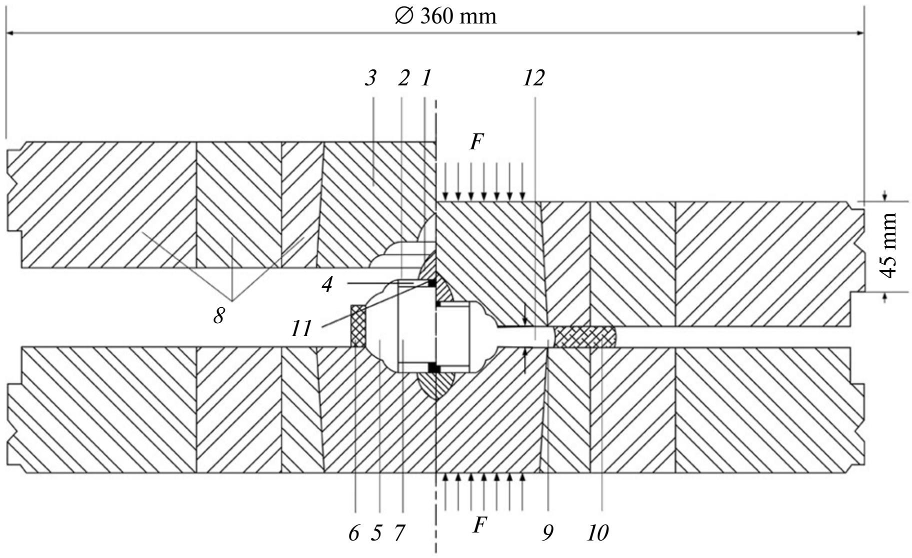

Fig. 3. HPA of the anvil type with a central cavity diameter of 55 mm before (*a*) and during (*b*) compression of the capsule: protection cone made of heat resistant alloy (*1*), anvil steel (*2*), plate of molybdenum (*3*), disk protection (*4*), deformable capsule (*5*), polymer ring (*6*), reactive mixture (*7*), strap (*8*), gasket (formed by extrusion of periphery capsule during compression) (*9*), polymer ring deformed (*10*), conducting cylindrical graphite (*11*), height of packing h_c (12).

Each operation of synthesis with new command has the following stages [39]:

– on-screen programming, elementary and a complex diagrams were chosen for maintenance of electrical current and hydraulic pressure parameterized in the system. The pressure increase can be performed in two variable steps due to the activation of one or two hydraulic pumps;

– the direct parameters (p_1, I, and T) are registered in the command system table. There are dimensions and weights of the components of synthesis and capsule at another table;

– the indirect parameters for the record were chosen;

– the execution of the process in the automatic system was activated by the push of a button on the graphical interface of the command system for control and registration;

– then, the pressure increase in the main cylinder of the press starts until p_1 = 102 or 105 MPa and an electrical current I = 850–965 A or other combinations;

– the value of pressure p_1 is maintained for 10 minutes either stable with an accuracy of ±0.2 MPa or varying from 102 to 105 MPa, periodically turning on and off the multiplier;

– the electric current was kept constant with an accuracy of ±0.3 % or uncontrolled (free);

– after 10 min of the simultaneous action of pressure and temperature, electric current was turned off and the pressure maintenance occurred without pump activation;

– after 30-s maintaining the pressure without control, there was a slow reduction to zero;

– process parameters are automatically recorded in charts and tables;

– the temperatures of the HPA components were also recorded using manual thermocouples and some peculiarities are observed during the synthesis;

– the product of synthesis (cluster) was broken in the axial and radial direction for prior assessment of the synthesis results in relation to the arrangement of crystals in the center and in the periphery, and habit of them. Then, the cluster was crushed and submitted to the process of recovery and purification of diamonds [40]. The coefficient of transformation of graphite into diamond [17] was applied to evaluate the productivity of the synthesis. All results must be recorded in the system.

The central composite design (CCD), which determines the parameters (Table 1), considering the results of experiments performed, is used to obtain the synthesis parameters more or less optimized with the purpose of studying the influence of automatic control of the synthesis parameters.

