TOMSK POLYTECHNIC UNIVERSITY — ТОМСКИЙ ПОЛИТЕХНИЧЕСКИЙ УНИВЕРСИТЕТ

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

Школа Инженерная школа новых производственных технологий
Направление подготовки (специальность) Материаловедение и технологии материалов
Отделение школы (НОЦ) Отделение материаловедения

МАГИСТЕРСКАЯ ДИССЕРТАЦИЯ

Тема работы
Исследование износостойкости поликристаллических алмазных покрытий на твердых сплавах на основе WC-Co

УДК: 621.793-032.81-047.37:669.018.25

Студент

Группа	ФИО	Подпись	Дата
4БМ7И	Халафов Рустам		

Руководитель

Должность	ФИО	Ученая степень, звание	Подпись	Дата
Ст. научный сотрудник НПЛ ИШНПТ ТПУ	Линник Степан Андреевич	К.т.н.		

КОНСУЛЬТАНТЫ:

По разделу «Финансовый менеджмент, ресурсоэффективность и ресурсосбережение»

Должность	ФИО	Ученая степень, звание	Подпись	Дата
Доцент ОСГН ТПУ	Скаковская Наталия Вячеславовна	К. ф. н., доцент		

По разделу «Социальная ответственность»

Должность	ФИО	Ученая степень, звание	Подпись	Дата
Доцент ООД ТПУ	Горбенко Михаил Владимирович	К.т.н., доцент		

ДОПУСТИТЬ К ЗАЩИТЕ:

Руководитель ООП	ФИО	Ученая степень, звание	Подпись	Дата
22.04.01 Материаловедение и технологии материалов	Овечкин Борис Борисович	К. т. н., доцент		

Томск – 2019 г.

Планируемые результаты обучения по ООП 22.04.01

Код результата	Результат обучения
Р1	Осуществлять сбор, анализ и обобщение научно-технической информации в области материаловедения и технологии материалов с использованием современных информационно-коммуникационных технологий, глобальных информационных ресурсов
Р2	Работать с патентным законодательством и авторским правом при подготовке документов к патентованию и оформлению ноу-хау
Р3	Выполнять маркетинговые исследования и анализировать технологический процесс как объекта управления, разрабатывать технико-экономическое обоснование инновационных решений в профессиональной деятельности
Р4	Руководить коллективом в сфере своей профессиональной деятельности, толерантно воспринимая социальные, этнические, конфессиональные и культурные различия
Р5	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.
Р6	Разрабатывать новые и модернизировать существующие технологии получения керамических, металлических материалов и изделий, в том числе наноматериалов
Р7	Внедрять системы управления качеством продукции в области материаловедения, эксплуатировать оборудование, позволяющее диагностировать материалы и изделия из них, в том числе наноматериалы
Р8	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
Р9	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
Р10	Самостоятельно осваивать новые методы исследования, изменять научный, научно-педагогический и производственный профиль своей профессиональной деятельности
Р11	Применять принципы рационального использования природных ресурсов, основные положения и методы социальные, гуманитарные и экономические подходы при решении профессиональных задач с учетом последствий для общества, экономики и экологии.
Р12	Использовать основные категории и понятия общего и производственного менеджмента в профессиональной деятельности

TOMSK POLYTECHNIC UNIVERSITY

ТОМСКИЙ ПОЛИТЕХНИЧЕСКИЙ УНИВЕРСИТЕТ

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

Школа Инженерная школа новых производственных технологий
Направление подготовки (специальность) Материаловедение и технологии материалов
Отделение школы (НОЦ) Отделение 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	Халафову Рустаму

Тема работы:

Исследование износостойкости поликристаллических алмазных покрытий на твердых сплавах на основе WC-Co	
Утверждена приказом директора ИШ НПТ	Приказ №_____ от ____________

Срок сдачи студентом выполненной работы:	

ТЕХНИЧЕСКОЕ ЗАДАНИЕ:

