

Министерство науки и высшего образования Российской Федерации
 федеральное государственное автономное
 образовательное учреждение высшего образования
 «Национальный исследовательский Томский политехнический университет» (ТПУ)

Инженерная школа новых производственных технологий
 Направление подготовки (специальность) Компьютерное моделирование получения, обработка и переработка материалов
 Отделение школы (НОЦ) Материаловедения

МАГИСТЕРСКАЯ ДИССЕРТАЦИЯ

Тема работы
Исследование физико-механических свойств алмазных покрытий
УДК 621.793. 7-419.8-032.81

Студент

Группа	ФИО	Подпись	Дата
4ВМ7Е	Кунашенко Сергей Евгеньевич		

Руководитель

Должность	ФИО	Ученая степень, звание	Подпись	Дата
Старший научный сотрудник	Линник Степан Андреевич	Кандидат технических наук		

КОНСУЛЬТАНТЫ:

По разделу «Финансовый менеджмент, ресурсоэффективность и ресурсосбережение»

Должность	ФИО	Ученая степень, звание	Подпись	Дата
Доцент	Скаковская Наталья Вячеславовна	Кандидат технических наук		

По разделу «Социальная ответственность»

Должность	ФИО	Ученая степень, звание	Подпись	Дата
Доцент	Горбенко Михаил Владимирович	Кандидат технических наук		

ДОПУСТИТЬ К ЗАЩИТЕ:

Руководитель ООП	ФИО	Ученая степень, звание	Подпись	Дата
22.04.01 Материаловедение и технология материалов	Овечкин Борис Борисович	Кандидат технических наук		

Томск – 2019 г.

Планируемые результаты обучения по ООП 22.04.01 Материаловедение и технологии материалов

Код результата	Результат обучения (выпускник должен быть готов)
P1	Осуществлять сбор, анализ и обобщение научно-технической информации в области материаловедения и технологии материалов с использованием современных информационно-коммуникационных технологий, глобальных информационных ресурсов
P2	Работать с патентным законодательством и авторским правом при подготовке документов к патентованию и оформлению ноу-хау
P3	Выполнять маркетинговые исследования и анализировать технологический процесс как объекта управления, разрабатывать технико-экономическое обоснование инновационных решений в профессиональной деятельности
P4	Руководить коллективом в сфере своей профессиональной деятельности, толерантно воспринимая социальные, этнические, конфессиональные и культурные различия
P5	Внедрять в производство технологии получения керамических, металлических материалов и изделий, в том числе наноматериалов, быть готовым к профессиональной эксплуатации современного оборудования и приборов, позволяющих получать и диагностировать материалы и изделия различного назначения.
P6	Разрабатывать новые и модернизировать существующие технологии получения керамических, металлических материалов и изделий, в том числе наноматериалов
P7	Внедрять системы управления качеством продукции в области материаловедения, эксплуатировать оборудование, позволяющее диагностировать материалы и изделия из них, в том числе наноматериалы
P8	Действовать в нестандартных ситуациях, нести социальную и этическую ответственность за принятые решения, выбирать наиболее рациональные способы защиты и порядка в действиях малого коллектива в чрезвычайных ситуациях
P9	Общаться в устной и письменной формах на государственном языке РФ и иностранном языке для решения задач профессиональной деятельности, подготавливать и представлять презентации планов и результатов собственной и командной деятельности, формировать и отстаивать собственные суждения и научные позиции
P10	Самостоятельно осваивать новые методы исследования, изменять научный, научно-педагогический и производственный профиль своей профессиональной деятельности

Министерство науки и высшего образования Российской Федерации
 федеральное государственное автономное
 образовательное учреждение высшего образования
 «Национальный исследовательский Томский политехнический университет» (ТПУ)

Инженерная школа новых производственных технологий
 Направление подготовки (специальность) Компьютерное моделирование получения, обработка и переработка материалов
 Отделение школы (НОЦ) Материаловедения

УТВЕРЖДАЮ:
 Руководитель ООП
 _____ Б.Б Овечкин
 (Подпись) (Дата)

ЗАДАНИЕ
на выполнение выпускной квалификационной работы

В форме:

Магистерской диссертации

(бакалаврской работы, дипломного проекта/работы, магистерской диссертации)

Студенту:

Группа	ФИО
4БМ7И	Куншенко Сергей Евгеньевич

Тема работы:

Исследование физико-механических свойств алмазных покрытий	
Утверждена приказом директора ИШНПТ	Приказ № _____ от _____

Срок сдачи студентом выполненной работы:	13.06.2019
--	------------

ТЕХНИЧЕСКОЕ ЗАДАНИЕ:

Исходные данные к работе	Синтезировать алмазные пленки с различной долей алмазной фазы в HFCVD реакторе
---------------------------------	--

<p>Перечень подлежащих исследованию, проектированию и разработке вопросов</p>	<ol style="list-style-type: none"> 1. Аналитический обзор литературы 2. Синтезирование алмазных пленок при разной концентрации метана 3. Оценка фазовой чистоты и микроструктуры алмазных пленок с помощью рентгеноструктурного анализа 4. Определение плотности тонких пленок алмаза 5. Определение твердости алмазных пленок, синтезированных в различных условиях 6. Исследования трибологических свойств алмазных пленок
<p>Перечень графического материала <i>(с точным указанием обязательных чертежей)</i></p>	<p>Демонстрационный материал (презентация в MS PowerPoint)</p>

<p>Консультанты по разделам выпускной квалификационной работы <i>(с указанием разделов)</i></p>	
<p>Раздел</p>	<p>Консультант</p>
<p><i>Финансовый менеджмент...</i></p>	<p>Скаковская Наталья Вячеславовна, доцент, ТПУ</p>
<p><i>Социальная ответственность</i></p>	<p>Горбенко Михаил Владимирович, доцент, ТПУ</p>
<p>Названия разделов, которые должны быть написаны на иностранном языке:</p>	
<p>Introduction</p>	
<p>Review</p>	
<p>Materials and methods</p>	
<p>Practical part</p>	

<p>Дата выдачи задания на выполнение выпускной квалификационной работы по линейному графику</p>	
--	--

Задание выдал руководитель:

Должность	ФИО	Ученая степень, звание	Подпись	Дата
Старший научный сотрудник	Линник Степан Андреевич	Кандидат технических наук		

Задание принял к исполнению студент:

Группа	ФИО	Подпись	Дата

4БМ7И	Кунашенко Сергей Евгеньевич		
-------	-----------------------------	--	--

Abstract

The final qualifying work is presented on 85 pages, including 39 figures, 13 tables, 38 literature sources.

Keywords: Diamond, diamond films, diamond coating, CVD, HFCVD reactor, hardness, density, wear rate, MCD, UNCD, microstructure, X-ray

The object of the research are: Diamond films synthesized with different proportions of the diamond phase in the HFCVD reactor

The work purpose - Research of physical and mechanical properties of diamond coatings

The following methods were used in the research: X-ray phase analysis, transmission electron microscopy, CVD deposition

As a result of the research: The physico-mechanical properties of diamond films synthesized at different CH₄ concentrations were obtained and investigated.

Degree of implementation: Research work

Applications: Polycrystalline diamond is currently used as hardening and wear-resistant coatings on cutting tools, output windows of high-power lasers, highly efficient heat sinks for power electronics, corrosion-resistant semiconductor electrochemical electrodes, etc

Economic efficiency / importance of the work: The use of diamond coatings will significantly increase the physico-mechanical characteristics of cutting tools.

In the future, we plan to research: The physico-mechanical properties of multilayer diamond coatings.

Content

Introduction.....	9
1 Literature review	11
1.1 Diamond.....	11
1.2 The history of diamond synthesis	12
1.3 Synthetic Diamond	12
1.4 High Pressure High Temperature (HPHT) Diamond	14
1.5 Low Pressure Low Temperature CVD Diamond Synthesis.....	15
1.6 Diamond Synthesis.....	15
1.7 Hot Filament Vapor Deposition.....	17
1.9 Types of diamond films.....	19
1.9.1 Microcrystalline diamond (MCD)	20
1.9.2 Nanocrystalline diamond (NCD)	21
1.9.3 Ultrananocrystalline diamond (UNCD)	22
1.10 Application areas for CVD diamond films	22
2 Materials and methods.....	24
2.1 Surface preparation for diamond plating.....	25
2.1.1- Surface preparation	25
2.1.2 Surface cleaning.....	26
2.1.3 Etching and Sowing	28
2.2 Deposition of CVD coating on the surface of the material	28
2.3 Determination of thin film density using ImageJ.....	32
2.4 Wear rate	34
2.5 Measurement of the microhardness of diamond coatings	36
2.6 Raman spectroscopy.....	38
3 Practical part	40
3.1 The XRD patterns of diamond films.....	42
3.2 Raman analyses.....	42
3.3 Nanoindentation measurements.....	44
3.4 Measurement of the density of diamond films	45
3.5 Tribological tests.....	47

4. Financial management, resource efficiency and resource saving	54
4.1 Analysis of competitive technical solutions.....	54
4.2 Planning the study of the physico-mechanical properties of diamond coatings	56
4.3 Determination of the complexity of the work	56
4.4 Development of the project schedule.....	57
4.5 Design Cost Budget.....	61
4.6 Formation of costs for the project	65
5 Social responsibility	68
5.1 Legal and organizational security issues.....	69
5.2 Professional and social security	70
5.2 Ecological safety	79
5.3 Organizational security arrangements.....	80
5.4 Features of legislative regulation of design solutions.....	81
5.5 Safety in emergencies.....	82
Conclusion	83
References.....	84

Introduction

Currently, interest in the field of application of various materials in semiconductors of electronics is actively growing. One of the promising materials is diamond. This material differs from others in a large number of parameters that are extreme and necessary for semiconductor electronics, for example, the highest thermal conductivity of all solids ($900\text{-}2300 \text{ W / m} \times \text{K}$), chemical and radiation resistance, as well as wide bandwidth of optical radiation. From the depths of ultraviolet to far infrared. Pure diamond is a good electrical insulator (resistivity is $\sim 10^{16} \text{ Ohm} \times \text{cm}$ at room temperature), when doping diamond its resistivity can vary over a wide range from 10 to 10^6 ($\text{Ohm} \times \text{cm}$), which turns it into a wide-gap semiconductor with a forbidden width zones 5.4 eV .

The coefficient of thermal expansion (CTE) of diamond is one of the lowest, and at room temperature, it can compare with Invar CTD ($0.8 \times 10^{-6} \text{ K}$). Despite these advantages, there are many technological problems in the massive use of diamonds. It is difficult and nontechnological to create electronic structures with a complex geometry based on them, especially with dimensions applicable in micro- and nanoelectronics.

The use of high purity natural diamond is very expensive and time consuming.

Therefore, the use of diamond in the form of polycrystalline films that can deposit on homo- and heterosubstrates appears to be very promising.

The use of synthetic diamond allows us to simplify the process and reduce its cost, but for its production diamond, substrates of high purity are required. Further processing is very difficult, because the use of conventional methods of etching, grinding, etc. is inefficient due to the resistance of diamond to various influences.