Table 1. Experimental planning to optimize diamond synthesis

Sample	Pressure p_1, MPa	Electrical current, A
1	104.0	955.0
2	105.4	955.0
3	104.0	955.0
4	104.0	955.0
5	103.0	965.0
6	104.0	955.0
7	104.0	933.8
8	104.0	976.2
9	104.0	955.0
10	105.0	970.0
11	105.0	940.0
12	102.6	955.0
13	103.0	940.0

130 experiments were made (65 without the automatic control of the synthesis process and 65 with the control) to ensure the rules of statistics.

Sintering of cBN

The process of sintering of the cBN composite was carried out using the 630-ton press, also with modernized command in accordance with the cited project. A toroid-type HPA with a central cavity of diameter 13.5 mm [41] was used to generate high pressure and high temperature. The sintering was performed at pressure in HPA from 6.5 to 8.0 GPa and power of 1000 W.

The cBN composites were prepared according to the following sequence of operations:

– the mixture of the cBN powder (kubonit, 10/14 micron offered by ISM, Ukraine) with the Al binder powder with a particle size of 106/125 μm (proportion of 90 wt% cBN) to form the reaction mixture;

– the compaction of the mixture into the mold under a pressure of 800 MPa, to obtain compacts with a diameter of 5.0 mm and 4.5 mm in height;

– the compact is placed within the graphite heater tube and this assembly is placed into the deformable calcite capsule with covers of graphite and calcite;

– drive of the command for generation and maintenance of the parameters of pressure and temperature for a certain time (1 min), in accordance with the cyclic diagram chosen [42];

– the maximum number of cycles is chosen equal to five. The samples were divided into five groups according to number of cycles. The reference of samples is done, for example, as follows: five samples of group 3 (three cycles) has index – 3.5.

RESULTS AND DISCUSSION

Diamond synthesis

The calibration of the 2500-ton Press-HPA system made with new command determines that the equations for pressure are p_2 (GPa) = 5.06 – (8.55/(1 + exp (p_1 – 10.87)/29.31)) and for temperature T (°C) = 1.749I – 120.

Capacity of the new control system

Figures 4 and 5 show typical curves of maintenance of direct and indirect parameters recorded and parameterized in the SCADA system during synthesis with high productivity (up to 6 grams of diamonds per operation). In the process of synthesis with controlled electrical current, it is kept the same constant at 890 A, (see Fig. 4), and the process without controlled electrical current suffers a change of the initial current 850 A to the maximum 890 A and to the final 920 A, respectively, (see Fig. 5). The other parameters recorded are: voltage V, power W, resistance R, flow cooling HPA Q, gasket critical height h_c, temperatures of input T_i and output T_o of cooling HPA water and temperature.

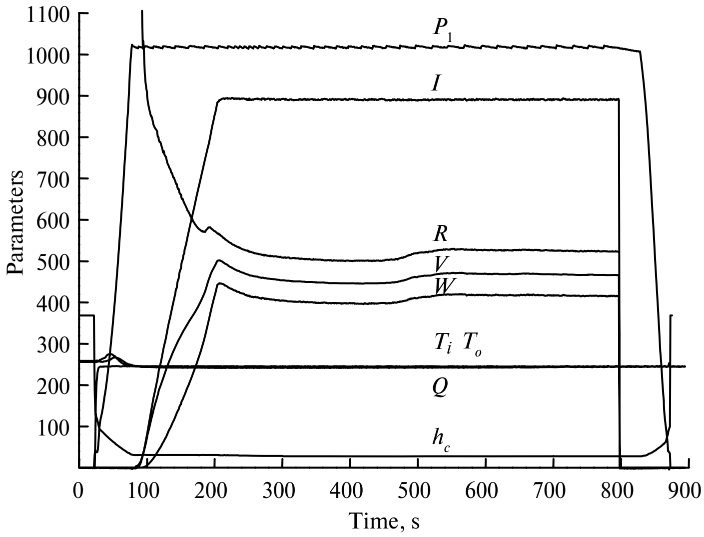

Fig. 4. Curve parameters for the synthesis performed at p_1 = 102 MPa, I = 890 A, the sample capsule height – 30.48 mm, total weight – 154.22 g, yield (coefficient of transformation) – 33.23 %: p_1, MPa×10; I, A; V, V×10^2; W, W×10^{-1}; R, Ω×10^5; Q, l/h×10^2; T_i, °C×10; T_o, °C×10; h_c, mm×10.

