

FROM THE HISTORY OF PHYSICS

PACS number: **01.65.+g**

Mysteries of diamond synthesis at the Institute of High Pressure Physics of the Academy of Sciences of the Soviet Union

S M Stishov

DOI: https://doi.org/10.3367/UFNe.2019.03.038545

Abstract. A history of diamond synthesis at the Institute of High Pressure Physics, Academy of Sciences of the Soviet Union (IHPP), is described based on archival materials. Many details of those events are still unclear. In particular, it is not known what IHPP researcher was the first to see in his apparatus the first diamond synthesized in the USSR. Nor is it entirely clear why the high-pressure group at the Institute of Crystallography, Academy of Sciences of the Soviet Union, which was quite close to success, was ruthlessly liquidated. The history described allows the reader to better understand the organization of science in the USSR that is often idealized nowadays.

Keywords: diamond, borazon, belt, anvils

There is a large number of reports and memoirs concerning the synthesis of diamonds in the laboratories of ASEA (General Swedish Electric Company, Sweden) and General Electric (USA). Nothing similar exists about corresponding work performed in the USSR. It is clear that this work was classified, but a lot of time has passed since then, and the very state of the USSR no longer exists. Unfortunately, none of the participants in that work took upon themselves the task of writing memoires, and now they are no longer with us. Nevertheless, using the retained archival materials, it is possible to partially restore the course of events that finally led to the hardly explicable success achieved at the Institute of High Pressure Physics (IHPP) of the USSR Academy of Sciences.

Attempts to obtain artificial diamond have a long history. According to legends, everything began with the experiments of Florentine academicians, who evaporated diamond with a focused sunbeam. Subsequently, the French scientist Antoine Loran Lavoisier (1743–1794) was more cautious: he did the same experiment with a diamond placed in a sealed vessel and ascertained that the only compound formed in the vessel as a result of the combustion of the diamond was carbon dioxide. Thus, it became clear that diamond consists of pure coal (carbon).

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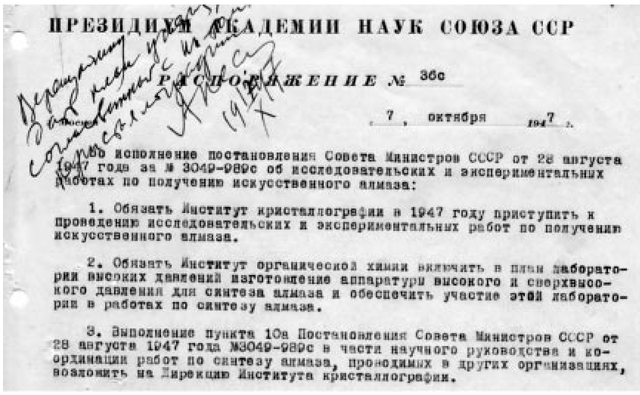
Received 1 March 2019
Uspekhi Fizicheskikh Nauk **189** (7) 752 – 758 (2019)
DOI: https://doi.org/10.3367/UFNr.2019.03.038545
Translated by S N Gorin; edited by V L Derbov

Apparently, since those times, humanity began to dream about the transformation of chimney or furnace soot into a placer of diamonds. It is not possible to describe all attempts made in this area. I will mention only two of them, which attracted definite interest at that time. The first was the experiments of the English chemist J B Hannay, who heated different organic materials in metallic tubes whose ends were closed by forging. Hannay obtained small transparent crystals, which were then deposited at the British Museum.

Almost no one believed that Hannay's small crystals were diamonds. However, many years later, the English crystallographer Kathleen Lonsdale investigated these crystals by the X-ray diffraction method and, to everyone's surprise, revealed that they indeed proved to be diamonds. The reason for the surprise consisted in the fact that the method of their preparation used by Hannay, as we know nowadays, could not provide conditions necessary for obtaining diamonds; thus, the appearance of diamonds in Hannay's experiments is a mystery. Possibly, Hannay used small diamond crystals as seed crystals and for some reasons believed that the crystals he revealed were the product of his studies; or someone simply put these crystals secretly into the mixture in order to stop exhausting experiments. A similar case happened much later in experiments carried out at the General Electric (GE) Research Laboratory (see below). At approximately the same time, the French chemist H Moissan crystallized carbon from molten iron and obtained small hard crystals, which proved to be crystals of silicon carbide (the iron employed appeared to contain a sufficiently large amount of dissolved silicon).

One way or another, no one succeeded in synthesizing artificial diamonds before the war years in the middle of the 20th century. However, in the war and postwar years, at least three countries, namely, the USA, the USSR, and Sweden, which had no natural resources of diamonds at that time, faced the problem of obtaining them artificially. As was noted in the memoirs of Tracy Hall [1] (one of the main participants of successful synthesis of diamonds at General Electric), the task of synthesizing diamonds was laid out by Chauncey Guy Suits in 1951, when he was the director of the GE Research Laboratory. The motivation for this solution was quite obvious. The company was engaged in the mass production of light bulbs, the main part of which is a tungsten filament. The necessary tungsten filaments were drawn out using expensive diamond dies, the diamonds for which were bought in South Africa or Brazil. The Swedish company ASEA was able to start research aimed at the synthesis of diamond already in 1942 [2]. It is interesting that while in the USA and Sweden the task of synthesizing diamonds was set at



ПРЕЗИДИУМ АКАДЕМИИ НАУК СОЮЗА ССР

РАСПОРЯЖЕНИЕ № 36с

7 октября 1947 г.

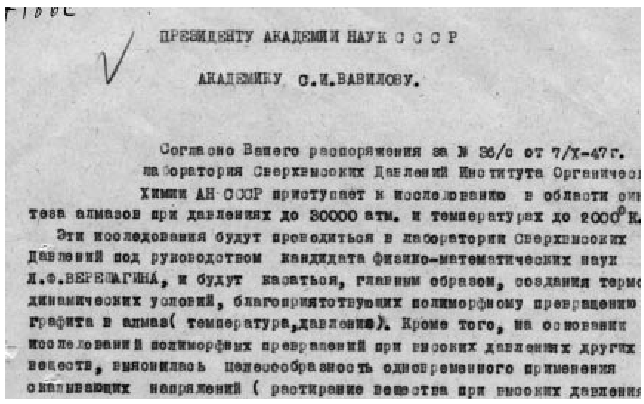
Во исполнение постановления Совета Министров СССР от 28 августа 1947 года за № 3049-989с об исследовательских и экспериментальных работах по получению искусственного алмаза:

1. Обязать Институт кристаллографии в 1947 году приступить к проведению исследовательских и экспериментальных работ по получению искусственного алмаза.

2. Обязать Институт органической химии включить в план лаборатории высоких давлений изготовление аппаратуры высокого и сверхвысокого давления для синтеза алмаза и обеспечить участие этой лаборатории в работах по синтезу алмаза.

3. Выполнение пункта 10а Постановления Совета Министров СССР от 28 августа 1947 года №3049-989с в части научного руководства и координации работ по синтезу алмаза, проводимых в других организациях, возложить на Дирекцию Института кристаллографии.