Исходные данные к работе	*В работе исследовали износостойкость поликристаллических алмазных покрытий на твёрдых сплавах на основе WC-Co. Поликристаллические алмазные покрытия получили химического осаждения из газовой среды в тлеющем разряде. Длительность изготовления покрытий составляла 4-5 часов, в качестве установки использовался уникальный собранный нами CVD реактор тлеющего разряда. Процесс химического осаждения из газовой среды плазменным методом заключается в разложении прекурсоров и активации поверхности подложки и ионного ассистирования. Весь процесс можно поделить на 3 этапа: 1) Откачка воздуха и поддержание давления в камере; 2) Поддержание требуем температуры в реакторе и подачи газа в заданной пропорции 3) Осаждение покрытий на образцы. Данная установка безопасна, но требует соблюдение правил безопасности работы в лаборатории ИШНПТ ТПУ.*

Перечень подлежащих исследованию, проектированию и разработке вопросов	*1. Литературный обзор, включающий информацию о видах получения покрытий, особенности каждого метода, видов покрытий на твердосплавных инструментах, применение CVD технологии, а также износостойкость твердосплавных режущих инструментов.* *2. Синтез микрокристаллических алмазных покрытий на концевые фрезы.* *3. Анализ полученных покрытий с помощью фазового анализа, рамановской спектроскопии и наноиндентирования.* *4. Исследование износостойкости концевых фрез без и с покрытием.* *5. Обсуждение результатов исследование и составление выводов.* *Дополнительные разделы: «Финансовый менеджмент, ресурсоэффективность и ресурсосбережение», «Социальная ответственность».*
Перечень графического материала *(с точным указанием обязательных чертежей)*	*Презентация ВКР в Power Point*

Консультанты по разделам выпускной квалификационной работы
(с указанием разделов)

Раздел	Консультант
Финансовый менеджмент…	*Скаковская Наталия Вячеславовна, доцент ОСГН ТПУ*
Социальная ответственность	*Горбенко Михаил Владимирович, доцент ИШНКБ ТПУ*
Английский язык	

Названия разделов, которые должны быть написаны на иностранном языках:
Введение - Introduction
Литературный обзор - Literature review
Экспериментальная часть – Experimental Section
Финансовый менеджмент – Financial management
Социальная ответственность - Social responsibility

Дата выдачи задания на выполнение выпускной квалификационной работы по линейному графику	

Задание выдал руководитель:

Должность	ФИО	Ученая степень, звание	Подпись	Дата
Старший научный сотрудник НПЛ ИШНПТ ТПУ	Линник С. А.	к. т. н.		

Задание принял к исполнению студент:

Группа	ФИО	Подпись	Дата
4БМ7И	Халафов Рустам		

ABSTRACT

Final qualification work 100 pages, 33 figures, 18 tables, 70 references, 1 application.

Key words: Chemical vapor deposition, CVD, diamond, diamond coating, polycrystalline diamond coating, hard alloys, wear resistance, cutting tools, mills.

The object of study is polycrystalline diamond coating on hard alloy cutting tools based on WC-Co, XRD analysis, Raman spectroscopy, nanoindentation, wear resistance.

The goal of this research is to study wear resistance of polycrystalline diamond coated end mills and uncoated end mills, comparison results and also to create the new reactor for deposition based on CVD methods.

In this research were also carried out XRD test, Raman spectroscopy, SEM and nanoindentation tests for proving of obtaining polycrystalline diamond coatings on end mills, wear resistance tests.

As a result of research, create AC glow discharge CVD reactor for diamond coating deposition, diamond coatings on end mills were obtained and made a comparison of wear resistance between uncoated and diamond coated end mills.

Basic structural, technological and technical-operational characteristics: the characteristics of the investigated diamond coatings need to be clarified by additional experiments.

Implementation degree: high.

Application area: aerospace industry, aircraft industry, automotive industry, composite machining, graphite and carbon fiber machining, optical application etc.

Economic efficiency / significance of the work: According to the results of the research, the tasks were fulfilled. However, since this research is related to prospecting works, it is premature to evaluate its effectiveness. Efficiency can only be determined after carrying out applied research, the result of which will be the production of the final product.

In future we are planning to increase properties of obtained diamond coating.