Today, diamond know as a material with an exceptional combination of unique properties. Extreme values of hardness, thermal conductivity, transparency, chemical inertness and many other properties make diamond extremely attractive for use in almost any branch of science and technology. However, such factors as its

extremely high cost, difficulty of processing and small size, seriously limit the scope of application of single-crystal diamond, despite the development of the method of synthesis at high pressures and temperatures (HPHT).

The development of gas-phase deposition technology (CVD) made it possible to create not only single crystals, but also polycrystalline diamond in the form of films and plates.

Due to this, polycrystalline diamond is currently used as hardening and wear-resistant coatings on cutting tools, output windows of high-power lasers, highly efficient heat sinks for power electronics, corrosion-resistant semiconductor electrochemical electrodes, etc.

1 Literature review

1.1 Diamond

Humans have revered diamond since ancient times mainly as gemstone. Its scarcity, hardness and bright sparkle makes it a status symbol. Apart from being the much sought after gemstone, the many remarkable properties of diamond have similar appeal to material scientists and engineers. Among the properties are hardest known material, chemical inertness, and highest thermal conductivity at room temperature, least compressible and highest stiffness. When doped, diamond exhibit semiconducting properties with a larger band gap of 5.4 eV, which can be useful for the fabrication of microelectronics devices for high frequency and high power applications. Diamond, which is carbon in the most concentrated form, involves sp^3 hybridization of the valence electrons in carbon to form chemical bonds. In diamond, each carbon atom shares all four of its available electrons with adjacent carbon atoms, forming tetrahedral unit, with 1.54 Å in length [1]. This shared electron-pair bonding forms the strongest known chemical linkage, sp^3 hybridized electrons forming the covalent bond, which is responsible for the many superlative properties of diamond. This bond form equal angles of $109^\circ 28'$ separating each carbon atoms. The repeating structural unit of diamond consists of eight atoms, which are fundamentally arranged, in a cube [2] as shown in Figure 1.1.

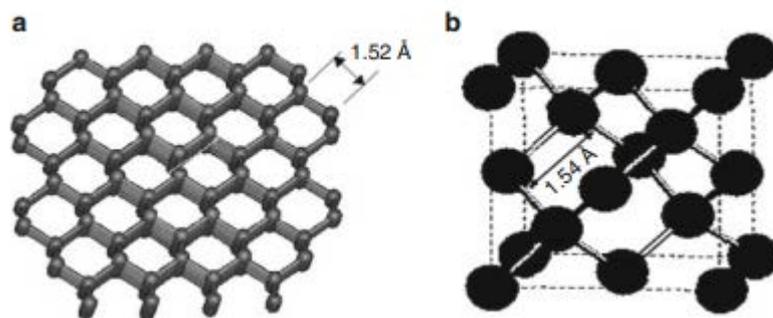


Figure 1.1 (a) Diamond lattice (b) Face-centered cubic structure of diamond [2]

With this cubic form and its highly symmetrical arrangement of atoms, diamond crystals can develop in a variety of different shapes known as ‘crystal

habits'. The octahedron shape is the most common crystal habit. However, diamond crystal can also form cubes, dodecahedra, and even combinations of these shapes.

1.2 The history of diamond synthesis

Many statements about the possibility of diamond deposition were discussed between 1879 and 1928; most of these allegations were carefully analyzed, but none of them was ever confirmed. In the 40s of the 20th century, the first tests of diamond coating deposition were carried out.

For the first time, reproducible synthesis was performed in 1953: the Swedish scientist Balthazar Platen constructed an installation in which a cube sample was compressed with six pistons from different sides. In 1953, the world's first artificial diamonds were obtained.

Systematic research has been conducted on the cultivation of diamonds using CVD and HPHT methods. These two methods still dominate the production of synthetic diamonds.

To date, several technologies use for the production of artificial diamonds. Historically, the first, and the main today due to the relatively low cost - the use of high pressure and high temperature (high-pressure high temperature - HPHT). The equipment for this method is multi-ton presses that can develop pressures up to 5 GPa at 1500 ° C. The second method is chemical vapor deposition (chemical vapor deposition (CVD)), when a plasma of carbon atoms is created above the substrate, from which atoms gradually condense to the surface to form a diamond. The third method uses the formation of nanoscale diamonds using a shock wave from explosives [3].

1.3 Synthetic Diamond

Pressure High Temperature (HPHT) technique, which duplicates the condition of natural diamond formation [1]. Using the HPHT technique synthetic diamond can be converted directly from graphite under specific conditions. At present, synthetic diamond formed using the high pressure high temperature

technique is a major source of industrial diamond, mainly as abrasives and drill bits [4]. Alternatively, the Chemical Vapour Deposition (CVD) technique is the method to synthesis diamond at low pressure and low temperature conditions from gas phase precursors. The main attraction of CVD diamond is its film form where new potential applications such as coatings or optical mirrors are possible. The possibility of CVD diamond synthesis was first proposed in 1955, when Bundy et al. [5] presented the carbon phase diagram as illustrated in Figure 1.2. The diagram shows the thermodynamic stability for graphite and diamond at different temperatures and pressures. The position of the diamond/graphite equilibrium line was established by thermodynamic calculations based upon the measured physical properties of graphite and diamond in the temperature range from 300 to about 1,200 K and by experiments on growing or graphitization of diamond. It has been proposed that a temperature, pressure phase diagram for carbon which included a wide temperature, pressure area at lower pressures and temperatures in which there would be in apparent equilibrium state or ‘pseudo-equilibrium’ between diamond and graphite [6]. The quantitative of free energies of graphite and diamond determination by Rossini et al. showed that their difference at one atmosphere, 0 K, is only about 2.5 KJ mol⁻¹ [7].

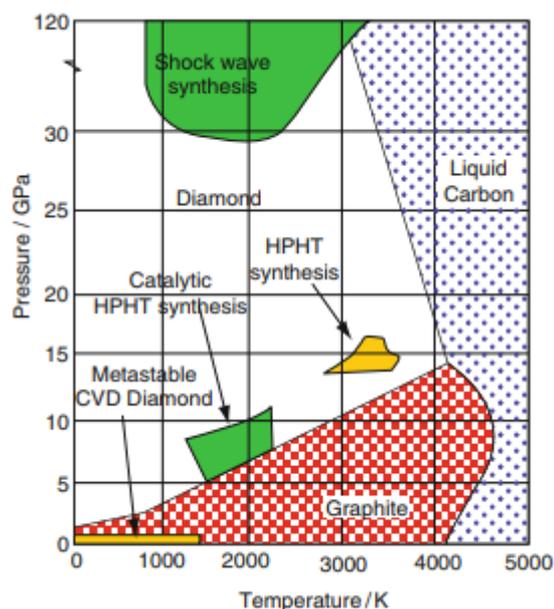


Figure 1.2 Phase diagram of carbon [5] – gives a short description of how the phasediagram was constructed from experimental data and calculations

1.4 High Pressure High Temperature (HPHT) Diamond

The carbon phase diagram illustrated in Figure 1.2 shows that diamond is unstable with respect to graphite except at high temperatures ($>1,300\text{C}$) and high pressures (>40 kbar). In 1955, H. T. Hall and co-workers at General Electric developed the solventcatalytic HPHT method [5, 8]. In this procedure, graphite was compressed in a hydraulic press in the presence of a suitable molten metal catalyst/solvent until carbon dissolved and diamond crystallized. Sufficiently high temperature was provided by passing an electric current through the sample. Molten metals like Ni, Co or Fe are commonly used which dissolve graphite at elevated pressures of between 5 and 10 GPa, and temperatures in the range of 1,300–2,300C. The dissolved carbon re-crystallizes into diamond at normal pressures and temperatures. The transition of graphite to diamond is accompanied by volume decrease of 43%, posing severe technical problems to maintain suitable pressure, being a vital condition [9]. If the pressure is not maintained, the formed diamond would convert itself back to graphite immediately. Synthetic diamond crystals with sizes ranging from few nanometres to millimetres were recovered from the process by dissolving the metal catalysts in acid. There are applications of HPHT diamond materials. Diamond grit are used as cutting tool in industries and the main bulk of diamond used here are synthetic more viable than the use of natural diamond. Russell Hemley at Carnegie institution had found that most HPHT synthetic diamond is yellow and most CVD diamond is either milky white or greyish, limiting their optical applications. Colourless diamond are costly to produce using the HPHT method and this situation had limits the general applications of these diamonds as gems, in optics, and in scientific research. CVD diamond, which is grown under non-thermal equilibrium conditions, can be stable even at low pressure [10]. CVD diamond which can be produced in film form at this condition has similar properties as natural diamond and therefore provided tremendous prospective applications in industry

1.5 Low Pressure Low Temperature CVD Diamond Synthesis

The notion of growing diamond from gas at pressures lower than atmospheric and temperatures lower than 1,000°C may seem counter intuitive, but the mechanism involved is totally alien of the HPHT process. The first reported success of low-pressure low temperature was reported by Eversole in 1962, whereby the optimal temperature and pressures were 900°C and 0.1 Torr respectively [11].

In 1968, Angus [12] showed that synthetic diamonds were identical with natural diamond and later showed that semiconducting diamond can be obtained by doping diamond grown from methane and hydrogen with boron [22, 23]. Japanese scientists at the National Institute for Research in Inorganic Materials (NIRIM) in Japan developed various diamond thin film deposition techniques such as CVD with the activation of carbon-containing gases, physical vapor deposition (PVD) from a solid carbon source and hybrid processes involving solids, carbon and catalytic materials and gases as the sources. These were done both on diamond or nondiamond substrates. Generation of atomic hydrogen near deposition surface was found to be the major common factor in diamond growth [11].

1.6 Diamond Synthesis

Chemical vapor deposition (CVD) is a scientific term for the process of growing thin films, first developed over forty years ago. In general terms, this technology is the formation of a substance from a hot reaction gas mixture, which condenses on a controlled surface (substrate). Unlike the synthesis of crystals at high pressures and temperatures, the CVD method is mainly, though not always, used at pressures below atmospheric and is used to grow coatings of a few microns thick on a surface of a few square centimeters. The development of diamond technology by CVD over the past decade has led to the ability to grow films in millimeters thick and without a substrate and over a larger area, which is most often used to grow high-purity structures of semiconductor devices. At present, CVD technology is often used in industry for growing high-purity polycrystalline diamond raw materials, which were previously synthesized only as thin films and only in

laboratory research processes. Element Six has adapted and developed this technology for the production of diamond films.

Today, for gas-phase synthesis of diamond, various schemes are used in which the initial gaseous medium contains oxygen, carbon dioxide and other elements in addition to carbon. However, the use of the carbon-hydrogen system is the most common. As a rule, a mixture of molecular hydrogen and methane is used, whose concentration in hydrogen is of the order of (0.5–10) %.

Figure 1.3 shows a schematic diagram of the gas-phase synthesis of diamond. For effective deposition, the gas mixture must be strongly activated, since hydrogen molecules have great strength. This operation has two main objectives: 1. The dissociation of hydrocarbon molecules into fragments that actively react with the surface of the substrate. 2. Dissociation of molecular hydrogen to create a super-equilibrium concentration of hydrogen atoms above the substrate.