Figure 1. Order of the Presidium of the Academy of Sciences of the USSR dated October 7, 1947 concerning the start of work on the synthesis of artificial diamonds.



ПРЕЗИДЕНТУ АКАДЕМИИ НАУК СССР

АКАДЕМИКУ С.И.ВАВИЛОВУ.

Согласно Вашего распоряжения за № 36/с от 7/X-47 г. лаборатория Сверхвысоких Давлений Института Органической Химии АН СССР приступает к исследованию в области синтеза алмазов при давлениях до 30000 атм. и температурах до 2000°К.

Эти исследования будут проводиться в лаборатории Сверхвысоких Давлений под руководством кандидата физико-математических наук Л.Ф.ВЕРЕЩАГИНА, и будут касаться, главным образом, создания термодинамических условий, благоприятствующих полиморфному превращению графита в алмаз(температура,давление). Кроме того, на основании исследований полиморфных превращений при высоких давлениях других веществ, выяснилась целесообразность одновременного применения скалывающих напряжений (растирание вещества при высоких давлениях

Figure 2. Letter to S I Vavilov from A N Nesmeyanov concerning the readiness of L F Vershchagin to start investigations in the field of the synthesis of artificial diamonds.

the level of a separate (although large) company, in the USSR this problem acquired national state importance because of the decree of the Council of Ministers of the USSR dated August 28, 1947, on research and experimental work to obtain artificial diamonds (Fig. 1).

In accordance with the decree of the Council of Ministers, the Presidium of the USSR Academy of Sciences, by a decision dated October 7, 1947, obliged

(1) the Institute of Crystallography of the USSR Academy of Sciences (ICAS) to start "research and experimental work on obtaining artificial diamonds";

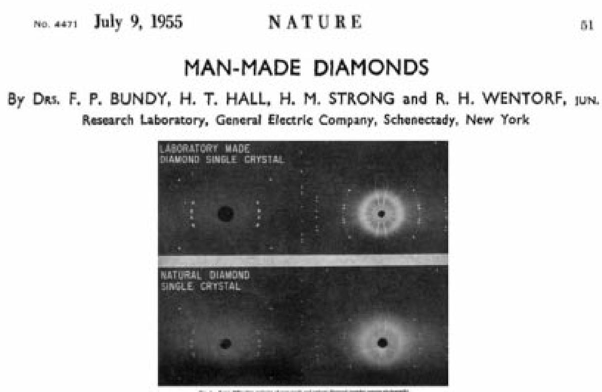
(2) the Institute of Organic Chemistry of the USSR Academy of Sciences (IOC) to include in the plan of the High-Pressure Laboratory the production of equipment for high and ultrahigh pressures for the synthesis of diamonds.

Simultaneously, the Presidium of the USSR Academy of Sciences entrusted ICAS with the responsibility for the scientific guidance and general coordination of work on the synthesis of diamonds.

Almost instantly, A N Nesmeyanov, who was then the director of IOC, in a letter dated October 22, 1947, reported to the President of the USSR Academy of Sciences S I Vavilov that the appropriate studies at pressures to 30, 000 atm and temperatures to 2000 K would be headed by Candidate of Science in Physics and Mathematics L F Vereshchagin (Fig. 2).

It is interesting that by that time articles F Rossini and R Jessup [3] and O I Leipunskii [4] had already been published, from which it was evident that higher pressures would be required for the synthesis of diamond. However, at that time, no one in the world, including the patriarch in the field of high pressures, the Nobel Laureate Persi W Bridgman (Harvard University), had the technology to obtain the necessary combination of pressure and temperature (60,000 bars and 2000 K). Since those times, i.e., beginning on October 22, 1947, no information connected with the development of equipment for the synthesis of diamond has apparently come from Vereshchagin's laboratory. And seemingly nothing essential has occurred at the Institute of Crystallography either.

Suddenly, in July 1955 in the journal *Nature* a paper titled "Man-Made Diamonds" appeared (Fig. 3), in which, on behalf of the team from the Research Laboratory of the General Electric Company (Fig. 4), it was reported that diamond had been synthesized for the first time in the world



No. 4471 July 9, 1955 NATURE 51

MAN-MADE DIAMONDS

By Drs. F. P. BUNDY, H. T. HALL, H. M. STRONG and R. H. WENTORF, Jun.

Research Laboratory, General Electric Company, Schenectady, New York

Figure 3. First article on the synthesis of diamond at the Research Laboratory of General Electric.

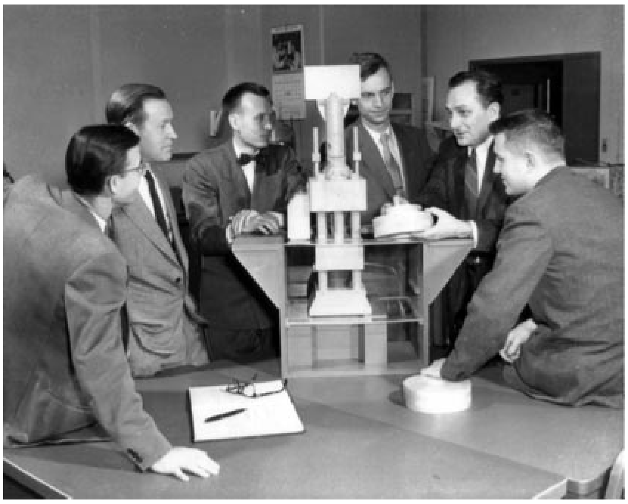
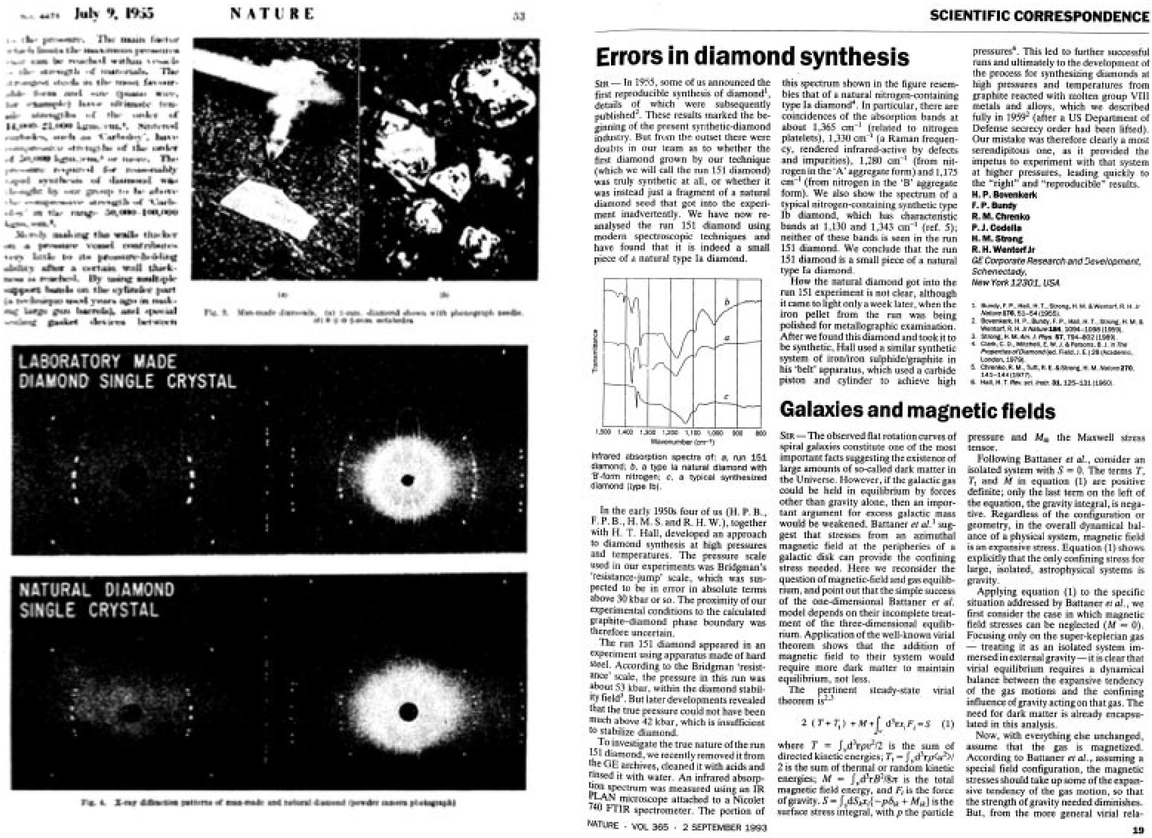