Content

Introduction

Nowadays the high hardness and wear resistance qualify diamond coatings for tool applications are investigated. CVD diamond coated hardmetal tools have similar properties to PCD (sintered high-pressure diamond) but the production costs of CVD diamond are significantly lower. Simple WC–Co hardmetal is composed of WC and a Co binder phase. The binder phase dissolves various amounts of W and C depending on the C balance in the hardmetal alloy [1]. Hardmetal tools are produced by sintering between 1300 and 1500 °C, depending on the alloy composition. For example cutting tool, which are used for composites, graphite etc. They are being increasingly employed in the automotive and aeronautical industries due to their favorable strength to weight ratio. They are used for example in the production of engine housings and blocks, cylinder crank cases and cylinder heads. The hard, abrasive particles contained in the soft composite matrix places significant challenges on the tool in the machining of these alloys however. Diamond coated tools have proven themselves to be suitable for this machining task due to their high hardness and excellent thermal conductivity. Wear-resistance coatings for cutting tools are used for improving performance and increasing productivity [1]. They can reduce friction on the contact surfaces of the tool, increase the hardness and wear-resistance of the tool's surface layer, to increase thermal insulation and reduce the flow of the heat into the depth of the tool. Due to these properties [2]:

- Increasing the hardness of the surface layer makes it possible to expand the field of application of tools from the point of view of processing materials with high hardness, for example, hardened steels with hardness up to 70 HRC.
- Increased wear resistance reduces tool costs;
- Reducing cutting forces allows to increase feed rate;
- Increasing heat resistance and changing the heat balance in the cutting zone allows varying cutting modes, enhancing productivity, which

reducing the cost of products. In addition, the possibility of "dry" processing;

- Reducing friction and heat dissipation allows you to increase the cutting speed, and also reduces the adhesion of the processed material to the tool surface, which improves the quality of the machined surface.

In addition to these advantages there are also some drawbacks for tools with coatings. However coating leads to increase the radius of curvature of the cutting edge. Therefore, it is necessary to use an uncoated tool or a thin-coated tool specifically designed for such materials to process highly viscous materials.

In this research we create a new AC glow discharge plasma CVD reactor, because of the hard geometry of substrates (mills) and find optimum region for obtaining microcrystalline diamond (MCD) coatings on WC-Co end mills. Nanohardness, Raman spectra and XRD analyses were also carried out. Wear-resistance properties were investigated both for initial and diamond coated end mills.

1. Literature review

1.1 Methods and equipment for applying coatings

The composition and properties of wear-resistant coatings largely depend on the technology of their application. Methods for creating such coatings by deposition divided into physical (PVD) and chemical (CVD). Within these two groups there is a fairly large number of coating methods, including combined or supported or activated methods process from other sources of energy [3].

As the name of the processes used (PVD and CVD), they are based on essentially different phenomena. The end result in both cases is precipitation from the gas phase of the coating material on the substrate [4].

During physical deposition (PVD), the coating material changes from a solid to a gas phase as a result of evaporation due to thermal energy or as a result of sputtering due to the kinetic energy of the collision particles of material. The energy, distribution and density of a particle stream are determined by the deposition method, the process parameters and the shape of the particle source. PVD coating is carried out at temperatures up to 450° C, which practically does not impose restrictions on used materials to be coated. This is especially important when coating high-speed steel when the process temperature does not exceed the tempering temperature of hardened steel (about 550° C). PVD processes are carried out in vacuum or in the atmosphere of the working gas with sufficient low pressure (about 10-2 mbar). This is necessary to facilitate the transfer of particles from the source (target) to the product (substrate) with a minimum number of collisions with atoms or gas molecules. The same condition determines the obligatory direct flow of particles. The resulting coating applied only to the part of the product that is oriented to the source of particles. The deposition rate depends in this case on the relative location of the source and material [5-8].

For uniform coating, it is necessary to systematize the movement of the material or the use of several definitely located sources.

At the same time, since the coating is applied only on surfaces “in the line of sight of the source” the method allows selectively coat only certain parts surface, leaving others without a layer applied. It is absolutely impossible when using the chemical method precipitation. The main factors determining the quality of the coating deposited by the method of physical deposition are the purity of the starting materials and the reaction gas, as well as the required level of vacuum [9-11].

There are various variations of the physical method depositions, the main of which will be discussed below.

The method of chemical deposition (CVD) has practically no restrictions on the chemical composition of the coatings. All particles can be deposited on the surface material. What coatings are formed depends on combinations of materials and process parameters. If the process takes place when the space is filled with a reactive gas (oxygen, nitrogen or hydrocarbons), as a result of the chemical reaction between the atoms of the deposited metals and the gas molecules, oxide, nitride and carbide coatings are deposited. Coating composition depends on the partial pressure of the gas and the deposition rate of the coating [12].