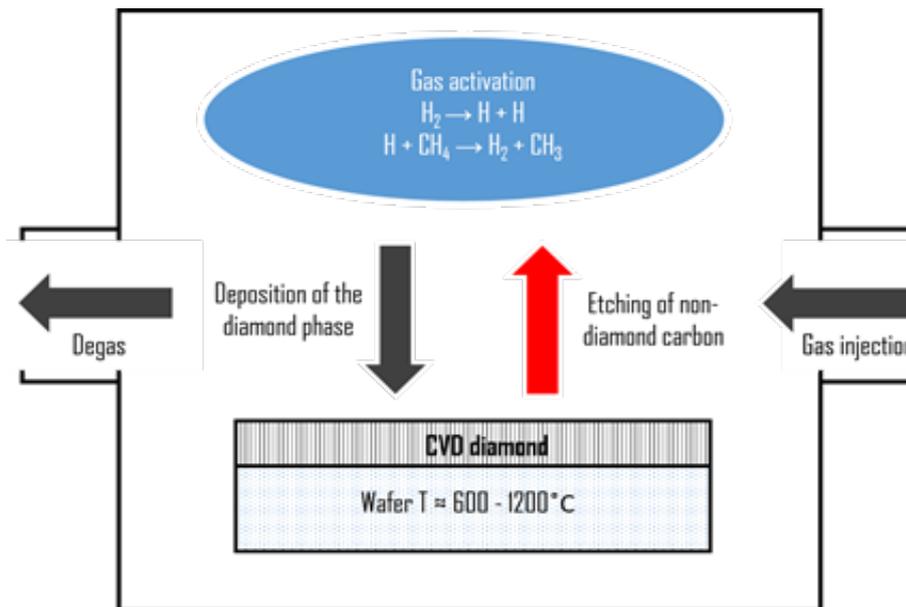


Figure 1.3 - Diagram of the process of deposition of diamond from the gas phase

The resulting active radicals, due to convection, diffusion and gas flow move to the substrate. Above the substrate there is a diffusion layer through which the particles must pass to the surface. Various processes occur on the surface of the substrate, such as: absorption and desorption, surface and bulk diffusion of active particles, chemical reactions promoting diamond growth or slowing it down. All

these processes largely depend on the temperature of the substrate, its material and structure. As the substrate, as noted, poly- and single-crystal diamond can be used, as well as a very wide range of materials (Si, W, Ti, Mo, Ni, etc.).

The main conditions for diamond deposition are high substrate temperature and gas pressure. The substrate temperature varies from 600 to 1200⁰ C. With a decrease in temperature, the growth rate of diamond is greatly reduced. In order to obtain the maximum growth rate of diamond, the substrate temperature must be in the temperature range from 1000 to 1100⁰ C. Most chemical processes during diamond growth occur on the surface of a solid, and therefore with increasing temperature the rate of these processes increases. [13]

The gas pressure at which diamond precipitates can vary from a few tenths of Torr [14] to the atmosphere [15], depending on the type of activator, the composition of the gas mixture, the area of deposition, etc. The most common is precipitation in the range from 20 to 200 Torr. Deposition at low pressures is characterized by a low growth rate due to the low concentration of molecules. At the same time, for sufficient gas activation at pressures close to atmospheric pressure, very high energy density is required. Therefore, although in such systems the growth rate can be extremely high, the deposition area is at best only a few square centimeters. Therefore, for each type of reactor, there is an optimal pressure range at which deposition proceeds with the greatest efficiency.

1.7 Hot Filament Vapor Deposition

One of the simplest, cheapest and most versatile methods of diamond deposition using the CVD method can be called the hot thread method. As an activator of the gas mixture in this method, electrically heated filaments made of refractory metals (W, Mo, Ta) are used. Figure 1.4 shows common reactor layouts for diamond deposition using the hot thread method.

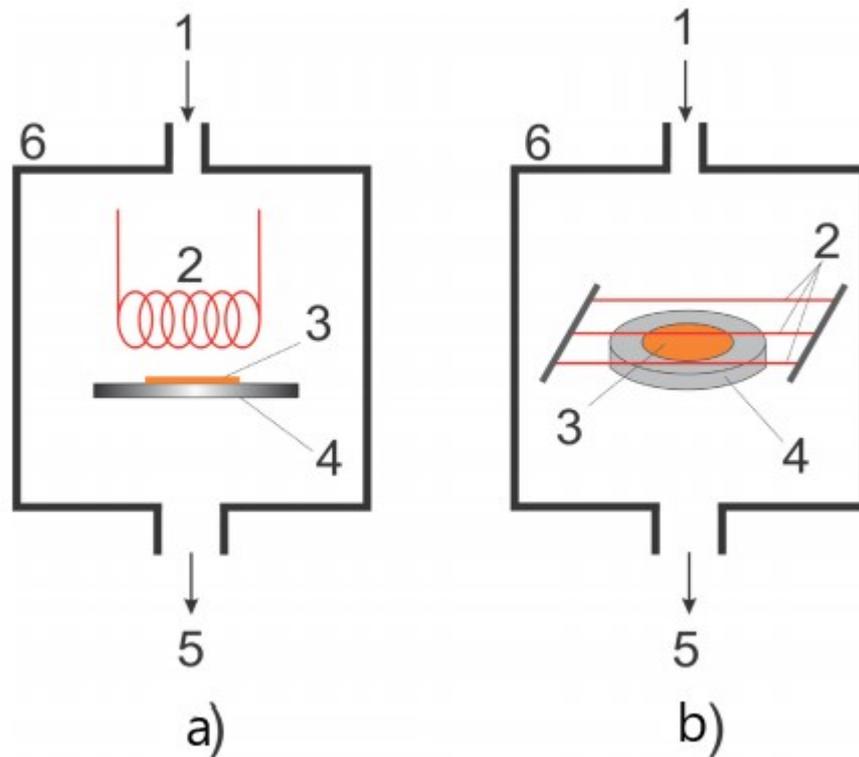


Figure 1.4 - Diagrams of reactors for deposition of diamond by the hot filament vapor deposition method 1 - supply of H₂ / CH₄ gas mixture, 2 - filament, 3 - substrate, 4 - heater, 5 - pumping, 6 - vacuum chamber

The design, developed more than 30 years ago [20], has changed little and is still used in industry. Usually, a mixture of methane and hydrogen in the ratio of 1:100 is fed to a thread twisted into a spiral (Figure 1.4 (a)), heated to a temperature of 1800 - 2400^o C. There are also systems in which several threads simply stretch parallel to each other (Figure 1.4 (b)). The deposition areas in such systems reach 1500 cm². Due to the thermal dissociation of hydrogen and methane molecules on the surface of the filament, active radicals are accumulated above the substrate. Diamond is deposited on the substrate, the temperature of which is provided at the level of 800 – 1000^oC, either by the energy of the thread, or by a separate heater. The maximum growth rate of the diamond film in this method reaches 1 micron / h. Although usually active radicals are transported to the substrate only due to diffusion, the use of enhanced gas convection made it possible to achieve a film growth rate of up to 5 μm / h. The main disadvantage of this method is that the red-hot thread is located near the substrate and, of course, contaminates the growing film

with the material of the thread. For this reason, this deposition technique is not suitable for semiconductor technology tasks. On the other hand, for other tasks, this method is very attractive because of its simplicity and low cost of equipment. To study the process of atomic hydrogen production in this method, the authors of work [21] used infrared resonance enhanced multiphoton ionization spectroscopy. Studies have shown that at a distance of 8 mm from a tungsten filament with a temperature of 2500 °C, the concentration of hydrogen atoms decreases by as much as if the percentage of methane in the mixture increases from one to three. The use of the luminescence method with laser excitation in showed that with an increase in the amount of methane in the mixture from 1 to 5%, the amount of atomic hydrogen drops by 30%. In [22], the authors used highly sensitive laser adsorption spectroscopy to determine the density of CH₃ radicals at different distances from the filament. It was shown that the density maximum of the methyl radicals is at a distance of 4 mm from the surface of the thread. These features were confirmed by theoretical modeling in. Without a doubt, the development of the hot-thread deposition method for diamond provided a wealth of information about the processes in the gas phase. The data obtained were widely used in the development of other deposition methods.

1.9 Types of diamond films

Diamond material can be classified in certain groups. A classification can be seen in Figure 7. First, one distinguishes natural and synthetic diamonds based on their origin of formation. Synthetic diamond can be either single crystalline or polycrystalline. Single crystal diamond is generally classified by the amount of impurities in the crystal structure. Synthetic polycrystalline diamond (subject of the present study) can be classified by the average size of the constituent diamond grains.

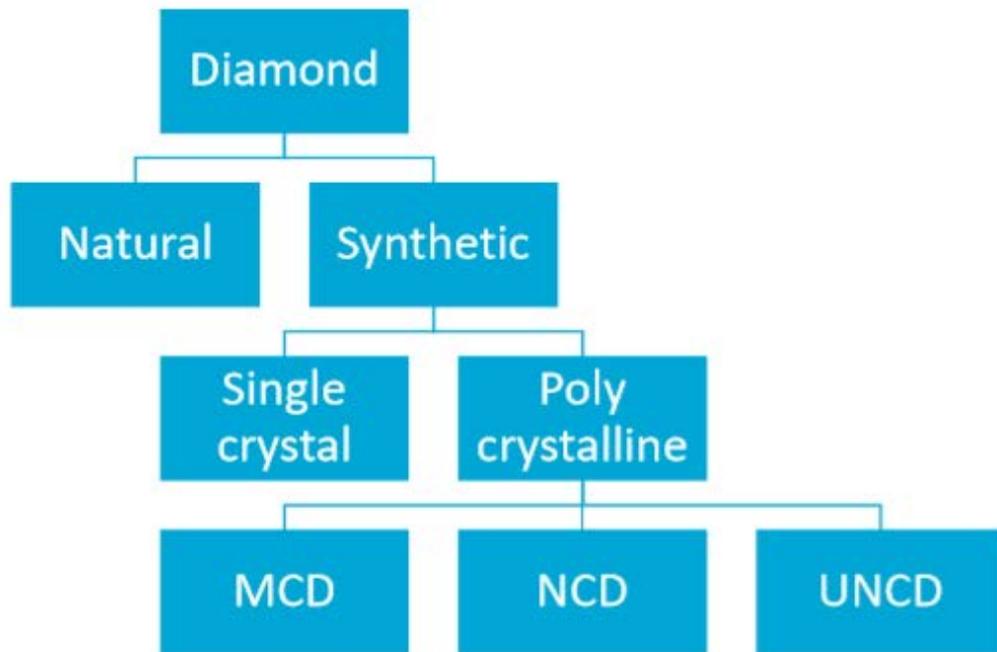


Figure 1.6 Classification (incomplete) of diamond materials

A polycrystalline diamond film consists of a compact agglomeration of multiple crystals (or grains) with random or preferential orientation (i.e. textured films) and a certain size distribution. The size of these grains is usually measured as seen from the top surface, and can be controlled by the process conditions as previously discussed. The size of the grain determines the surface to volume ratio. A smaller grain leads to a higher surface to volume ratio and results in more contaminants in the film [27]. These contaminants aggregate in the grain boundaries of the material. CVD diamond films can be classified into three groups according to the size of their grains: micro-, nano-, or ultrananocrystalline diamond (MCD, NCD or UNCD), respectively.