Figure 4. Researchers at the Laboratory of General Electric who first implemented the synthesis of diamond (from left to right): Francis Bundy, Herbert Strong, Tracy Hall, Robert Wentorf, Anthony Nerad (team leader), James Cheney (technician). It is instructive that the project manager Anthony Nerad never included his name in the group of authors of diamond synthesis.

[5] (information for the press had already appeared in February 1955 but was not widely known then). However, no detailed information about the conditions of the synthesis



July 9, 1955 NATURE 53

LABORATORY MADE DIAMOND SINGLE CRYSTAL

NATURAL DIAMOND SINGLE CRYSTAL

SCIENTIFIC CORRESPONDENCE

Errors in diamond synthesis

SIR — In 1955, some of us announced the first reproducible synthesis of diamond[1], details of which were subsequently published[2]. These results marked the beginning of the present synthetic-diamond industry. But from the outset there were doubts in our team as to whether the first diamond grown by our technique (which we will call the run 151 diamond) was truly synthetic at all, or whether it was instead just a fragment of a natural diamond seed that got into the experiment inadvertently. We have now reanalysed the run 151 diamond using modern spectroscopic techniques and have found that it is indeed a small piece of a natural type Ia diamond.

Infrared absorption spectra of: a, run 151 diamond; b, a type Ia natural diamond with 'B'-form nitrogen; c, a typical synthesized diamond (type Ib).

In the early 1950s four of us (H. P. B., F. P. B., H. M. S. and R. H. W.), together with H. T. Hall, developed an approach to diamond synthesis at high pressures and temperatures. The pressure scale used in our experiments was Bridgman's 'resistance-jump' scale, which was suspected to be in error in absolute terms above 30 kbar or so. The proximity of our experimental conditions to the calculated graphite–diamond phase boundary was therefore uncertain.

The run 151 diamond appeared in an experiment using apparatus made of hard steel. According to the Bridgman 'resistance' scale, the pressure in this run was about 53 kbar, within the diamond stability field[3]. But later developments revealed that the true pressure could not have been much above 42 kbar, which is insufficient to stabilize diamond.

To investigate the true nature of the run 151 diamond, we recently removed it from the GE archives, cleaned it with acids and rinsed it with water. An infrared absorption spectrum was measured using an IR PLAN microscope attached to a Nicolet 740 FTIR spectrometer. The portion of this spectrum shown in the figure resembles that of a natural nitrogen-containing type Ia diamond[4]. In particular, there are coincidences of the absorption bands at about 1,365 cm^{-1} (related to nitrogen platelets), 1,330 cm^{-1} (a Raman frequency, rendered infrared-active by defects and impurities), 1,280 cm^{-1} (from nitrogen in the 'A' aggregate form) and 1,175 cm^{-1} (from nitrogen in the 'B' aggregate form). We also show the spectrum of a typical nitrogen-containing synthetic type Ib diamond, which has characteristic bands at 1,130 and 1,343 cm^{-1} (ref. 5); neither of these bands is seen in the run 151 diamond. We conclude that the run 151 diamond is a small piece of a natural type Ia diamond.

How the natural diamond got into the run 151 experiment is not clear, although it came to light only a week later, when the iron pellet from the run was being polished for metallographic examination. After we found this diamond and took it to be synthetic, Hall used a similar synthetic system of iron/iron sulphide/graphite in his 'belt' apparatus, which used a carbide piston and cylinder to achieve high pressures[6]. This led to further successful runs and ultimately to the development of the process for synthesizing diamonds at high pressures and temperatures from graphite reacted with molten group VIII metals and alloys, which we described fully in 1959[2] (after a US Department of Defense secrecy order had been lifted). Our mistake was therefore clearly a most serendipitous one, as it provided the impetus to experiment with that system at higher pressures, leading quickly to the "right" and "reproducible" results.

H. P. Bovenkerk
F. P. Bundy
R. M. Chrenko
P. J. Codella
H. M. Strong
R. H. Wentorf Jr
GE Corporate Research and Development, Schenectady, New York 12301, USA

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Galaxies and magnetic fields

SIR — The observed flat rotation curves of spiral galaxies constitute one of the most important facts suggesting the existence of large amounts of so-called dark matter in the Universe. However, if the galactic gas could be held in equilibrium by forces other than gravity alone, then an important argument for excess galactic mass would be weakened. Battaner et al.[1] suggest that stresses from an azimuthal magnetic field at the peripheries of a galactic disk can provide the confining stress needed. Here we reconsider the question of magnetic-field and gas equilibrium, and point out that the simple success of the one-dimensional Battaner et al. model depends on their incomplete treatment of the three-dimensional equilibrium. Application of the well-known virial theorem shows that the addition of magnetic field to their system would require more dark matter to maintain equilibrium, not less.

The pertinent steady-state virial theorem is[2,3]

$$2(T+T_1)+M+\int_V d^3r\, x_i F_i = S \quad (1)$$

where $T=\int_V d^3r\,\rho v^2/2$ is the sum of directed kinetic energies; $T_1=\int_V d^3r\,\rho\langle u^2\rangle/2$ is the sum of thermal or random kinetic energies; $M=\int_V d^3r\,B^2/8\pi$ is the total magnetic field energy, and F_i is the force of gravity. $S=\int_S dS_k x_i[-p\delta_{ik}+M_{ik}]$ is the surface stress integral, with p the particle pressure and M_{ik} the Maxwell stress tensor.

Following Battaner et al., consider an isolated system with $S=0$. The terms T, T_1 and M in equation (1) are positive definite; only the last term on the left of the equation, the gravity integral, is negative. Regardless of the configuration or geometry, in the overall dynamical balance of a physical system, magnetic field is an expansive stress. Equation (1) shows explicitly that the only confining stress for large, isolated, astrophysical systems is gravity.