When using the CVD method, chemical reactions occur in close proximity or on the surface processed material. In contrast to PVD processes in which solid coating materials are transferred to the gaseous phase by evaporation or spraying, during the CVD process, a mixture of gases is fed into the coating chamber, and the required chemical reactions require temperatures up to 1100 ° C. This condition significantly limits the number of materials for which CVD coating can be applied. If hard alloys withstand such heating with almost no consequences, the heat-treated high-speed steels lose as a result leave their properties [13-15].

CVD processes occur at pressures of 100–1000 Pa. The coating is applied to the entire surface of the product. There is no need to rotate the product as with the PVD method. This is one of the benefits of CVD [16]. To get the same properties of the entire coating in the volume of the working chamber (especially large) it is necessary to ensure optimal gas flows.

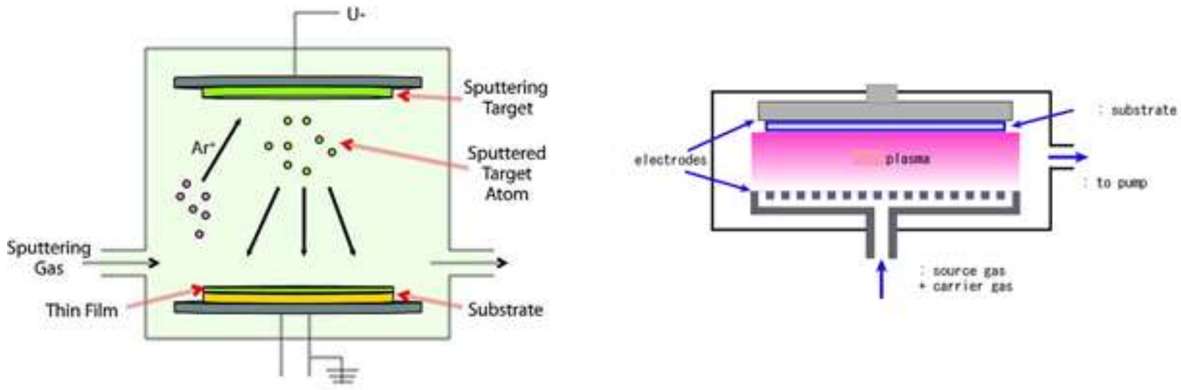
For this purpose, special gas supply systems are used, the so-called gas shower. CVD installations, as a rule, have rather large dimensions. To prevent dangerous emissions of gases into the atmosphere, a special filter system is used. Due to the high temperature of deposition, which provides partial diffusion of the applied material into the substrate, CVD coatings are characterized better adhesion (adhesion) [17].

Scope of the two main methods of application coatings are determined by their above specified properties. CVD processes take place at high temperatures and higher pressure. As a result, the method is absolutely unsuitable for creating coatings on high-speed steel products. Even for hard alloys such temperatures lead to negative consequences - in the surface layer, a decrease in the viscosity of the alloy with a coating of compared to uncoated hard metal [18-21].

This is a result of decarburization of the boundary zone and the formation of the so-called solid alloy phase - a brittle surface zone of 3–5 microns thick. To reduce the harmful effects of temperature on the properties of a hard alloy, a CVD coating method has been developed at temperatures around 800 ° C, which is called the CVD medium temperature method (MT-CVD) as opposed to the high temperature method (HT-CVD). The method allowed to reduce the decrease in viscosity, but did not solve the completely existing problem [22-24].

The appearance of gradient hard alloys with variable the depth of the composition and the application of multilayer coatings can compensate for the decrease in the viscosity of the alloy under the influence of temperature.

PVD and CVD methods also differ in the type of internal stresses in the layer of the applied coating. With the PVD method there are compressive stresses, and with the CVD method -stretching. Tensile stresses improve adhesion of the coating and the substrate. It is also necessary to take in attention to the fact that CVD methods are less sensitive to the quality of material preparation before applying to its coating, while with the PVD method, the material must undergo a long-term multi-stage cleaning, otherwise the properties of the coating cannot be guaranteed [25-29]. There is a difference between PVD and CVD processes comparison is shown in figure 1.



a) b)

Figure 1 – a) PVD and b) CVD processes comparison

As a result of the indicated differences of the two methods, their fields of application were determined. The chemical method is used for coating carbide inserts used primarily for turning. Such plates are available in large quantities and can load CVD installations. The important role is also played by the lack of long-term surface preparation and the need to move the product during the coating process. According to German tool companies, in 2000 more than 70% of the turning plates were produced with a CVD coating.