1.9.1 Microcrystalline diamond (MCD)

If a film has an average grain size of more than 500 nm, it is classified as an MCD film [28] (see Figure 1.7 a). MCD films are grown in a hydrogen rich environment with a $\frac{CH_4}{H_2}$ ratio of around 0.5% [35]. Most MCD films show a very high surface roughness. This is due to their highly columnar structure. During the growth of the MCD film, little to no re-nucleation occurs. Therefore, the seed

crystals grow into columns, which stretch from the nucleation side to the surface of the film with no renucleation, as illustrated in Figure 1.7 b. MCD diamond can be applied in optical applications after polishing. MCD is most often used for cutting and drilling too, for example, due to their high abrasiveness.

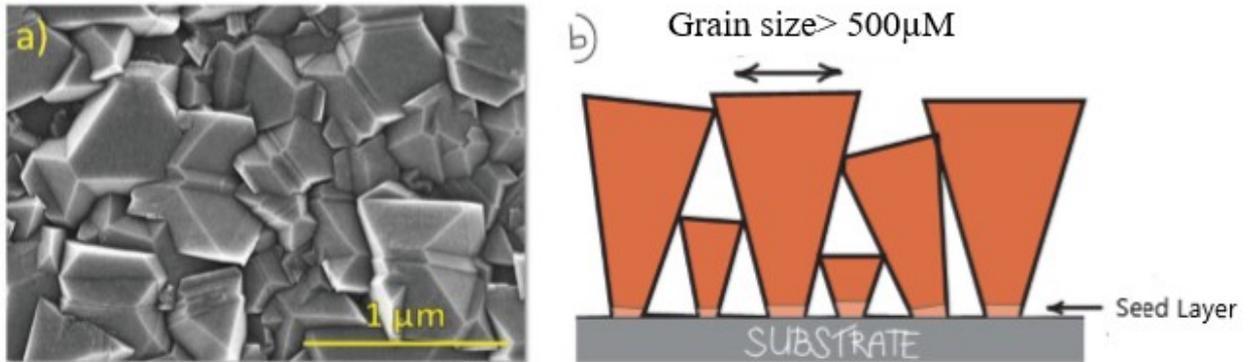


Figure 1.7 a) top view SEM image of MCD. b) Illustration of the section view.
adapted from [29]

1.9.2 Nanocrystalline diamond (NCD)

The NCD films have grains which are smaller than ca. 100 nm in size (see Figure 1.8 a) and are grown in a hydrogen rich environment with a $\frac{CH_4}{H_2}$ ratio of around 3-5%. This reduces the surface roughness considerably when compared to MCD. Due to this fact the NCD film is the most utilized type of diamond films to date. The definition of NCD film is, however, not unanimous among different research groups in the diamond field, given that grain size is also dependent on the film thickness. This means that a thin MCD film could be classified as NCD simply because the grains have not grown enough due to the limited renucleation rate. In conclusion, NCD films are polycrystalline diamond films with a low to moderate renucleation rate and an average grain size that is less than 100 nm (see Figure 1.8 b). Due to the smaller grain sizes, the surface roughness of an NCD film is lower than that of a MCD film.

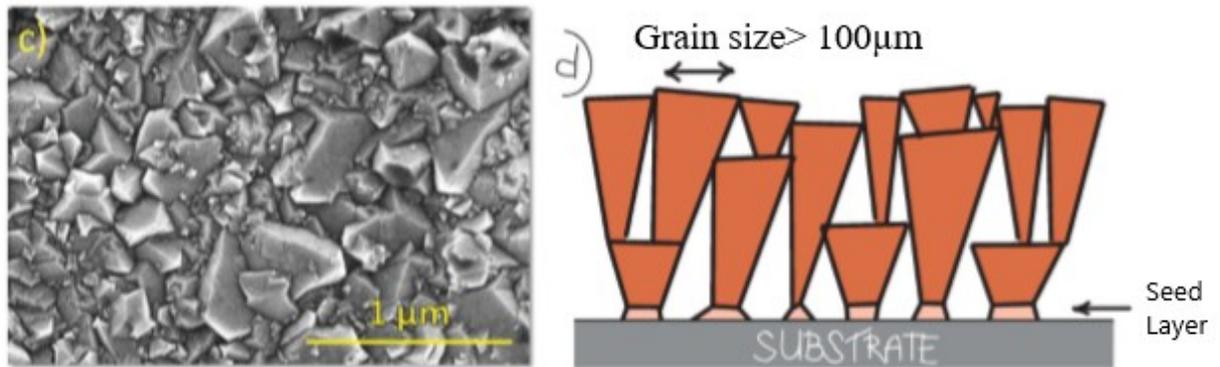


Figure 1.8 a) top view SEM image of NCD. b) illustration of the section view.
adapted from [29]

1.9.3 Ultrananocrystalline diamond (UNCD)

UNCD has an average grain size of 2- 5 nm [30] and is grown in a gas mixture that consists of hydrogen (01%), methane (1%) and argon (98-99%). Due to the specific CVD process conditions, continuous re-nucleation occurs (see Figure 1.9 a). Due to the growth mechanism of UCND, the grain boundaries contain between 2-5% sp² carbons. An advantage of UNCD is that the grain size and the film thickness are independent from each other (see Figure 1.9). This offers the possibility to form very smooth mirror-like films with a large thickness.

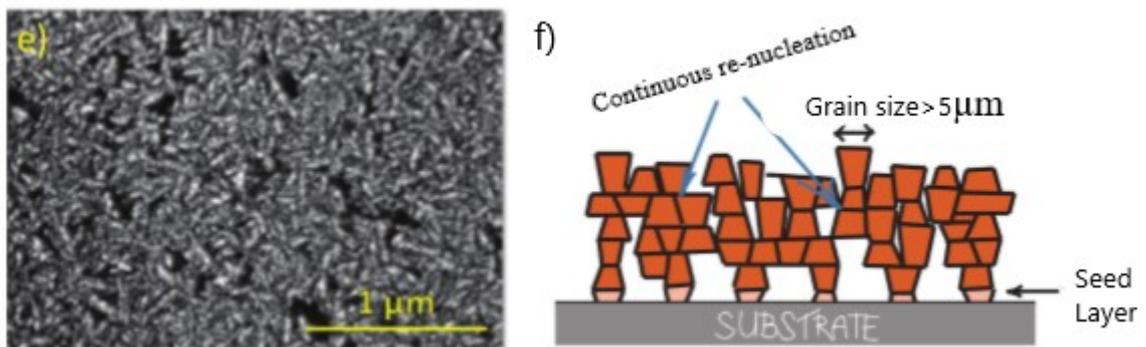


Figure 1.9 a) top view SEM image of UNCD. b) Illustration of the section view.
adapted from [29]

1.10 Application areas for CVD diamond films

CVD diamond is used in a large number of microelectronic, optoelectronic and specialty applications [31], including: laser diode, laser

crystal cooling, microwave device and power semiconductor heat spreaders, submounts and substrates [32-34]. In one case, it was an enabling material for reduction of multi-chip power module packages size [32], and flip chip packaging has increasingly incorporated diamond into the design. The exceptional broad band infrared, terahertz and ultraviolet transparency of diamond combined with its low scatter makes it highly applicable in optical applications. The unique combination of optical transparency and high thermal conductivity led to use CVD diamond in a variety of applications requiring these properties. For example, CVD diamond windows are used in short-wave UV, infrared, CO₂ laser exit windows and output couplers and high-power microwave devices [33]. The parts can be anti-reflection coated for further transparency increase. These windows can be supplied as free-standing parts or mounted to a flange.

The properties of diamond are increasingly utilized in high-energy research for both detection and imaging applications. As a detector material, diamond's key characteristics include beam resistance, fast response and low noise. The radiation resistance of CVD diamond led to its use in particle physics detectors [33] and millimetre-wave vacuum edge-cooled windows for electron cyclotron heating in thermonuclear plasma fusion energy research [35]. also found its use as lenses, monochromators and beam s Diamond splitters. High-power military and space applications, challenged for size and weight found diamond to be a useful material. CVD diamond also found its way into the designs of RF Power packages, amplifiers, radar devices and infrared cameras.

2 Materials and methods

We used mirror-polished hard alloy samples of H10F (Sandvic Coromant) with cobalt content of 10% and grain size of tungsten carbide (WC) 0.6–1 μm as substrates for diamond film deposition. Each sample was a disk of $\text{Ø}20 \times 5$ mm.

Table 2.1 - Results of the elementary analysis of WC-Co

Element	Weight %	In %
W	96.55	67.7
C	3.45	35.3
Co	0.00	0.00
O	0.00	0.00

To determine the density of the film, as well as to carry out tests for wear, mirror polished Si (1 0 0) plates of 5×20 mm in size and SiC balls (6 mm in diameter) were used as substrates.

Table 2.2 - The parameters of the deposition of diamond coatings

Gas	CH_4, H_2
Substrate temperature	850 ± 20 ° C
The distance between the tungsten filaments and the substrate	10 ± 1 mm
Pressure	20 ± 1 Torr
Coating deposition rate	$0.5 - 2$ $\mu\text{m} / \text{h}$
Voltage	80 V
Current	30 A

2.1 Surface preparation for diamond plating

2.1.1- Surface preparation

In sandblasting, abrasive particles are accelerated from the abrasive blasting unit with the help of compressed air energy. Surface cleaning is the removal of unwanted particles from the surface of the material. The surface hardens after cleaning and is ready for coating.

Abrasive blast cleaning is carried out for metal structures; remove old paint, rust and other contaminants. In addition, when blasting the secondary scale is removed, it is formed on the new steel. Angled abrasive particles roughen the surface and create a profile or recess. New, softer types of abrasive (including plastic and wheat starch), as well as special low-pressure abrasive blasting equipment used for the dry method of removing coatings from modern composite materials. This allows you to clean planes, helicopters, cars, trucks and boats without abrasive blasting, which can disrupt the surface structure. Sandblasting apparatus shown in Figure 2.1.



Figure 2.1 – Sandblasting machine T06304

Sandblasting chamber has the form of a box in which there is an opening for placing hands and a window with the help of which the cleaning process is controlled. In this case, it is impossible to hire additional staff to stay in the cell. All

work is done automatically. Sandblasting chambers resemble an injector; To ensure their effectiveness, compressed air is required, which is pre-cleaned of oil.

The sanding gun is the equipment by means of which manual cleaning by means of sand is made. The scope of its use is most often limited to the construction and automotive industry.

The sanding device consists from:

- Compressed air source;
- Sand tanks;
- Handles based on high strength material;
- A hose or pipe that connects all components into a single structure.

The sandblasting chamber is connected to the compressor using the set pressure. Before direct processing, an abrasive of the desired diameter is selected. Then the abrasive material is poured into the chamber. Next, the sample is placed in the chamber. The camera closes tightly. The sample surface is partially or completely sandblasted. If necessary, turn on the filter and the backlight.

The sandblasting system consists of three main components: a compressor, a sandblaster and an abrasive.

The compressor must create a sufficient amount of air pressure in order to move the abrasive from the abrasive jet apparatus to the work surface. Abrasive powder is poured into the apparatus and through the metering valve is fed into the air flow, in the path of which there should be no obstacles. The desired surface finish is achieved by adjusting the compressed air acting on the abrasive.