Applying equation (1) to the specific situation addressed by Battaner et al., we first consider the case in which magnetic field stresses can be neglected ($M=0$). Focusing only on the super-keplerian gas — treating it as an isolated system immersed in external gravity — it is clear that virial equilibrium requires a dynamical balance between the expansive tendency of the gas motions and the confining influence of gravity acting on that gas. The need for dark matter is already encapsulated in this analysis.

Now, with everything else unchanged, assume that the gas is magnetized. According to Battaner et al., assuming a special field configuration, the magnetic stresses should take up some of the expansive tendency of the gas motion, so that the strength of gravity needed diminishes. But, from the more general virial rela-

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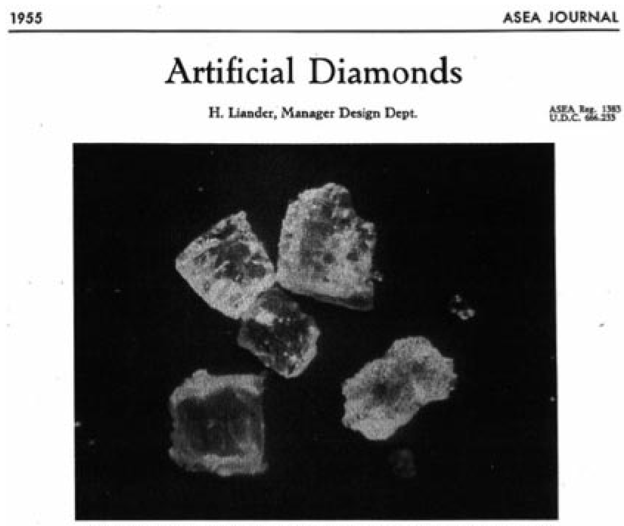
Figure 5. Message on the error made in the first attempts to synthesize diamond in the General Electric Laboratory, in which a seeding splinter of a natural diamond was taken for an artificially grown diamond.

and high-pressure equipment was disclosed. Considerably later, it was revealed that one of the allegedly synthetic diamonds shown in the photograph in [5] proved to be a natural diamond that was used as a seed [6] (Fig. 5).

The paper [5] was almost instantly translated into Russian and appeared in the December issue of the journal *Uspekhi Fizicheskikh Nauk* [7]. In that same 1955, an article was published in the almost inaccessible journal of the ASEA company, which reported on the successful synthesis of diamond in the laboratory of the company already in 1953 as a result of more than 10 years of effort [8] (Fig. 6).



1955 ASEA JOURNAL

Artificial Diamonds

H. Liander, Manager Design Dept.

A considerably magnified photograph of the first artificial diamonds produced by ASEA on the 15th February, 1953. (45702)

Figure 6. Belated communication on the synthesis of diamond in the ASEA laboratory (Sweden) that occurred in 1953.

It is obvious that this information became known both in the Division of Science of the Central Committee of the Communist Party of the Soviet Union (CC CPSU) and in the Council of Ministers; therefore, naturally, they inquired how the decree of August 28, 1947 was being fulfilled. Judging by the available materials, the 'curator' of the diamond problem in the USSR Academy of Sciences was at that time the 'Grey Cardinal' of the academy, A V Topchiev, the Chief Scientific Secretary of the USSR Academy of Sciences, who was in his time the organizer of a conference that had to rout Soviet physics but never took place. Apparently, he was 'called on the carpet,' and he in turn summoned the persons accountable to him from the Institute of Crystallography and from the already separate High-Pressure Laboratory of the USSR Academy of Sciences.

On the whole, the situation seems to have been as follows. No equipment suitable for the synthesis of diamond was obtained by the Institute of Crystallography from the High-Pressure Laboratory. Moreover, as can be judged from the list of publications by L F Vereshchagin [9], the corresponding work in the laboratory was practically not conducted. Therefore, in these circumstances, at ICAS independent work started on the creation of high-pressure equipment, and significant success was achieved. Indeed, at ICAS a double-cylinder hydraulic press was designed and constructed with a power of the cylinders of 600 and 800 tons, capable of realizing the mechanical backing for a high-pressure chamber [10]. Simultaneously, a high-pressure cylinder–piston chamber was created, in which it was possible to obtain a pressure of approximately 60 kbars sufficient for the synthesis of diamond [10, 11]. Judging by the publications, all this occurred in 1955–1959. Victory seemed to be near. However, in 1958 history decided otherwise.

The year 1958 can be called the year of the 'great turning point.' In March 1958, Kathleen Lonsdale with her colleague revealed nickel in the artificial diamonds produced in the GE Laboratory [12]. It became clear that in the synthesis of diamonds nickel served as a solvent-catalyst. Note that it was Leipunskii who had already indicated the importance of using transition metals (iron) to transform graphite into diamond [4]. However, apparently, no one remembered this or did not know about this. Indeed, Tracy Hall used troilite (iron sulfide, FeS) as a catalyst in the first successful experiments on the synthesis of diamond, based on the association of troilite and diamond in a meteorite from the Arizona crater [13].

In April 1958, Hall, who by that time had already left the GE Laboratory, published a description of several devices for obtaining high pressures, including modified Bridgman anvils and the so-called tetrahedral-anvil setup capable of generating pressures to 100,000 atm at a temperature of 3000 °C [14] (Fig. 7).

In the same article [14], Hall described the method of creating high temperatures in chambers with a solid pressure-transmitting medium with the aid of short-circuited heaters. The latter information was very essential, since, for example, in the Swedish experiments on the synthesis of diamond, the high temperature was created by means of thermit mixtures.

In July 1958, L F Vereshchagin, already the director of the Institute of High-Pressure Physics of the USSR Academy of Sciences, participated in the Gordon conference on high pressures in the USA, where Tracy Hall made a report, "High-Pressure, High-Temperature Development." Apparently, precisely as a result of this trip, Vereshchagin concluded that the game was worth the candle and that it was worth taking an active part in solving the diamond problem.

It is unknown what occurred in August and September 1958; but on October 3, 1958, an order of the Presidium of the USSR Academy of Sciences appeared about the unsatisfactory state of work on the synthesis of diamond and about the transfer of the functions of the leading organization on the problem of diamond and Borazon to the Institute of High-Pressure Physics (Fig. 8). Here, it is necessary to explain that in 1957 R G Wentorf reported the synthesis of a cubic superhard modification of boron nitride (BN) (which was called 'Borazon') at high pressures; however, no details of the synthesis were disclosed [15].

It is not completely clear why Academician A V Shubnikov, director of the Institute of Crystallography, gave up so easily. He probably lost faith in the possibility of solving the problem with the aid of his research workers. The degree of Shubnikov's frustration is illustrated well by his letter of May 6, 1959, addressed to E G Ponyatovskii, the key player in the diamond business at ICAS. In this letter, Shubnikov suggested that Ponyatovskii think about his further work at ICAS in view of the liquidation of the work on the synthesis of diamond and Borazon not agreed with L F Vereshchagin (see Fig. 9). Thus, the task of eliminating the competitors was solved, apparently, not without A V Topchiev's help. Nevertheless, it is unclear why the administration of the USSR Academy of Sciences did not want to have a parallel group competing with Vereshchagin's, as this was frequently done to solve important problems and as Vereshchagin later did himself by organizing several competing groups within one institute.