Milling plates are more sensitive to the possible decrease in viscosity in the surface zone due to constant work with variable loads, and here the proportion of plates with CVD coated below [30].

The market for creating coatings is divided between CVD and PVD methods as follows. High-speed steel coatings are applied only using PVD method. For other tool materials (hard alloys, ceramics and superhard materials) the share of instruments with CVD coating in 2000 was about 41%, the share of tools with PVD coating - 12%. In 2005, as expected, the proportion of PVD coatings should have increased to 15% (CVD: 38%).

Recently, another variation of the CVD method was developed, which made it possible to reduce the coating temperature to almost the temperatures used in the PVD method, called P-CVD (from the words "plasma" and CVD). In practice, the method is a combination of two main methods, since the application of a CVD coating method occurs in plasma environment (as with PVD).

Some diamond properties are show below [31]:

- Extreme mechanical hardness (~ 90 GPa);
- It is one of the most durable materials with the highest volumetric modulus (1.2 x 10^{12} N / m2) and the lowest compression ratio (8.3 x 10^{-13} m2 / N;
- The highest value of thermal conductivity at room temperature (2 x 10^{3} W / m / K);
- The coefficient of thermal expansion (CTE) at room temperature (0.8 x 10^{-6} K) is comparable to the CTE of Invar;
- Wide bandwidth of optical radiation from deep UV to far IR;
- This is a good electrical insulator (dielectric) (resistivity ~ 10^{16} Ohm·cm at room temperature);
- When doping a diamond, its resistivity can vary in a wide range from 10 to 10^{6} Ohm · cm, which turns it into a wide-gap semiconductor with a band gap of 5.4 eV;
- It has high chemical anti-corrosion properties;
- It is biologically compatible;
- Shows low or "negative" electronic affinity.

1.2 Chemical vapor deposition process of obtaining diamond coatings

The process of chemical vapor deposition, as its name implies, involves a chemical reaction in the gas phase that takes place above the surface of a solid substrate, as a result of which the final reaction product precipitates onto the surface of this substrate. All CVD methods for creating diamond films require a method of activating carbon-containing molecules of the original reaction product. These methods include thermal (for example, with a hot wire) or plasma method (glow discharge plasma, high-frequency plasma, microwave plasma) or the use of plasma combustion (oxyacetylene, or plasma torches) [32]. In figure 1.2 and figure 1.3 are shown two of these most popular experimental methods and some typical operating conditions.