During the maintenance level of the hydraulic pressure p_1, the SCADA system makes use of the controller with parameters of proportional, integral and derivative (PID) in order to maintain pressure p_1 constant. These PID parameters are adjusted using a specific methodology in accordance with the process. It may be noted that the pressure increase is performed in one step due to the activation of two hydraulic pumps. The increase of the electric current is parameterized at a ramp with a constant rate.

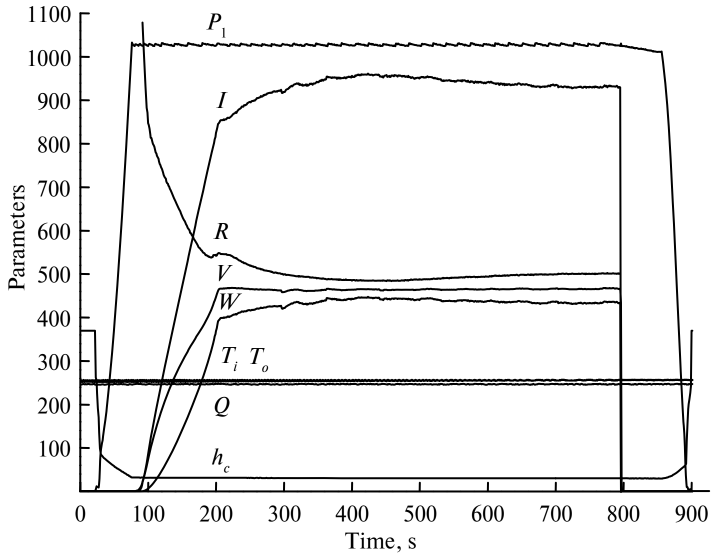

Fig. 5. Curve parameters for synthesis performed at p_1 = 103 MPa, free current I_i = 850 A, $I_{\max}$ = 963 A, I_f = 930 A, sample height – 30.26 mm, total weight – 154.01 g, yield – 35.67 %: p_1, MPa×10; I, A; V, V×10^2; W, W×10^{-1}; R, Ω×10^5; Q, l/h×10^2; T_i, °C×10; T_o, °C×10; h_c, mm×10.

However, as it can be seen in Figs. 4 and 5, the p_1 pressure is not exactly constant during the maintenance. That is due to the use of the hydraulic multiplier for generation of p_1 pressure in the main cylinder of the 2500-ton press. The jumps in the line of the graph related to the change of course of multiplier are observed. This effect leads to a less accurate maintenance of p_1 up to 1.2 % and is linked to the cyclic operation of the HPA. This cyclic operation can negatively influence the process and the HPA. The results presented further clarify the issue. The curve of electrical current I also did not show the linearity and accuracy of electrical current maintenance with dispersion up to 0.8 %, which is quite high. This may be connected with the action of the multiplier.

In the process without controlled electrical current tests show that the heating electrical circuit, consisting primarily of the thyristor module and heating transformer, tends to keep the voltage stable at a value of 4.6 Vca±0.06 to initial electrical current of 850 A (see Fig. 5). After the end of the heating ramp, electrical power and current are the inverse of electrical resistance and increase slowly for 200 s. This can be explained by melting the solvent/catalyst alloy that increased electrical conductivity of the reactive mixture. After this time heating electric current gradually decreases because of the formation of diamonds.

Table 2 presents a comparison between the parameters of the synthesis with and without control of electrical current. This difference in control mode can perhaps influence the quality of diamonds obtained and must be considered in the future developments.

In general, the integrals of the power curves showed that, on average, the consumption of electrical current in synthesis processes without automatic control was higher by 5 % kWh. However, on average, the productivity of this process is higher by 2.37 %. The obtained results of comparison only point to the particularities of each synthesis and would not reveal the unique advantages of each type of current control. It could only be noted that the process with current control has increased the HPA service life because of the low temperature.