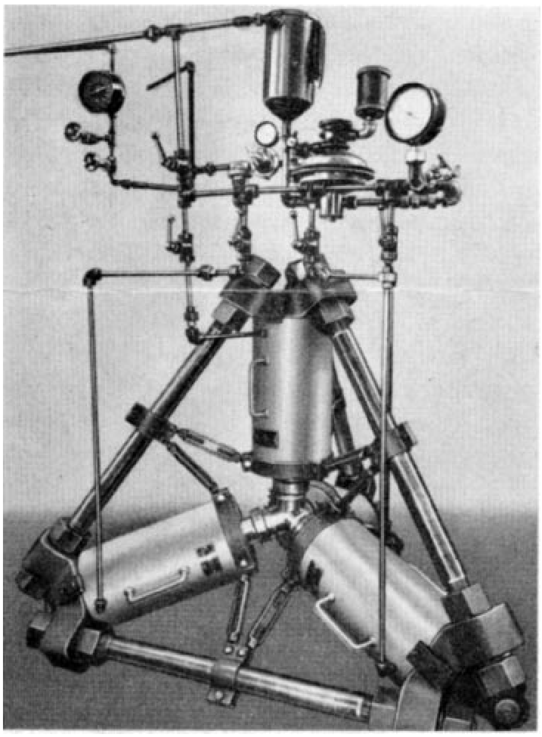
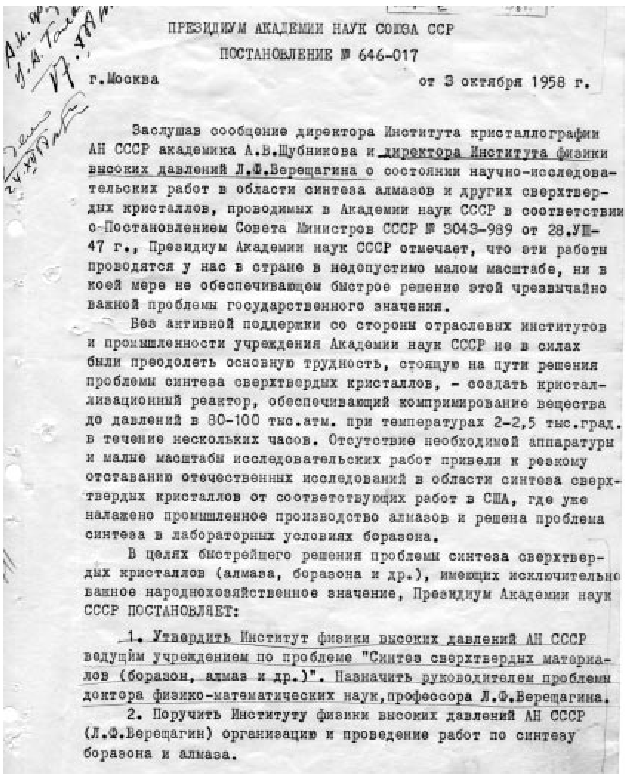


Figure 7. Hall's tetrahedral-anvil setup.



ПРЕЗИДИУМ АКАДЕМИИ НАУК СОЮЗА ССР

ПОСТАНОВЛЕНИЕ № 646-017

г.Москва от 3 октября 1958 г.

Заслушав сообщение директора Института кристаллографии АН СССР академика А.В.Шубникова и директора Института физики высоких давлений Л.Ф.Верещагина о состоянии научно-исследовательских работ в области синтеза алмазов и других сверхтвердых кристаллов, проводимых в Академии наук СССР в соответствии с Постановлением Совета Министров СССР № 3043-989 от 28.УШ-47 г., Президиум Академии наук СССР отмечает, что эти работы проводятся у нас в стране в недопустимо малом масштабе, ни в коей мере не обеспечивающем быстрое решение этой чрезвычайно важной проблемы государственного значения.

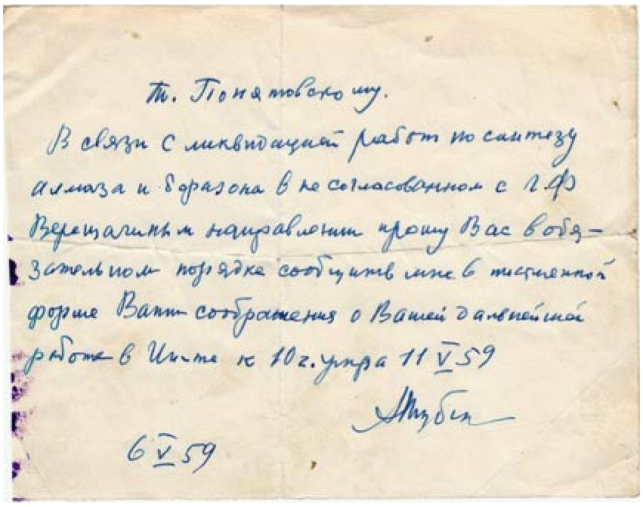
Без активной поддержки со стороны отраслевых институтов и промышленности учреждения Академии наук СССР не в силах были преодолеть основную трудность, стоящую на пути решения проблемы синтеза сверхтвердых кристаллов, - создать кристаллизационный реактор, обеспечивающий компримирование вещества до давлений в 80-100 тыс.атм. при температурах 2-2,5 тыс.град. в течение нескольких часов. Отсутствие необходимой аппаратуры и малые масштабы исследовательских работ привели к резкому отставанию отечественных исследований в области синтеза сверхтвердых кристаллов от соответствующих работ в США, где уже налажено промышленное производство алмазов и решена проблема синтеза в лабораторных условиях боразона.

В целях быстрейшего решения проблемы синтеза сверхтвердых кристаллов (алмаза, боразона и др.), имеющих исключительно важное народнохозяйственное значение, Президиум Академии наук СССР ПОСТАНОВЛЯЕТ:

1. Утвердить Институт физики высоких давлений АН СССР ведущим учреждением по проблеме "Синтез сверхтвердых материалов (боразон, алмаз и др.)". Назначить руководителем проблемы доктора физико-математических наук, профессора Л.Ф.Верещагина.

2. Поручить Институту физики высоких давлений АН СССР (Л.Ф.Верещагин) организацию и проведение работ по синтезу боразона и алмаза.

Figure 8. Order of the Presidium of the USSR Academy of Sciences dated October 3, 1958.



Т. Понятовскому.

В связи с ликвидацией работ по синтезу алмаза и боразона в не согласованном с Л.Ф. Верещагиным направлении прошу Вас в обязательном порядке сообщить мне в письменной форме Ваши соображения о Вашей дальнейшей работе в Ин-те к 10 ч. утра 11 V 59

Шубник

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Figure 9. Letter from A V Shubnikov, director of the Institute of Crystallography, to E G Ponyatovskii, one of the active members of the diamond group, with the demand to stop any work not agreed with Vereshchagin.