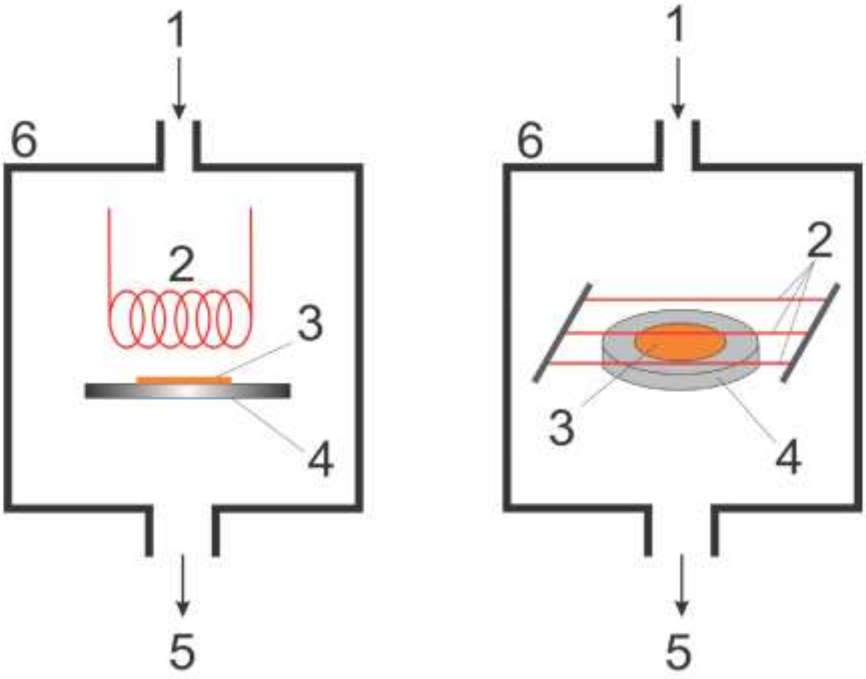


Figure 1.2 - Examples of the two hot wire types of CVD reactors

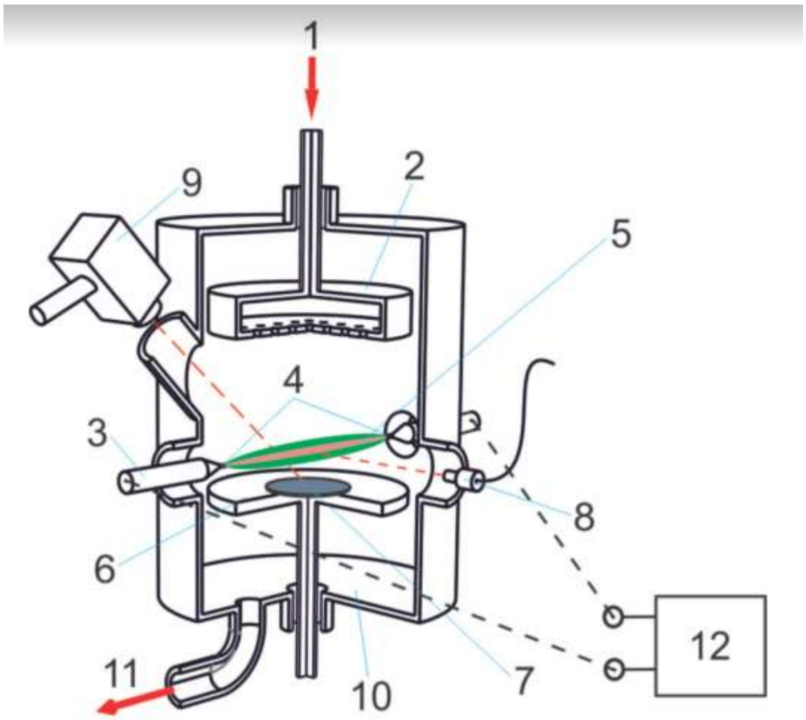


Figure 1.3 - Schematic illustration of experimental AC glow discharge CVD reactor

Since each method is different in details, they are all divided according to common features. For example, the growth of a diamond film (rather than the deposition of other, not so clearly defined, forms of carbon) usually requires that the substrate be maintained at a temperature in the range of 1000–1400 K and that the source gas is diluted with an excess of hydrogen.

The resulting films are polycrystalline with morphology sensitive to exact growth conditions (see below). Growth rates for different deposition processes can vary considerably. Higher growth rates can usually be achieved only due to the corresponding loss of film quality. “Quality” is understood here as a subjective factor. It is considered as a measure related to the ratio of the amount of sp3-bonded carbon (diamond) to the amount of sp2-bonded carbon (graphite) in the sample, the composition of the sample (for example, the content of C-C bonds relative to the number of C-H bonds) and its crystallinity. In general, methods of deposition of diamond coatings by burning hydrocarbons are characterized by high film growth rates (usually 100–1000 μm / h), but the film often grows only in very small local areas and with poor control of the deposition process, which leads to low-quality diamond films [33-35].

On the contrary, the deposition of diamond films by the methods of hot wire or plasma-chemical gas-phase deposition has much slower growth rates (0.1–10 μm / h), but in this way high quality films are obtained. One of the big questions facing researchers in CVD-diamond technology is the need to increase growth rates to economically viable values (hundreds of microns per hour or even millimeters per hour) without degrading the film quality. Some progress has been made in this direction through the use of reactors with film deposition in microwave plasma, since it was found that the performance of the deposition process is approximately linearly dependent on the applied power of the microwave generator.