2.1.2 Surface cleaning.

Ultrasonic Cleaning is a safe and efficient modern procedure, which ensures perfect cleaning within the shortest time possible. It has many advantages over ordinary cleaning methods. Ultrasonic can remove even the most tenacious deposits from parts, which would be difficult to reach by hand; it also reduces the risk of injury or infection, which may occur during manual cleaning.

The ultrasonic cleaning mechanism consists of an ultrasonic generator which produces ultrasonic vibrations within a cleaning solution (liquid) at a frequency of

around 45,000 oscillations per second. These micro-vibrations within the liquid create microscopic bubbles by cavitation. The bubbles then implode and generate acoustic shock energy, which removes even the most ingrained dirt, particularly from parts that traditional cleaning methods cannot reach.



Figure 2.2 – Ultrasonic bath

The cleaning process begins with the immersion of the object in a special bath filled with a specific solution (or distilled water). The generator, mounted in the camera body or completely immersed in the solution, creates ultrasonic vibrations in the liquid, which “divide” the liquid into millions of small bubbles (cavitation effect). Bubbles instantly collapse, emitting tremendous energy, while not damaging the object itself, penetrate into its most inaccessible places and clear of contaminants. The higher the frequency of the waves, the better the cleaning process.

The generator is usually made on the basis of piezoelectric transducers (lead zirconate lead titanate (PZT), barium titanate, magnetostriction).

The effect of cavitation arising from the generation of ultrasonic waves contributes to the best removal of dirt from the surface of the object. An important

aspect that affects the degree of purification of objects - the selection of a particular solvent. Often, the composition of the solvent contains substances (detergents and wetting), which have a direct impact on the degree of purification. Depending on the properties of the item being cleaned, it is necessary to maintain a certain temperature of the solvent. Basically, warm solutions are used ($T = 50 \dots 65 \text{ }^\circ\text{C}$). However, when sterilizing medical devices, it is necessary to maintain the temperature below $38 \text{ }^\circ\text{C}$, in order to avoid protein denaturation. Aqueous solutions are less effective in comparison with special solvents, since they do not have in their composition a chemical component of purification.

A number of large-sized ultrasonic baths are combined into one complex with degreasing machines for more economical use, due to the possibility of repeated use of the solvent. However, the cost of such ultrasonic baths is higher.

2.1.3 Etching and Sowing

Etching of WC-Co samples occurs in HNO_3 : H_2O (1: 3) at $70 \text{ }^\circ\text{C}$ in an ultrasonic bath for 3-5 minutes, then again with ultrasound in distilled water for 10-20 minutes, and then in acetone for 10-15 minutes. After that, the surface of the substrate is seeded with diamond growth centers in an ultrasonic bath with a stable aqueous suspension of nanodiamond particles 5–9 nm in size and 1 wt. % Concentration for 10 min. After sowing, all samples were sonicated in acetone for 10-15 minutes.

Before the procedure of applying a diamond coating on a Si substrate (1 0 0) and SiC, the samples were sequentially treated with ultrasound in acetone, an aqueous solution of nanodiamond (5–9 nm) and again in acetone. This procedure is necessary to clean the substrate and increase the density of nucleation of diamond crystallites

2.2 Deposition of CVD coating on the surface of the material

During the deposition of the film, we used a specialized CVD reactor. The reactor is based on the well-known “hot filament vapor deposition” method.

The general scheme of the installation is shown in Figure 2.3.

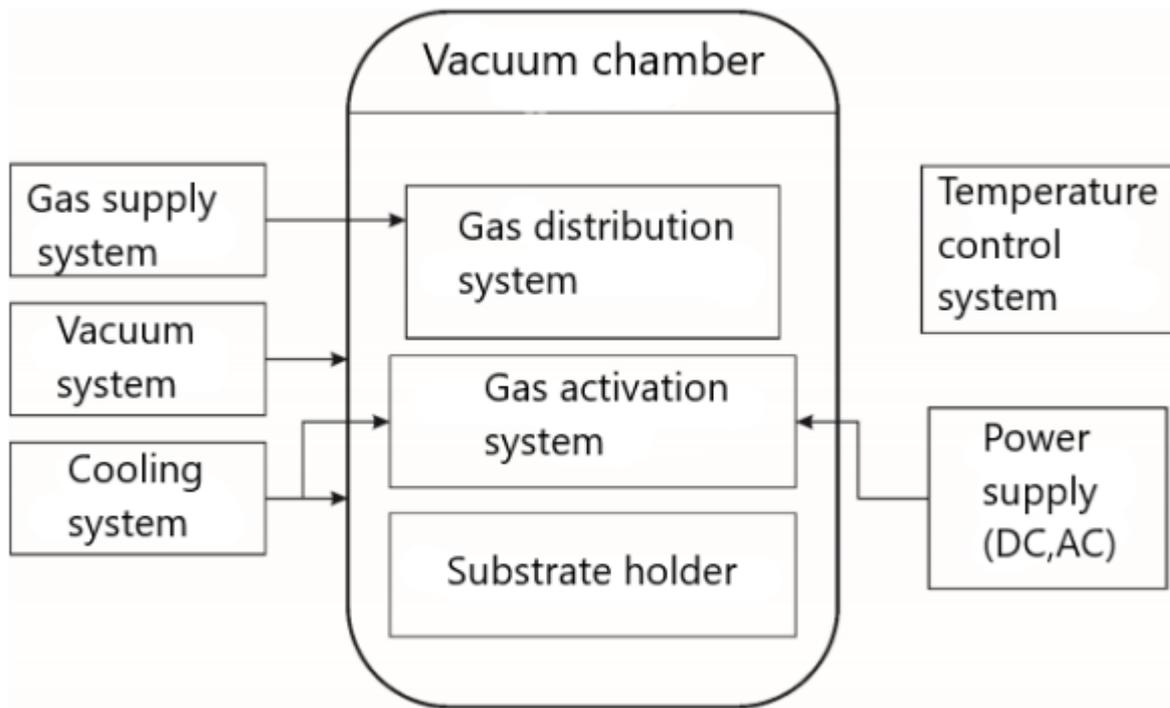


Figure 2.3 -Block installation diagram

The main components of the installation are: gas supply system, vacuum system, vacuum chamber, cooling system, power source, as well as, system temperature control.

The vacuum chamber, as well as the system of vacuum fittings and piping, is made of stainless steel grade 12X18H10T. Due to the fact that the vacuum chamber is in process work exposed to high temperatures, the installation is equipped with a system cooling. The vacuum system of the installation includes a foreline pump and system vacuum valves, which allows you to maintain the necessary pressure in the vacuum chamber, by adjusting the cross section of the exhaust channel. Feed system and adjustment gas is represented by Bronkhorst EL-FLOW precision mass flow controllers with adjustment accuracy 0.01 ml / min. Activation of the working gas mixture is carried out tungsten filaments, the intensity of which is provided by a constant voltage power source. As a tool to control the temperature of the samples during the synthesis diamond coatings used infrared thermal imager ULIRvision (rang measured temperatures: 250 ÷ 1200 ° C, measurement error ± 2 ° C).

Hot filament vapor deposition is a technique that is widely used to apply diamond films.

In this method, gases passing through a carburized tungsten or tantalum filament, which heats up when an electric current passes to about 2400 ° C. Additional dilution gases of argon and hydrogen can also be included.

The effect of the hot filament is that the gas molecules disintegrate into atomic particles, thereby increasing their reactivity. The subsequent reaction then causes precipitation of a solid, i.e. a film on the substrate.



Figure 2.4– Hot filament

Obviously, in a process such as HFCVD, the filament plays a crucial role. The most commonly used filaments are tantalum and tungsten due to their high melting point and high electron emission. The refractory metals that form carbides (for example, tungsten and tantalum), as a rule, should carbonize their surface before supporting the deposition of diamond films. The process of carburizing filament leads to the consumption of carbon from the hydrocarbon precursor gas. Consequently, there is a certain incubation time for the nucleation process, which produces diamond films. Therefore, this process may affect the early stages of film growth, although it is insignificant for longer periods of growth. In addition, the

expansion due to the introduction of carbon leads to the formation of cracks along the entire length of the wire. The development of these cracks is undesirable because it reduces the service life of the filament, but does not adversely affect the quality of the films produced.

In the hot filament vapor deposition (HFCVD) reactor there are tungsten (W) or tantalum (Ta) threads that are heated to 2000–200 ° C. They are placed 5–10 mm above the substrate.

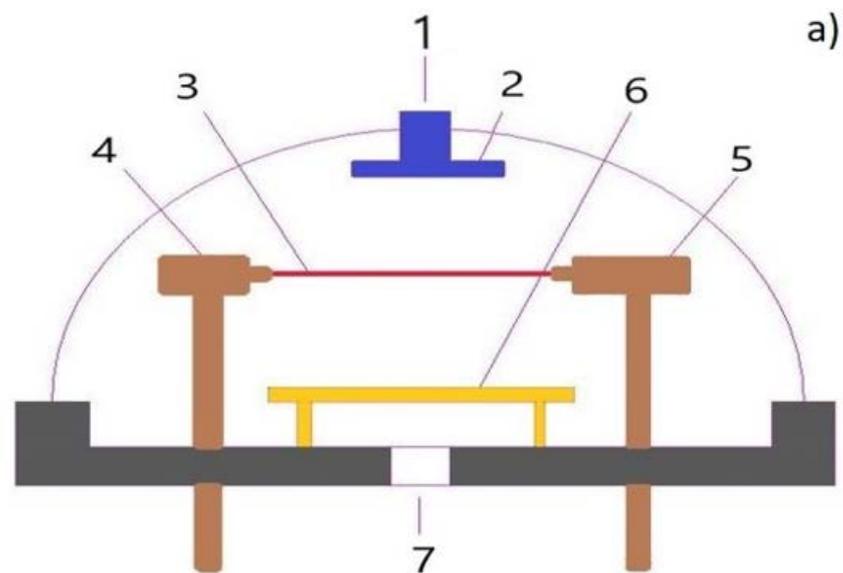


Figure – 2.5 Scheme of reactor (a) and general view of the HFCVD reactor (b): 1- gas supply; 2- gas distribution system; 3- tungsten filament; 4,5- fixation and tension system; 6- substrate; 7- pumping out

Diamond coatings are deposited in a chemical precipitation reactor using hot tungsten filaments, as well as a mixture of hydrogen and methane ($H_2: CH_4 = 50: 1$ for MCD and $H_2: CH_4 = 25: 2$ for UNCD). The substrate temperature during deposition is maintained at 800 ± 25 ° C using an infrared thermal imager (ULIRvision TI170). The pressure in the reactor during the deposition is maintained at 20 ± 1 Torr (Pfeiffer Vacuum CMR 372), the current was 6.5 ± 0.01 A per filament.

2.3 Determination of thin film density using ImageJ

The calculation of the density of the diamond film was performed according to our own developed methods. The obtained diamond coatings are subjected to chemical etching for 30-50 minutes, depending on the acid concentration, as well as the thickness of the substrate.

In this technique, we use paraffin wax that has been heated to a liquid state for good detachment of our coating from the substrate. Paraffin is used to gently peel off the film from the surface of the substrate so as not to damage the resulting film during chemical etching. During chemical etching, the substrate material is completely corroded, but the region of the substrate that has been pre-treated with paraffin remains intact. After etching, the diamond film is washed in distilled water, followed by gasoline treatment.