To solve the problem, Vereshchagin organized several groups that worked in parallel, with communication among them being categorically forbidden (naturally, all information was classified). The complete information came only to Vereshchagin. Each of the groups created its high-pressure apparatus, copying American models in one way or another. As a result, a piston-in-cylinder apparatus was created with a quasi-hydrostatic support of the piston, which was first suggested by F Boyd from the Geophysical Laboratory in Washington [16]; Hall's tetrahedral apparatus [17] was reproduced; and a simplified copy of the belt apparatus [18] was made. Simultaneously, attempts (apparently unsuccessful) were undertaken to synthesize diamonds. Nevertheless, Vereshchagin constantly sent letters about progress in the synthesis of Borazon directly to Topchiev's address, bypassing L A Artsimovich, who was at that time the academician-secretary of the USSR Academy of Sciences (see below). Here, something else is of interest.

In particular, in a letter to the Vice President of the USSR Academy of Sciences A V Topchiev dated March 19, 1959, it was asserted (Fig. 10): "BN is obtained, with a specific weight of > 3 g cm^{-3}, with a cubic structure."

And simultaneously, as if made by order (and, maybe, precisely by an order), a decree of the Council of Ministers of March 20, 1959 appeared, confirmed by a decision of the Presidium of the USSR Academy of Sciences, which obliged IHPP to synthesize crystals of Borazon in 1959 and crystals of artificial diamond in 1960-1961 (Fig. 11).

In a letter from IHPP to the CC CPSU dated March 25, 1959, it was announced that the rotation X-ray diffraction pattern of BN indicated that the BN crystal lattice had a more complex nature as compared to the American data.

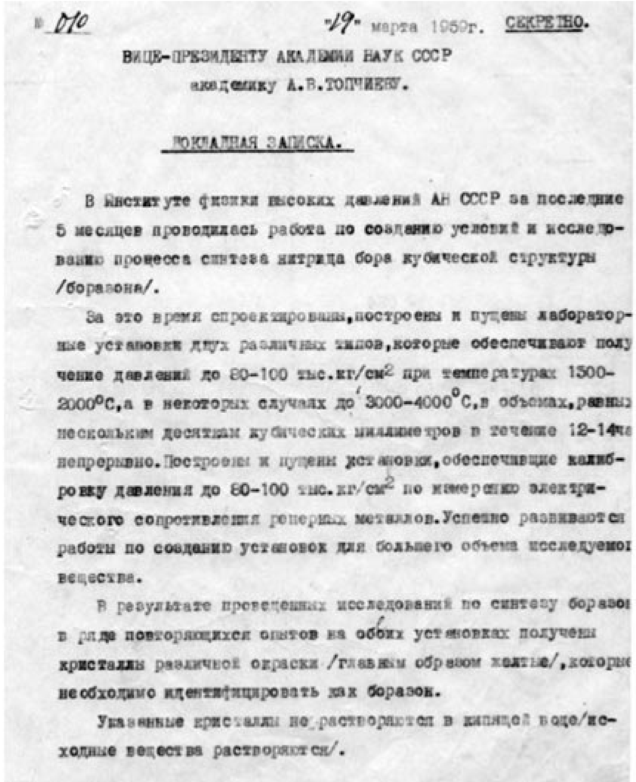
Let us present extracts from subsequent letters from IHPP to the higher authorities.

"On October 20, 1959.

BN, spec. weight > 3 g cm^{-3}, contaminated with Ni."

"On November 3, 1959.

BN, lattice parameter is greater than in the American samples, spec. weight > 3 g cm^{-3}, contaminated with Ni. In the last experiments: $a = 4.3$ Å (3.615 Å in the samples of Wentorf)."



№ 010 «19» марта 1959г. СЕКРЕТНО.

ВИЦЕ-ПРЕЗИДЕНТУ АКАДЕМИИ НАУК СССР
академику А.В.ТОПЧИЕВУ.

ДОКЛАДНАЯ ЗАПИСКА.

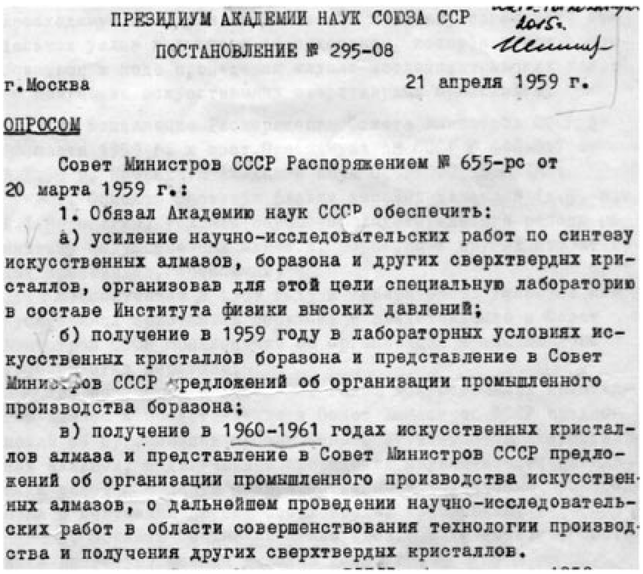
В Институте физики высоких давлений АН СССР за последние 5 месяцев проводилась работа по созданию условий и исследованию процесса синтеза нитрида бора кубической структуры /боразона/.

За это время спроектированы, построены и пущены лабораторные установки двух различных типов, которые обеспечивают получение давлений до 80-100 тыс.кг/см² при температурах 1500-2000°С, а в некоторых случаях до 3000-4000°С, в объемах, равных нескольким десяткам кубических миллиметров в течение 12-14ча[сов] непрерывно. Построены и пущены установки, обеспечивающие калибровку давления до 80-100 тыс.кг/см² по измерению электрического сопротивления реперных металлов. Успешно развиваются работы по созданию установок для большего объема исследуемо[го] вещества.

В результате проведенных исследований по синтезу боразо[на] в ряде повторяющихся опытов на обеих установках получены кристаллы различной окраски /главным образом желтые/, которые необходимо идентифицировать как боразон.

Указанные кристаллы не растворяются в кипящей воде/исходные вещества растворяются/.

Figure 10. Letter of March 19, 1959 from Vereshchagin to Topchiev with a report on supposedly prepared Borazon.



ПРЕЗИДИУМ АКАДЕМИИ НАУК СОЮЗА ССР
ПОСТАНОВЛЕНИЕ № 295-08

г.Москва 21 апреля 1959 г.

ОПРОСОМ

Совет Министров СССР Распоряжением № 655-рс от 20 марта 1959 г.:

1. Обязал Академию наук СССР обеспечить:

а) усиление научно-исследовательских работ по синтезу искусственных алмазов, боразона и других сверхтвердых кристаллов, организовав для этой цели специальную лабораторию в составе Института физики высоких давлений;

б) получение в 1959 году в лабораторных условиях искусственных кристаллов боразона и представление в Совет Министров СССР предложений об организации промышленного производства боразона;

в) получение в 1960-1961 годах искусственных кристаллов алмаза и представление в Совет Министров СССР предложений об организации промышленного производства искусственных алмазов, о дальнейшем проведении научно-исследовательских работ в области совершенствования технологии производства и получения других сверхтвердых кристаллов.