Table 2. Comparison between synthesis process parameters with and without electric current control heating pad after the diagram A

Parameters	Uncontrolled current	Controlled current
Electrical resistance: 200 s after heating current set	Reaches the lowest level, after increases gradually until the end of the process	Reaches the lowest level, after 50 s increases and remains constant
Electrical power: 200/2 after heating current set	Follows an inverse of electrical resistance	Following behavior of electrical resistance
Voltage	Stable w/ variation of ±0.06 VAC	Following behavior of electrical resistance
Electrical resistance: 200 s after heating current set	Reaches highest level, after gradually reduces until the end of the process (920 A)	Constant (880 A)
Distance between parts of HPA (h_c – gasket critical height)	Gradually reduces to 0.20 mm with an average rate of 1.6 μm/s	Reduce 0.40 mm during 100 s with an average rate of 3.4 μm/s

The results obtained allow continuing research (see Report 2), giving more attention to the behavior of the parameters, which can be used in CI. The ability displayed by the new command gives the possibility to say that the future development of the automatic system based on CI is possible, but requires more research.

Розроблено нову систему управління пресами зусилля 630 (модель DO138B) і 2500 тон (модель DO044), яка контролює не тільки основні параметри синтезу і спікання надтвердих матеріалів, а також реєструє в цілому 11 параметрів. Проведено дослідження з використанням апаратів високого тиску типу "ковадла з заглибленнями". Обговорено особливості поведінки та вплив окремих параметрів і їх сукупності на створення надтвердих матеріалів і розглянуто можливість використання цих факторів для оптимізації і створення в майбутньому інтелектуального управління процесом синтезу.

***Ключові слова**: високий тиск, надтвердих матеріалів, синтез, спікання, стиснення, апарат високого тиску.*

Разработана новая система управления прессами усилием 630 (модель DO138B) и 2500 тонн (модель DO044), которая контролирует не только основные параметры синтеза и спекания сверхтвердых материалов, а также регистрирует в общей сложности 11 параметров. Проведены исследования с использованием аппаратов высокого давления типа "наковальни с углублениями". Обсуждены особенности поведения и влияние отдельных параметров и их совокупности на создание сверхтвердых материалов и рассмотрена возможность использования этих факторов для оптимизации и создания в будущем интелектуального управления процессом синтеза.

***Ключевые слова**: высокое давление, сверхтвердых материалов, синтез, спекание, сжатие, аппарат высокого давления.*

1. *Sverkhtverdye materialy. Poluchenie i primenenie* (Superhard Materials. Production and Properties): In 6 vol. / Ed. N. V. Novikov; National Academy of Sciences of Ukraine. Bakul Institut of Superhard Materials. – Vol. 1. Synthesis of Diamonds and Related Materials / Ed. A. A. Shul'zhenko. – Kiev, 2003. – 320 p.