The next steps in our methodology is the determination of the mass of our film, as well as the determination of the area and thickness of the film.

The density calculation was performed using ImageJ. In ImageJ, it is possible to calculate areas, statistical indicators of pixel values of various selected areas of interest on images that are selected manually or using threshold functions. The program can measure distances and angles. It can create density histograms and draw line profiles.

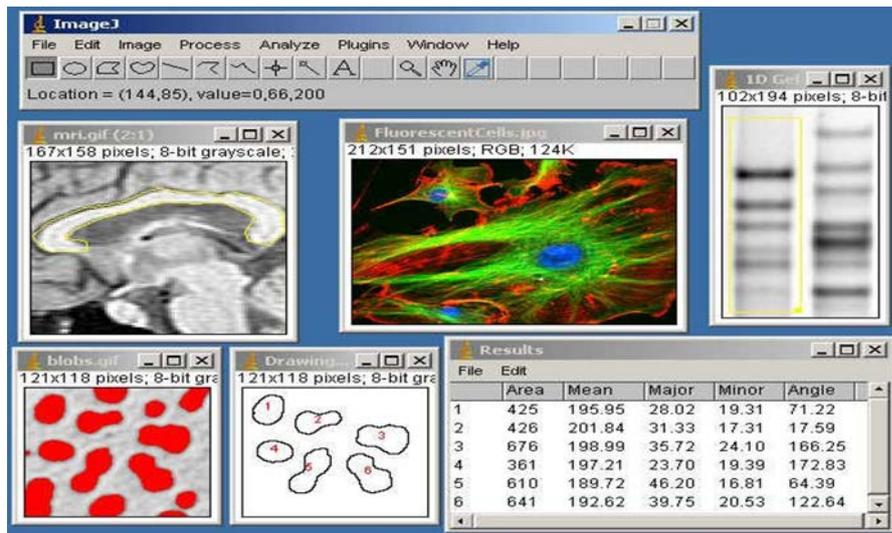


Figure 2.6- ImageJ program

The method of work in the program is as follows: to select a snapshot, click the File button, then Open. Before you start working with a snapshot, you should make sure that the program would produce the results in the dimension you want. Since we are studying images of microsections, we choose the dimension of millimeters (mm). This action will allow ImageJ to convert the image pixel size to mm. Using graph paper; we calculate the square of the square in order to calculate the calculation error. After receiving the error, calculate the area of the film. To calculate the film area, you need to click the Analyze => Measurement button and we get the result of the film area. The result can be saved in Excel.

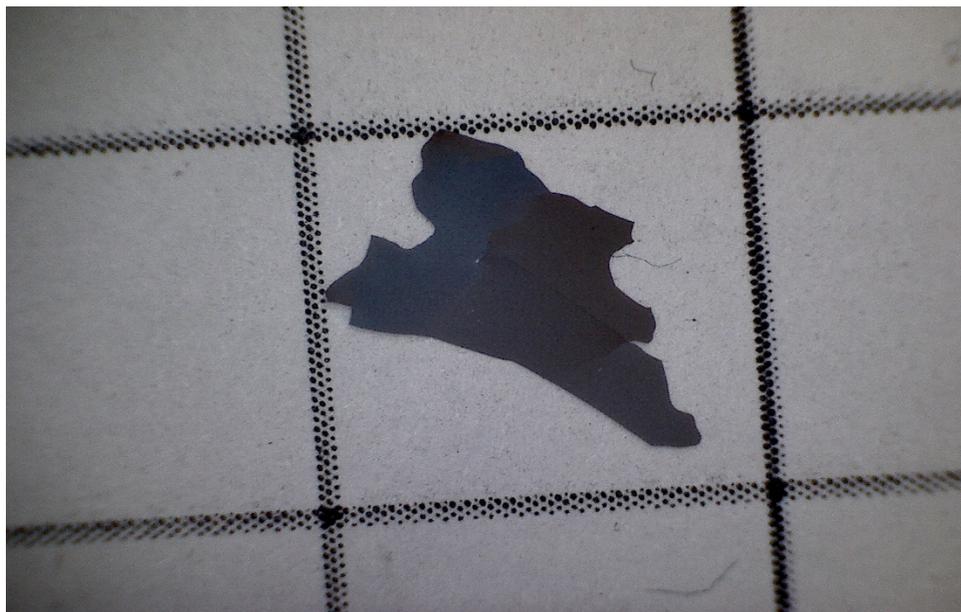


Figure 2.7 - Photograph to determine the area of the diamond film

In the developed method of determining the density of diamond films, we can calculate the area of a film with a complex geometry, as shown in Figure 2.7 with a measurement accuracy of up to 99%.

Measurement of the thickness of the diamond film is carried out on the microscop

The formula for calculating the density of the diamond film:

$$\rho = \frac{m}{V};$$

The developed technique for measuring the density of diamond films can be applied to all types of films with different geometries.

2.4 Wear rate

High Temperature Tribometer (PC-Operated High Temperature Tribometer TNT-S-AH0000)

Purpose and scope

The device is designed to measure friction coefficient, wear resistance, wear intensity in various temperature conditions, gas environment and lubricating liquids. The work of the High Temperature Tribometer is controlled by a personal computer. At the same time, the operation of the device is controlled, the measured values are displayed in real time. The software calculates the wear of the sample and the witness.



Figure 2.8 - High Temperature Tribometer (PC-Operated High Temperature Tribometer THT-S-AX0000)

The sample is fixed on a rotating disk. The friction coefficient is determined during the test by measuring the deflection of the elastic arm. The wear of the test sample is determined by measuring the track formed by the action of a fixed indenter on a rotating sample.

When two materials slide against each other, a certain volume of material is removed from both faces (i.e. they wear). This is defined as wear. Material is removed from both faces even if one material is much harder than the other.

In literature, the wear rate is usually reported on as a volume loss per run distance, per normal applied load.

$$\text{Wear rate} \frac{m^3}{N m} = \frac{\text{volume loss } [m^3]}{\text{normal load } [N] \times \text{run distance } [m]}$$

To obtain the volume loss of the film material, the following steps are taken. A cross section of the wear track is obtained. From this cross section, the area that is removed is determined by the trapezoidal rule. The volume loss is then calculated by multiplying the area loss with the circumference of the wear track.

$$\text{Volume loss } [m^3] = 2 \times \pi \times \text{radius } [m] \times \text{area loss } [m^2];$$

Calculation wear

To determine the magnitude of wear the wear scar on the ball will be measured by WLI. With the diameter of the wear, the volume loss of the counter body ball can be calculated. For the determination of the volume loss that occurred to the diamond film, a cross section of the wear track from the WLI image will be taken. From this cross section, the area loss can be calculated. After which the area loss will be converted to a volume loss of the film. Final this volume loss is converted to a wear rate of the film.

Ball wear

The volume loss of the ball can be derived from the size of the wear scar by using the following formula that is adapted from [26]

$$V(d) = \frac{\pi}{3} \left(R - \sqrt{R^2 - \left(\frac{d}{2}\right)^2} \right);$$

With V being the total volume that is removed from the sphere, R the original radius of the sphere, and d is the diameter of the wear scar (see Figure 2.8).

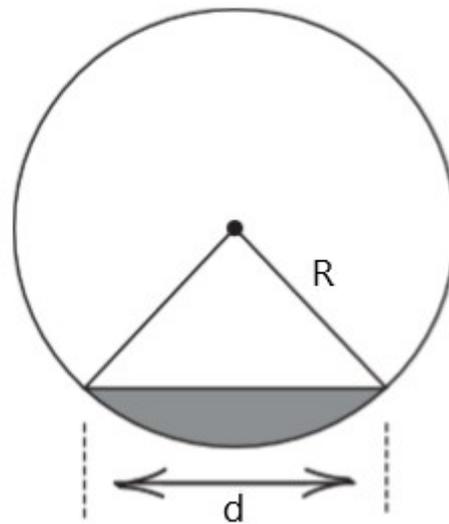


Figure 2.9 Sketch of the geometrical model for the calculation of the wear volume of the counterbody ball.

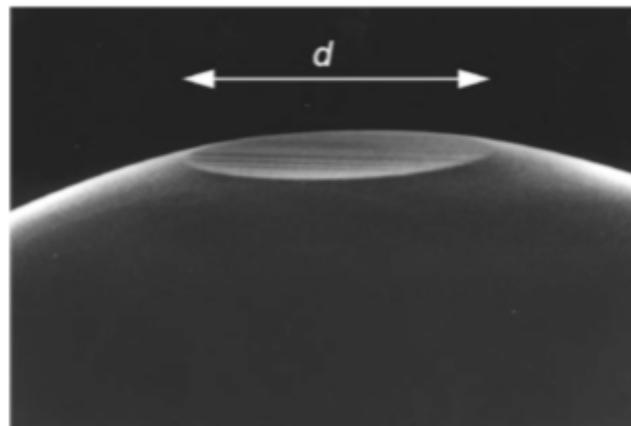


Figure 2.10 Wear scar diameter as viewed in a scanning electron microscope (SEM) [37]

It must be noted that the equation assumes that the wear scar is the result of a planar cut, as shown schematically in Figure 2.9. If the ball does not have a planar cut this equation is no longer valid.

2.5 Measurement of the microhardness of diamond coatings

The Nano Indenter® G200 system is an accurate, flexible, user-friendly test equipment for the nanoscale mechanical. G200 measures Young modulus and hardness, including strain measurement over 6 orders of magnitude, from

nanometers to millimeters. The system can also measure the complex modulus of polymers, gels and biological tissue as well as the creep response (stress rate sensitivity) of thin metal films. Modular options can accommodate a variety of applications: frequency-specific testing, quantitative scratching and wear testing, integrated probe-based imagination, high-temperature nanoindentation testing, extended load capacity up to 10N and custom test protocols.



Figure 2.11–The Nano Indenter ® G200

The technique of work on nanoindenter consists in measuring the nanohardness of diamond coatings. To get started on the nanoindenter, you need to run the Test Works 4 program. The sample is installed on the panel and adjusted using a standard. Select test requirements. The main requirements are: the number of injections, the depth of the indentation, the applied load on the tip.