Figure 11. Order of the Presidium of the USSR AS dated April 21, 1959 obliging the IHPP to produce crystals of Borazon in 1959 and artificial crystals of diamond in 1960–1961.

"On January 6, 1960.

Cubic BN has been synthesized; contaminated with Ni."

In the report of 1960 (in fact, written later), a value $a = 3.61 \pm 0.02$ Å is already given without any comments.

Strange and anecdotal assertions are contained in these letters. The lattice parameter of the material obtained is much greater than that of Borazon; furthermore, the material contains nickel. It is amusing that a nickel-containing material with a lattice parameter that is considerably greater than that of Borazon is obtained, but nevertheless it is asserted that it is Borazon that is obtained. And all these communications were accepted by A V Topchiev, the vice president of the USSR Academy of Sciences, who was a chemist, and by Head of the Division of Science of the CC CPSU V A Kirillin, a power engineer, who was then a corresponding member of the USSR Academy of Sciences.

The situation in reality was very simple. Since the details of the synthesis of Borazon in Wentorf's first article were not indicated [15], someone decided that if nickel worked as a catalyst in synthesising diamond, why can it not be used for the synthesis of Borazon as well. Apparently, the real Borazon was obtained at IHPP no earlier than March 1961, after Wentorf published the technology of synthesis that included the use of alkaline and alkaline-earth metals as catalysts [19]. However, judging by information on the website of the Kiev Institute of Superhard Materials, in reality, Borazon was obtained only in 1964.

It is remarkable that Vereshchagin had some samples of real 'American' Borazon, although this material was not, to put it mildly, easily accessible. It is possible that it was precisely this circumstance that gave the participants in the 'enterprise' confidence in the happy outcome. This entire affair with allegedly produced Borazon played the role of a smoke screen and made it possible to gain the necessary time. In October 1959, in *Nature* an article appeared (signed by the same team from the GE Research Laboratory) under the promising title "Preparation of Diamond," in which the conditions of diamond synthesis were described in sufficient detail [20] (Figs 12, 13).

This undoubtedly played its role, and already on March 25, 1960 Vereshchagin reported at the Presidium of the USSR Academy of Sciences the results of successful experiments on the synthesis of Borazon (although 'contaminated with nickel') and of diamond. In the order from the Presidium, it was written precisely: "crystals: contaminated with nickel"! It is surprising that in this resolution diamond, which was 'the holy grail' of the entire enterprise, is mentioned briefly and only in the second place after the monstrous untruth concerning the borazon (Fig. 14)!

Nevertheless, in 1960, diamonds were obtained at IHPP. However, it is still unknown who was the first to make it and when it was made; judging by the report (which was classified at that time) prepared in 1960, possibly the first successful synthesis of diamond was carried out on March 17, 1960 (Fig. 15).

Were all these circumstances expenses or advantages of the prohibition on communications between colleagues–participants in this 'diamond race'? Only Vereshchagin knew everything. It is supposedly known that the successful synthesis of diamond was performed not with the aid of some monstrous apparatuses, but using a modest device, which was a modification of Bridgman anvils. This device, in view of the form of its working volume, was called a 'lentil' and became the basis of the industrial production of diamonds in the USSR (Fig. 16). However, in contrast to descriptions of the

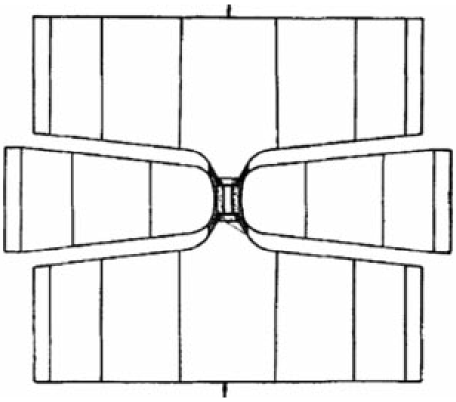
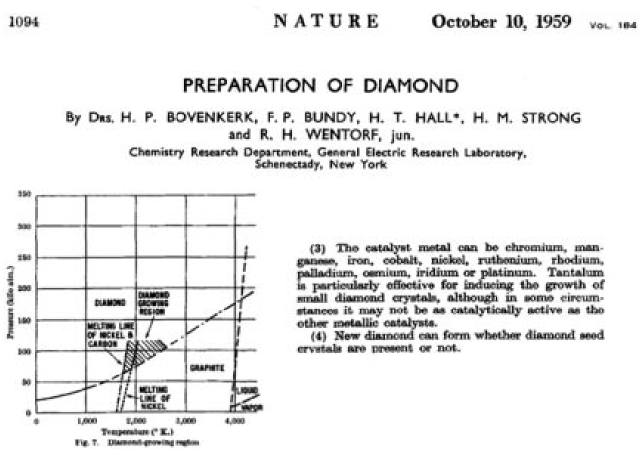


Figure 13. Diagram of the belt-type high-pressure apparatus used in the General Electric Research Laboratory for the synthesis of diamond. The apparatus consisted of two conic pistons and a shaped matrix made of a hard alloy and supported by steel rings.



1094 NATURE October 10, 1959 VOL. 184

PREPARATION OF DIAMOND

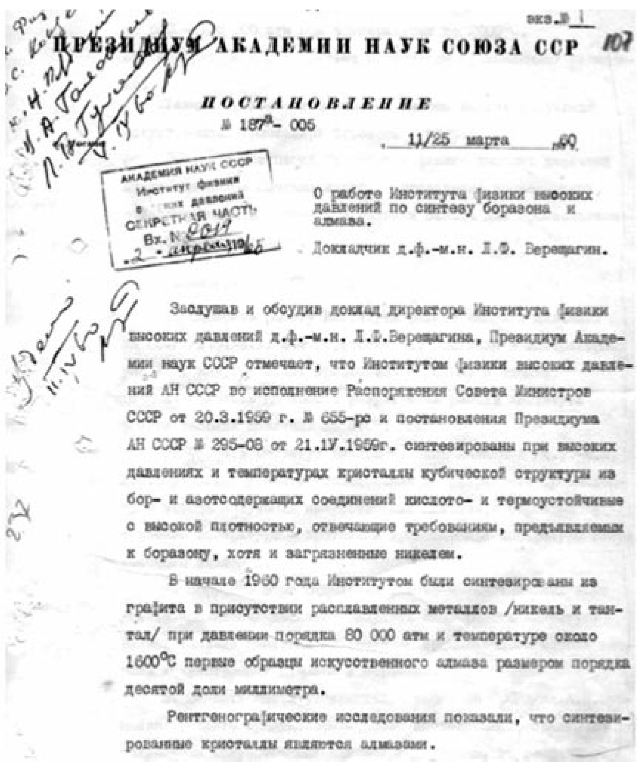
By Drs. H. P. BOVENKERK, F. P. BUNDY, H. T. HALL*, H. M. STRONG and R. H. WENTORF, jun.