2. *Novikov N. V.* New tendency in high pressure synthesis // Diamond Rel. Mater. – 1999. – **8**, N 8–9. – P. 1427–1432.
3. *Shipilo B. V., Dutov A. G., Shipilo N. V. et al.* Kinetic particularities of diamond crystallization from the melt of the Mn–Ni–C system // J. Superhard Materials. – 2002. – **24**, N 2. – P. 47–54.
4. *Bezrukov G. N., Poliakov V. P., Mukhametkulova M. M., Doladze N. T.* Study of the kinetics of graphite into diamond catalytic transformation under different thermodynamic conditions of the synthesis // J. Superhard Materials. – 1982. – N 2. – P. 7–10.
5. *Potemkin A. A., Poliakov V. P.* The evolution of pressure during diamond synthesis in the high pressure apparatus // High Pressure Sci. Technol. – 1998. – **7**. – P. 1004–1006.
6. *Wakatsuki M.* Formation and growth of diamond – for better understanding and control of the process // Ibid. – 1998. – **7**. – P. 951–956.
7. *Wang Y., Takanabe R., Kagi H. et al.* Advances in New Diamond Science and Technology / Eds. by Saito et al. – Tokyo: MY, 1994. – 521 p.
8. *Shulzhenko A. A., Maslenko Yu. S. Belousov I. S. et al.* Study of the pressure variation at high temperature in synthesis of superhard materials // Vliyanie vysokikh davlenii na veshchestvo (The influence of high pressure on the substances). – Kiev: IPM AN UkrSSR, 1977. – P. 113–117.
9. *Shulzhenko A. A., Bojko S. A. et al.* Sintez, spekanie i svoistva kubicheskogo nitrida bora (Synthesis, Sintering and properties of cubic boron nitride). – Kiev: Naukova Dumka. – 1993. – 256 p.
10. *Novikov N. V., Prikhna A. I., Borimsky A. I.* Steel high-pressure apparatus for synthesis of superhard materials // Proc. High Pressure in Research and Industry: 8th AIRAPT Conf., 19 EHPRG Conf., Uppsala, Sweden, 17–22 Aug. 1981. – Vol. 2. – P. 493–498.
11. *Ivakhnenko S. A., Shulzhenko A. A., Vitiuk V. I., Belousov I. S.* Pressure measurement in an anvil type high-pressure apparatus with a depression at 1200–1300 °C // Soviet J. Superhard Materials. – 1987. – N 4. – P. 9–11.
12. *Shipilo V. B., Dutov A. G., Shipilo N. V. et al.* Crystalization diamond at various temperatures and studies of their imperfect structure // J. Superhard Materials. – 2003. – N 4. – P. 46–51.
13. *Chepurnov A. I., Fedorov I. I., Sonin V. M.* Eksperimental'noe modelirovanie processov almazoobrazovaniya (Experimental Modeling of the Diamond Formation Processes). – Novosibirsk: Siberian Branch RAS, 1997. – 198 p.
14. *Novikov N. V., Levitas V. I., Leshchuk A. A., Idesman A. V.* Mathematical modeling of diamond synthesis process // High Pressure Research. – 1991. – **7**. – P. 195–197.
15. *Perevertaylo V. M., Loginova O. B*., *Ostrovskaia L. Yu*. The capillary phenomena in the process of diamond synthesis // Sverkhtverdye materialy. Poluchenie i primenenie (Superhard materials. Production and Properties): In 6 vol. / Ed. N. V. Novikov; National Academy of Sciences of Ukraine. Bakul Institut of Superhard Materials. – Vol. 2. Structure and properties of superhard materials, methods of study. – Kiev, 2004. – P. 41–83.
16. *Butylenko A. K., Ignatieva I.* The influence of pressure up to 60 kbar on the fusion diagram of the system Mn–Ni–C // DAN USSR. Serie A. – 1977. – N 2. – P. 168–171.
17. *Skury A. L. D., Bobrovnitchii G. S., Miller S. N.* The graphitization process and the synthesis of diamond from C–Mn–Ni system // Carbon. – 2004. – N 42. – P. 2369–2373.
18. *Strong H. M., Bundy F.* Accurate Characterization of the High-Pressure Environment / Ed. E. L. Lloyd. – NBS Special Pub., 1971. – Vol. 326. – P. 283–290.
19. *Samoylovitch M. I., Sanjerlinskii N. G.* The thermodynamic features of the direct transition of graphite into diamond and diamond crystallization from supersaturated carbon solution // Sintez mineralov (Synthesis of minerals): In 2 vols. – M.: Nedra, 1987. – Vol. 1**,** part III, chapter 14. – P. 301–316.
20. *Shalimov M. A., Kalashnikov I. A., Nikol'skaia I. V. et al.* Synthesis of diamond from heat treated graphitizing carbon raw material // Almazy i Sverkhtverdye Materialy. – 1977. – N 2. – P. 1–3.
21. *Katsay M. Ya., Sakovitch Yu.* Kinetics of the pressure change in an anvil-type HPA with cavities at different temperatures in the process of diamond spontaneous crystallization // High Pressure Physics and Technics. – 1987. – N 2. – P. 40–43.
22. *Owens C.* Industrial diamond applications, economics and view to the future // IDR. – 2000. – **60**, N 586. – P. 176–182.
23. *Berman R., Simon F. E.* The graphite-diamond equilibrium // Zeitschrift für Elektrochemie und Angewandte Physikalische Chemie. – 1955. – **59**. – P. 333–338.