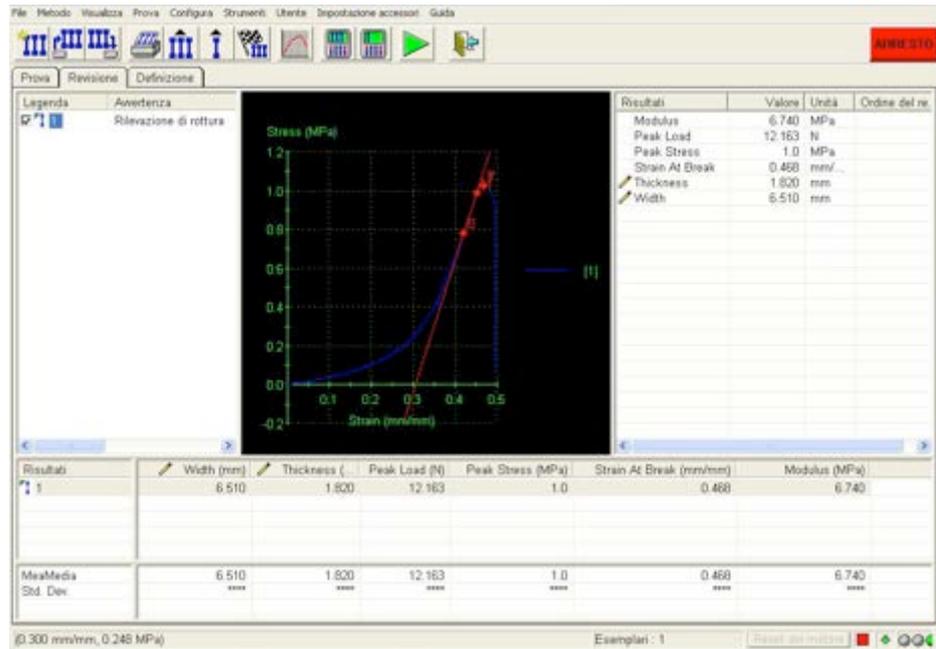


Figure 2.12 –Test works 4 programs

A diamond probe with a tip of up to 100 nm is used to apply to the sample surface. The load applied to the tip and the depth of penetration are subsequently measured during the process. The depth of the indentation is used to calculate the area of the tip that was in contact during the indentation. This area is used to measure the hardness of the material.

2.6 Raman spectroscopy

This method is not demanding on sample preparation and is not sensitive to absorption bands (unlike IR spectroscopy), and allows determining the presence of graphite sp^2 and diamond sp^3 phases in carbon coatings.

During the study, a sample is irradiated by a beam of monochromatic light, which is scattered by it. The main part (99.999%) of the scattered radiation on the sample has a similar frequency, which is also true for the source radiation (Rayleigh scattering). The remaining part of scattered radiation (Ramanovskaya) has a frequency brightened by the frequency of the excitation laser radiation (0.0001%). Rays that are received by dissection are assembled by a lens and transmitted through a light filter; it separates the Rayleigh radiation from Ramanovsky. Separated Raman rays, transmitted to the detector, are transmitted in the form of data to a computer.

The measurement of the Raman spectra was carried out on the complex Centur U HR (Fig. 2.13)

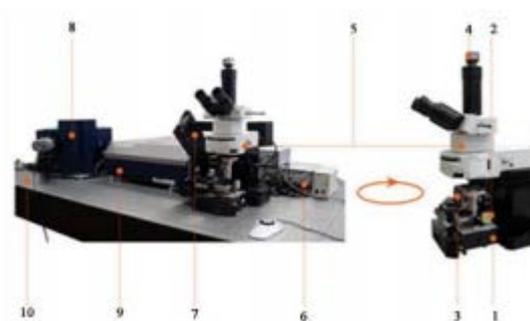


Figure 2.13 – the appearance of the installation Centaur U HR 1 - Ratis scanning base; 2 - Certus scanning head; 3 - z-slide for Vectus lens; 4 - video camera; 5 - optical microscope; 6 - the controller; 7 - opto-mechanical module; 8 - spectrometer; 9 - periscopic interface system with a microscope; 10 - optical table

**TASK FOR SECTION
"FINANCIAL MANAGEMENT, RESOURCE EFFICIENCY
AND RESOURCE SAVING"**

To student:

Group	Full name
4BM7E	Kunashenko Sergey

School	School of Advanced Manufacturing Technologies	Department	Materials Science and Technology
The level of education	Master degree	Direction / specialty	Computer Simulation of Materials Production, Processing and Treatment

Background data to the section "Financial management, resource efficiency and resource saving":

<i>1. The cost of scientific research (NI) resources: material, technical, energy, financial, informational and human</i>	<i>1. Materials costs -109000 rub.; Costs for salaries of performers – 179543, 7 rub.; Extrabudgetary funds deductions –59653, 03 rub. Overhead charges – 60884, 19rub.</i>
<i>2. Norms and standards of resource use</i>	<i>2. This research is carried out for the first time; therefore, there are no norms and standards for the use of resources.</i>
<i>3. Used tax system, rates of taxes, deductions, discounting and lending</i>	<i>3. The coefficient of deductions for payment to extra-budgetary funds - 30%</i>

The list of issues to be investigated, designed and developed:

<i>1. Assessment of the commercial potential of engineering solutions (IR)</i>	<i>1. Analysis of competitive technical solutions</i>
<i>2. Formation of the plan and development schedule and introduction of IR</i>	<i>2. Determination of work stages; the definition of labor-intensive work; Gantt graphics development</i>
<i>3. Justification of the necessary investments for the development and implementation of R & D</i>	<i>3. Project costing</i>
<i>4. Budgeting engineering project</i>	<i>4. This research is conducted within Federal project, so there is no need in investments from others.</i>
<i>5. Assessment of resource, financial, social, budgetary efficiency of R & D and potential risks</i>	<i>5. This research is carrying out without any exploitation costs.</i>

The list of graphic material (with the exact indication of the required drawings):

<i>1. Evaluation map for comparison of competitive technical solutions</i>
<i>2. Calendar schedule for conducting scientific research</i>

Date of assignment for the section on a linear schedule	
--	--

Task issued by a consultant:

Position	Full name	Degree, title	Signature	Date
Assistant professor	Skakovskaya N.V.	PHD		

The task was accepted by the student:

Group	Full name	Signature	Date
4BM7E	Kunashenko S.E		

4. Financial management, resource efficiency and resource saving

4.1 Analysis of competitive technical solutions

Analysis of technical competitive solutions helps to make correction in the project in order to successfully resist to competitors. Carrying out such analysis this is necessary to estimate advantages and disadvantages of competitors. Evaluation chart is made for it (table 4.1).

Diamond coating on cutting tools is analysis object.

Diamond like coating (DLC) and cutting tools without coating are chosen for comparison with analysis object.

Table 4.1 - Evaluation map for comparison of competitive technical solutions (developments)

Evaluation criterion	Weight of criterion	Mark			Competitiveness		
		B_{ϕ}	B_{k1}	B_{k2}	K_{ϕ}	K_{k1}	K_{k2}
1	2	3	4	5	6	7	8
Technical criterion for resource saving evaluation							
1. Increase productivity	0.3	2	3	1	0.6	0.9	0.3
2. Convenience in obtaining coatings	0.1	4	1	5	0.4	0.1	0.5
3. Energy efficiency	0.2	2	4	1	0.4	0.8	0.2
4. Reliability	0.1	1	5	2	0.1	0.5	0.2
5. Security	0.1	3	2	2	0.3	0.2	0.2
6. The need for memory resources	0.1	1	1	2	0.1	0.1	0.2
7. Quality of the intelligent interface	0.07	3	1	3	0.21	0.07	0.21
8. Ability to connect to a computer network	0.03	4	5	3	0.12	0.15	0.09
Total	1	20	22	19	2.23	2.82	1.9
Economical criterion of efficiency evaluation							

1. Competitiveness	0,3	1	3	2	0.1	0.9	0.6
2. Market penetration rate	0,05	3	5	1	0.1 5	0.2 5	0.0 5
3. Price	0,1	5	1	3	0.5	0.1	0.3
4. Estimated lifetime	0,2	4	2	2	0.8	0.4	0.4
5. Research funding	0,2	2	1	5	0.4	0.2	1
6. Development certification	0,05	1	4	2	0.0 5	0.2	0,1
Total	1	2 0	21	20	3.2	2.3	2.7

There are two types of criterion are used for estimation: technical and economical.

Index's weight add up to 1. Each of mark indexes evaluate by five-point scale.

Rivals competitiveness C

$$C = \sum W_i \times B_i \quad (4.1)$$

Where W_i – weight index;

B_i – mark of i-index.

All results are shown in chart 4.1. There is a sum of all competitiveness of each cutting tool with or without coating in line «In total».

The obtained calculation results are summarized in table 4.1. The “Total” line contains the sum of all competitiveness for each of the materials for cutting tools with polycrystalline diamond coating.

One of the important areas of efficiency of operation of cutting tools is the life. Diamond coated cutters are more wear-resistant, so their durability is several times longer than the service life of uncoated cutters and with a diamond-coated coating. Diamond-coated mills are more expensive than uncoated and diamond-coated coatings; however, due to their durability and quality, the overall price for machining a particular material is lower than when using uncoated mills and diamond-coated coatings.

4.2 Planning the study of the physico-mechanical properties of diamond coatings

In this paper, the project organization consists of two people: the project manager and the engineer. Work scheduling allows you to distribute duties between the project executors, calculate the salaries of employees, and also guarantees the implementation of the project on time. The sequence and content of work, as well as the distribution of performers are shown in table 4.2.

Table 4.2 - List of stages, work and distribution of performers

Main steps	№ work	Content of work	Position performer
Assignment	1	Drafting and approval of technical specification	S.S
Choice of direction research	2	Selection and study of materials on the topic	Engineer
	3	Choosing the direction of research	S.S
Theoretical and experimental studies	4	Research of diamond coating	Engineer
	5	The choice of material and methods for research	S.S
	6	Research on the durability of polycrystalline diamond coatings	Engineer
Discussion of the results	7	Processing of the results	Engineer
	8	Scientific rationale for the results and conclusions	Engineer
Compilation of a report	9	Development plan for the design of the project	S.S
	10	Report design	Engineer
Report protection	11	Protection of final qualifying work	Engineer

4.3 Determination of the complexity of the work

Labor costs in most cases form the bulk of the cost of development, so the important point is to determine the labor intensity of the work of each of the research participants

The complexity of the implementation of scientific research is estimated by experts in person-days and is probabilistic in nature, because depends on many factors difficult to take into account. To determine the expected (average) value of labor intensity, use the following formula [17]:

$$t_{\text{exp } i} = \frac{3t_{\text{min } i} + 2t_{\text{max } i}}{5}, \quad (4.3)$$

Where $t_{\text{exp } i}$ = expected complexity of the i-th work pers.-days;

$t_{\text{min } i}$ = the minimum possible complexity of the implementation of the specified i-th work (optimistic assessment: assuming the most favorable set of circumstances), pers.-days;

Based on the expected complexity of the work, the duration of each work in working days T_p is determined, taking into account the parallel execution of work by several performers. Such a calculation is necessary for sound payroll calculation, since the proportion of wages in the total estimated cost of scientific research is about 65%.

$$T_{w_i} = \frac{t_{\text{exp } i}}{N_i}, \quad (4.3.1)$$

where T_{w_i} - the duration of one work, *days*;

$t_{\text{exp } i}$ - the expected complexity of the performance of one work, *pers.-day*;

N_i - the number of performers performing simultaneously the same work at this stage, *pers.*

4.4 Development of the project schedule

The most convenient and visual is the construction of a tape schedule of research in the form of a Gantt chart.

The Gantt chart is a horizontal tape chart on which work on the topic is represented by lengthy segments, characterized by dates of the beginning and end of the work.

For the convenience of plotting, the duration of each of the stages of work from working days should be translated into calendar days. You must use the following formula [17]:

$$T_{c_i} = T_{w_i} \cdot k_{\text{cal}}, \quad (4.4)$$

Where T_{ci} - the duration of the i-th work in calendar days;

T_{wi} - the duration of the i-th job in working days;

k_{cal} - calendar factor.