Chemistry Research Department, General Electric Research Laboratory, Schenectady, New York

(3) The catalyst metal can be chromium, manganese, iron, cobalt, nickel, ruthenium, rhodium, palladium, osmium, iridium or platinum. Tantalum is particularly effective for inducing the growth of small diamond crystals, although in some circumstances it may not be as catalytically active as the other metallic catalysts.

(4) New diamond can form whether diamond seed crystals are present or not.

Figure 12. Description of conditions for the synthesis of diamond published in 1959 by the team of the General Electric Research Laboratory [20].



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ПРЕЗИДИУМ АКАДЕМИИ НАУК СОЮЗА ССР 107

ПОСТАНОВЛЕНИЕ

№ 187а-005

11/25 марта 60

АКАДЕМИЯ НАУК СССР Институт физики высоких давлений СЕКРЕТНАЯ ЧАСТЬ Вх. № 2019

О работе Института физики высоких давлений по синтезу боразона и алмаза.

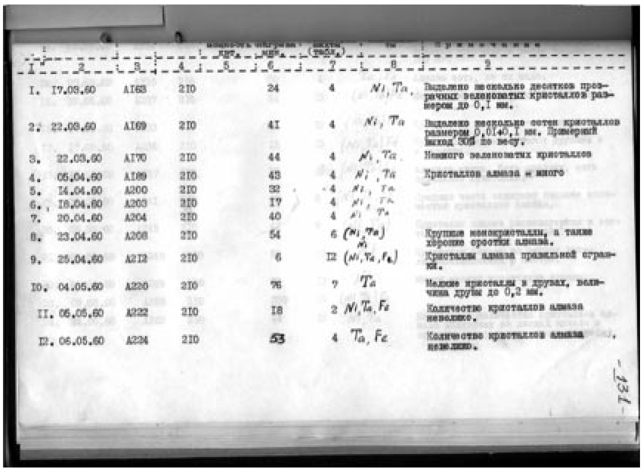
Докладчик д.ф.-м.н. Л.Ф. Верещагин.

Заслушав и обсудив доклад директора Института физики высоких давлений д.ф.-м.н. Л.Ф.Верещагина, Президиум Академии наук СССР отмечает, что Институтом физики высоких давлений АН СССР во исполнение Распоряжения Совета Министров СССР от 20.3.1959 г. № 655-рс и постановления Президиума АН СССР № 295-08 от 21.IУ.1959г. синтезированы при высоких давлениях и температурах кристаллы кубической структуры из бор- и азотсодержащих соединений кислото- и термоустойчивые с высокой плотностью, отвечающие требованиям, предъявляемым к боразону, хотя и загрязненные никелем.

В начале 1960 года Институтом были синтезированы из графита в присутствии расплавленных металлов /никель и тантал/ при давлении порядка 80 000 атм и температуре около 1600°С первые образцы искусственного алмаза размером порядка десятой доли миллиметра.

Рентгенографические исследования показали, что синтезированные кристаллы являются алмазами.

Figure 14. Order of the Presidium of the USSR Academy of Sciences dated March 25, 1960 announcing a successful synthesis of 'Borazon' (contaminated with nickel) and of diamond.



1	2	3	4	5	6	7	8	9
1.	17.03.60	A163	210		24	4	Ni, Ta	Выделено несколько десятков прозрачных зеленоватых кристаллов размером до 0,1 мм.
2.	22.03.60	A169	210		41	4	Ni, Ta	Выделено несколько сотен кристаллов размером 0,01÷0,1 мм. Примерный выход 30% по весу.
3.	22.03.60	A170	210		44	4	Ni, Ta	Немного зеленоватых кристаллов
4.	05.04.60	A189	210		43	4	Ni, Ta	Кристаллов алмаза много
5.	14.04.60	A200	210		32	4	Ni, Ta	
6.	18.04.60	A203	210		17	4	Ni, Ta	
7.	20.04.60	A204	210		40	4	Ni, Ta	
8.	23.04.60	A208	210		54	6	(Ni, Ta)	Крупные монокристаллы, а также хорошие сростки алмаза.
9.	25.04.60	A212	210		6	12	(Ni, Ta, Fe)	Кристаллы алмаза правильной огранки.
10.	04.05.60	A220	210		76	7	Ta	Мелкие кристаллы в друзах, величина друзы до 0,2 мм.
11.	06.05.60	A222	210		18	2	Ni, Ta, Fe	Количество кристаллов алмаза невелико.
12.	06.05.60	A224	210		53	4	Ta, Fe	Количество кристаллов алмаза невелико.

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Figure 15. Extract from the final report of IHPP on the diamond problem.

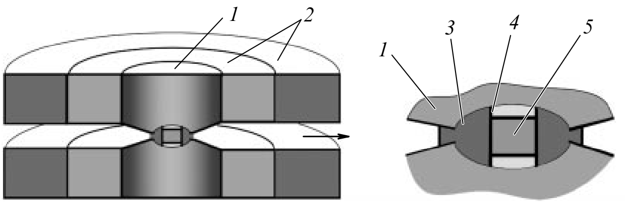

Figure 16. High-pressure 'lentil' chamber designed at IHPP: (1) shaped pistons made of a hard alloy; (2) steel supporting rings; (3) lithographic stone (pressure-transmitting medium); (4) heater; and (5) sample.

American samples given in [16–18], the description of the 'lentil' chamber which became known to everyone everywhere, was never officially published, and its real authors obtained neither fame, nor recognition. (Many years later, an original description of the 'lentil' chamber, with L F Vereshchagin, V N Slesarev, and V E Ivanov as the authors, was published only in the Sbornik Trudov (Collection of Papers) of IHPP in 1998). However, awards followed. Already in 1960, L F Vereshchagin was elected as a corresponding member of the USSR Academy of Sciences; then, in 1961, L F Vereshchagin, V A Galaktionov, and Yu N Ryabinin were awarded the Lenin prize "for studies in the physics of high pressures." In 1963, L F Vereshchagin became a Hero of Socialist Labor, and in 1966 he was elected a full member of the USSR Academy of Sciences.

It is interesting that Tracy Hall, the creator of the belt chamber and the author of the first successful synthesis of diamond in the USA, did not receive due recognition and left the Research Laboratory of the General Electric Company.

Such is the real, although, possibly, incomplete history of synthesis of artificial diamonds in the USSR. The history would be not worth describing, if it did not reflect the specific mechanisms of the functioning of Soviet science, which was determined by the control link chain: Central Committee of the CPSU–Council of Ministers–Gosplan (State Planning Commission)–State Scientific and Technical Committee of the USSR–military-industrial complex — and, finally, the USSR Academy of Sciences and applied-research institutes. Outside this chain, it was not possible to implement any expensive project and (or) a project which promised significant moral and material dividends. It seems that the team at the Institute of Crystallography did not possess the administrative resources necessary to successfully complete the project on the creation of artificial diamond initiated by the decree of the Council of Ministers of the USSR in 1947.

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