24. *Kennedy C. S., Kennedy G. C.* The equilibrium boundary between graphite and diamond // J. Geophysic. Res. – 1976. – **81**. – P. 2467–2470.
25. *Bundy F. P.* The pressure-temperature phase and reaction diagram for carbon // Mat. Res. Symp. Proc. – 1995. – **383**. – P. 3–20.
26. *Turkevich V. Z., Kulik O. G.* High pressure influence on the phase diagram construction of 3-*d* transition metals with carbon system // High. Pressure Res. – 1995. – **14**. – P. 175–180.
27. *Katsay M. Ya., Sakovitch Yu. N.* Determination of the *p*, *T*-region of spontaneous formation of diamonds in the system Mn–Ni–Graphite system // Soviet J. Superhard Materials. – 1989. – N 1. – P. 17–21.
28. *Vinogradov S. A.* Some Regularities of plastic deformation of the deformable capsule of HPA of the anvil with cavities type // Obrabotka materialov pri vysokikh davleniyakh (Treatment of Materials at High Pressures). – Kiev: IPM AN UkrSSR, 1987. – N 10. – P. 138–142.
29. *Bobrovnitchii G. S., Carvalho E. A.* Influence of system accuracy "Press–Device" for high pressure performance in the conditions of production of super hard materials // Proc. IV Latin American Congress of Mechanical Engineering, CIDIM-1999, Santiago, Chile. – Vol. 1. – P. 153–157.
30. *Glagovsky B. A., Laptchevsky N. V., Lysanov V. S. et al.* Automation and mechanization of technological processes of synthetic superhard materials production // Mechanization and Automation of Production. – 1980. – N 3. – P. 2–4.
31. *Fukunaga O., Ko Y. S., Konoue M. et al.* Pressure and temperature control in flat-belt type high pressure apparatus for reproducible diamond synthesis // Diamond Relat. Mater. – 1999. – **8**. – P. 2036–2042.
32. *Vianna W. S., Bobrovnitchii G. S., Monteiro S. N.* Monitoring of diamond synthesis by changes in the heating electric current // Proc. Int. Sci. Technol. Conf. on Superhard Tool Materials on the turn of the centuries: Production, Properties and Applications, Kiev, Ukraine, 4–6 July, 2001. – P. 284.
33. *Pink R. L.* Model for control of heating processes in the reaction volume of high pressure cell // Obrabotka materialov pri vysokikh davleniyakh (Treatment of materials by high pressure). – Kyev: IPM AN UkrSSR, 1987. – Iss. 10. – P. 132–137.
34. *Borimsky A. I., Jerebko V. A., Kotenko Y. V*. System of acquisition and processing information on phase transformations in materials at HP–HT parameters on the basis of a personal computer // *P*orodorazrushayushchii i metalloobrabatyvayushchii instrument – tekhnika i technologiya ego izgotovleniya i primeneniya (Rock Destruction and Metal-Working Tools – Techniques and Technology of the Tool Production and Applications): Collect. Sci. Papers. – Kiev: Bakul' ISM, 2003. – Iss. 6. – P. 179–184.
35. *Aliev R. A., Tserkovny A. E., Mamedov A. G.* Upravlenie proizvodstvom pri nechetkoi iskhodnoi informatsii (Management of Production with a Fuzzy Starting Information). – M.: Energoatomizdat, 1991. – 240 p.
36. *Kravets P. I.* Intelligent models and fuzzy algorithms and application of them for optimizing processes of synthesis of superhard materials. Reports 1 and 2 // J. Superhard Materials. – 2001. – N 3. – P. 15–23; N 4. – P. 29–37.
37. *Bobrovnitchii G. S., Belovol V. S.* New hydraulic presses for the production of superhard materials // Novye mashiny i technologii kuznechno-pressovogo proizvodstva (New Industry Machines and Technologies of Forging Press). – Moscow: Ed. VNIIMETMASH. 1991 – P. 15–24.
38. *Pat. 5087 Ukraine, ICL B01J3/06.* High pressure high temperature device / A. I. Prikhna, A. I. Borimsky, D. A. Nagorny. – Publ. 28.12.94.
39. *Vianna W. S., Bobrovnitchii G. S.* Optimization of the process of industrial diamond synthesis // 6th World Congresses of Structural and Multidisciplinary Optimization, Rio de Janeiro, Brazil, 30 May–03 June 2005: COPPE Publ. – Vol. 1. – P. 63–69.
40. *Skury A. L. D., Bobrovnitchii G. S., Monteiro S. N.* Experimental design methodology applied to study the diamond purification process // Diamond Relat. Mater. – 2004. – **13**. – P. 1638–1644.
41. *Pat. 3854854 US, IPC B01J3/06.* High pressure producing apparatus // L. F. Verestshagin, L. G. Khvostantsev. – Publ. 17.12.74.
42. *Pat. P10701534-8 Brasil.* Um processo da sinterização de materiais policristalinos e compósitos a base de materiais superduros / A. M. Ramalho, G. S. Bobrovitchii, A. L. D. Skury, S. N. Monteiro. – Publ. 12.04.07.

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