The calendar factor is determined by the following formula:

$$k_{cal} = \frac{T_{cal}}{T_{cal} - T_{off} - T_{hol}}, \quad (4.4.1)$$

Where T_{cal} - number of calendar days per year;

T_{off} - number of days off per year;

T_{hol} - number of holidays per year.

Example of calculation for stage 1 of work (Drafting and affirming of technical task):

$$t_{expi} = \frac{3t_{mini} + 2t_{maxi}}{5} = 2,8 \text{ чел} - \text{дней};$$

$$T_{wi} = \frac{2,8}{1} = 3 \text{ дней};$$

For a six-day working week (for an academic adviser), the calendar factor is

$$K_{cal} = \frac{T_{cal}}{T_{cal} - T_{off} - T_{hol}} = \frac{365}{365 - 51 - 15} \approx 1.22;$$

$$T_k = T_p \cdot k_{кал} = 3 \cdot 1.22 = 3.66 \approx 4 \text{ дней};$$

For a five-day working week (for an engineer), the calendar factor is

$$k_{cal} = \frac{T_{cal}}{T_{cal} - T_{off} - T_{hol}} = \frac{365}{365 - 102 - 15} \approx 1.47;$$

$$T_{cal} = T_w \cdot k_{cal} = 3 \cdot 1.47 = 4.41 \approx 4 \text{ дней};$$

The obtained calculation results are listed in table 4.4.

Table 4.4 - Timing indicators of scientific research

Title of work	Complexity of work	Duration of work in working days T_{wi}	Duration of work in calendar days T_{cal}

	$t_{\min i,}$ pers- days		$t_{\max i,}$ pers- days		$T_{\exp i,}$ pers- days		a	e	a	e
	a	e	a	e	a	e				
1. Drafting and approval of technical specifications	2	-	4	-	2,8	-	3	-	4	-
2. Selection and study of materials on the topic	4	-	6	-	4,8	-	5	-	7	-
3. Choosing the direction of research	2	-	4	-	2,8	-	3	-	4	-
4. Research of diamond coating	-	10	-	16	-	12,4	-	12	-	18
5. Choice of material and methods for research	2	-	4	-	2,8	-	3	-	4	-
6. Research of the wear resistance of diamond coatings	-	10	-	16	-	12,4	-	12	-	18
7. Investigation of the physical properties of diamond coatings	-	15	-	20	-	17	-	17	-	25
8. Scientific rationale for the results and conclusions	-	6	-	8	-	5,6	-	6	-	9
9. Development plan for the design of the project	2	-	6	-	3,6	-	4	-	5	-
10. Report design	-	20	-	30	-	24	-	24	-	35
11. Protection of final qualifying							-	1	-	1

work										
------	--	--	--	--	--	--	--	--	--	--

On the basis of table 4.4 a calendar schedule is built (tables 4.4.1).

Table 4.4.1 - Calendar schedule of the project on the topic

№	perf	T_{ki} ka	Duration of work																	
			Dec		Jan		Feb.			March			April			May			June	
			2	3	2	3	1	2	3	1	2	3	1	2	3	1	2	3	1	
1	s.s	4	█																	
2	s.s	7		█																
3	s.s	4			█															
4	e	18					█	█	█											
5	s.s	4							█											
6	e	18								█	█	█								
7	e	25										█	█	█	█					
8	e	9													█	█				
9	s.s	5																█		
10	e	35																█	█	
11	e	1																		█

█ - Scientific supervisor █ - Engineer

Based on the graph data (Table 4.4.1), it can be concluded that the duration of work on the study of the physicomechanical properties of diamond coatings will take 11 decades. The beginning of the project development will be in the second decade of February and will end in the first decade of July.

The value of the actual duration of work may be less (under favorable circumstances) of the calculated value, and more (under adverse circumstances), since the complexity is probabilistic in nature.

The duration of the project in calendar days is equal to

- 24 day (the duration of the project manager);
- 105 days (the duration of the project by an engineer).

4.5 Design Cost Budget

When planning a project budget, it is necessary to take into account all types of expenses that are associated with its implementation. To form a project budget, use the following cost group:

- material costs of the project;
- the basic salary of the project executors;
- additional salary of the project executors;
- deductions to extra-budgetary funds (insurance deductions);
- overhead

4.5.1 Calculation of material costs of the project

Material costs include: raw materials and materials purchased from the outside, purchased materials, stationery, cartridges, etc.

Table 4.5–Material costs

Materials and equipment	Unit of measurement	Amount	Price per one, rub	Material costs rub.
Paper	package	1000	0,2	200
Acetone	Litre	40	100	4000
Acid	Litre	6	800	4800
Blanks	Kilogram	20	5000	100000
Total			109000	

In total, material costs amounted to 109,000 rubles.

4.5.2 The basic salary of the project executors

The amount of salary expenditure is determined on the basis of the labor intensity of the work performed and the current system of salaries and tariff rates. The basic wage includes a premium paid monthly from the wage fund in the amount of 20–30% of the tariff or salary.

The article includes the basic salary (S_b) of workers directly involved in the implementation of NTI (including bonuses, additional payments) and additional salary (S_{ext}):

$$S_s = S_b + S_{ext}, \quad (4.5.2)$$

where S_b - the basic salary; S_{ext} - additional salary (12-20% from S_b).

The basic salary of the head (engineer):

$$S_b = S_d * T_w, \quad (4.5.2.1)$$

where T_w - the duration of the work performed by the project performer, working days;

S_d - average daily wage of an employee, rub.

$$S_d = \frac{S_m \cdot M}{F_d}; \quad (4.5.2.2)$$

where S_m - the monthly salary of an employee, rub.;

M - the number of months of work without leave during the year:

at holiday in 28 *working days* $M = 11$ months, 5-day week;

at holiday in the 56 *working days* $M = 10$ months, 6-day week;

F_d - valid annual fund of working time of the project executors, working days

Monthly salary of an employee:

$$S_m = S_{tr} \cdot (1 + k_{pr} + k_s) \cdot k_d; \quad (4.5.2.3)$$

where S_{tr} – salary at the tariff rate, rub;

k_{pr} is a premium coefficient equal to 0.3 (i.e., 30% of S_s);

k_s - the coefficient of additional payments and surcharges is approximately 0.2–0.5 (at research institutes and at industrial enterprises — for expanding the service industries, for professional skills, for harmful conditions: 15–20% of S_s);

k_d - district coefficient equal to 1.3 (for Tomsk).

4.5.3 Additional salary of the project performers

The costs of additional salaries for the executors of the topic take into account the amount of additional payments for deviation from normal working conditions, as well as payments related to the provision of guarantees and compensations, provided by the Labor Code of the Russian Federation.

Additional salary:

$$S_{ext} = k_{ex} \cdot S_b; \quad (4.5.3)$$

where k_{ex} - the coefficient of additional wages (at the stage of design is taken equal to 0,135).

Salaries are taken in accordance with the positions held by TPU.

Calculation of salary for scientific supervisor (six-day working week):

$$S_m = S_{tr} \cdot (1 + k_{pr} + k_s) \cdot k_d = 26300 \cdot (1 + 0.3 + 0.2) \cdot 1.3 = 51285 \text{ pyб} ;$$

$$S_d = \frac{S_m \cdot M}{F_d} = \frac{51285 \cdot 10}{365 - 66 - 56} = 2110.5 \text{ pyб};$$

$$S_b = S_d \cdot T_w = 2110.5 \cdot 22,4 = 47275,2 \text{ pyб};$$

$$S_{ext} = k_{ext} \cdot S_b = 0.135 \cdot 35456.3 = 4786.6 \text{ pyб};$$

Calculation of salary for engineer (six-day working week):

$$S_m = S_{tr} \cdot (1 + k_{pr} + k_s) \cdot k_d = 17000 \cdot (1 + 0.3 + 0.2) \cdot 1.3 = 33150;$$

$$S_d = \frac{S_m \cdot M}{F_d} = \frac{33150 \cdot 11}{365 - 117 - 28} = 1657.5 \text{ pyб}$$

$$S_b = S_d \cdot T_w = 1657.5 \cdot 79,8 = 132268,5 \text{ pyб}$$

$$S_{ext} = k_{ext} \cdot S_{ext} = 0.135 \cdot 17316 = 137904 \text{ pyб}$$

Results of calculations of salaries of performers are shown in table 4.7.

Table 4.7 – Calculations of salaries of performers

Project performers	S_{tr} , rub.	K_{pr}	K_s	K_d	S_m , rub	S_d , rub	T_w , work. day	S_b , rub	K_{ext}	S_{ext} , rub	Total, rub
Scientific supervisor	26300	0.3	0.2	1.3	51285	2110.5	16.8	47275.2	0.135	4786.6	52061.8
Engineer	17000	0.3	0.2	1.3	33150	1657.5	83.2	132268.5	0.135	186170	150885.5

As a result of these calculations, the basic wage of the project executors is calculated. From table 4.7 it can be seen that the rate of the scientific supervisor is the highest, but the final basic salary turned out to be the highest for the engineer, since the basic salary depends on the duration of the project.

4.5.4 Extrabudgetary funds deductions (insurance deductions)

Deductions to extra-budgetary funds include the norms established by the legislation of the Russian Federation of the state social insurance bodies (FSS), the pension fund (PF) and medical insurance (FFOMS) from the costs of remunerating employees.

The amount of payments to extra-budgetary funds is determined on the basis of the following formula:

$$S_{eb} = k_{eb} \cdot (S_b + S_{ext}); \quad (4.5.4)$$

Where k_{eb} – coefficient of deductions for payment to extra-budgetary funds (pension fund, fund of compulsory medical insurance, etc.).

Deductions to extrabudgetary funds are presented in table 4.8.

Table 4.8 – Extrabudgetary contributions

Project performers	Basic salary, rub	Additional salary, rub.
Scientific supervisor	47275.2	4786.6
Engineer	132268.5	18617.0
Coefficient of deductions for payment to extra-budgetary funds	0.3	
Total		
Scientific supervisor	15618,54	
Engineer	45265,65	

4.5.5 Overhead charges

Overhead costs take into account other expenses of the organization that are not included in previous items of expenditure: printing and photocopying of research materials, payment of communication services, electricity, postal and telegraph expenses, reproduction of materials, etc. Their value is determined by the following formula:

$$S_{over} = (\text{sum of articles } 1 \div 4) \cdot k_{over} \quad (4.5.5)$$

where k_{over} – coefficient taking into account overhead costs, we take in the amount of 16%.

$$S_{over} = (S_m + S_b + S_{ext} + S_{eb}) \cdot 0.16;$$

$$S_{over} = (109000 + 179543,7 + 23403.6 + 60884,19) \cdot 0.16 = 59653,03 \text{ py6}$$

4.6 Formation of costs for the project

The definition of the budget of the project costs is given in table 4.9.

Table 4.9 - Calculation of the budget of expenditures of NTI

Article name	Sum, <i>rub</i>	Total in %
Costs for basic salaries	179543.7	41.6
Costs for additional salaries	23403.6	5.4
Overhead charges	60884.19	14
Extrabudgetary funds deductions	59653.03	13.8
Material costs of project	109000	25.2
Budget of the project	432484.52	100

The budget of all project costs is 432484.52 rubles. The largest percentage of the budget is the material costs for project (41.6 %).