Currently, the typical rated power for a microwave plasma reactor is about 5 kW. It is expected that the next generation of such reactors will have power ratings up to 50–80 kW. This gives a much more realistic performance of the diamond film deposition, but the cost of the process, of course, increases. Thermodynamically, graphite, rather than diamond, is a stable form of solid carbon at room temperature and atmospheric pressure. The fact that diamond films can be formed by CVD methods is inextricably linked with the presence of hydrogen atoms, which are formed as a result of the "activation" of hydrogen gas, either thermally or as a result of electron bombardment. These hydrogen atoms are believed to play a crucial role in the process of plasma-chemical deposition of diamond films:

- Hydrogen atoms are responsible for the cleavage reactions of stable hydrocarbon molecules in the gas phase, which result in the formation of highly reactive carbon-containing radical fractions. This is important because stable hydrocarbon molecules do not react to initiate diamond film growth. Reactive radicals, especially methyl, CH3, can diffuse to the surface of the substrate and react to form the C – C bonds necessary to build up the diamond lattice;
- Hydrogen atoms complete the “dangling” carbon bonds on the growing surface of the diamond film and prevent them from forming cross-links, leading to the creation of a graphite-like surface;
- Hydrogen atoms etch both diamond and graphite, but under typical film growth conditions in a plasma-chemical reactor, the growth rate of a diamond film exceeds the etching rate, while for other forms of carbon (graphite, for example), everything happens with exactly the opposite. This is believed to be the basis for the preferred deposition of diamond rather than graphite.

One of the main tasks that attracts much attention is the study of the mechanism of heteroepitaxial growth, that is, the initial stages of deposition, during which diamond originates on non-diamond substrates. Several experiments have shown that "pre-abrasive treatment" of non-diamond substrates reduces the time of onset of the nucleation of diamond grains and increases the density of nucleation sites [36]. This inevitably causes an increase in diamond growth rates, since the formation of a continuous diamond film is essentially a crystallization process that occurs through the nucleation of nuclei accompanied by the three-dimensional growth of numerous microcrystallites until they finally merge into a continuous film (Figure 2) [37].

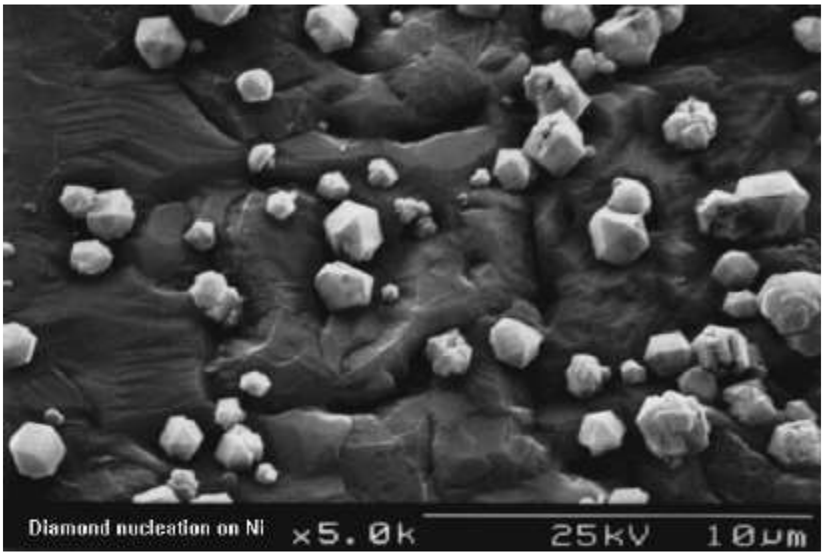


Figure 2 - Diamonds are initially formed as separate microcrystals, which gradually grow until they are joined into a continuous film. This image, obtained in a raster electron microscope, shows small diamond crystals that originate on the Ni surface

The abrasive process is usually carried out by polishing the substrate with an abrasive paste (usually diamond powder with a particle size of 0.1–10 μm in powder), either mechanically or with ultrasonic excitation. Regardless of the abrasive processing method used, the need to create defects on the surface before deposition of a diamond film in such a poorly specified way may prevent the use of the CVD diamond deposition method in some applications, for example, in the electronics industry, where the geometry of the chip elements often has submicron sizes.

This problem has led to the search for more manageable methods of enhancing nucleation, for example, using ion bombardment. This method is often used in a microwave growth reactor: a simple application of a negative bias of several hundred volts to the substrate allows ions to (i) bombard the surface, (ii) penetrate the grid, and (iii) form an intermediate carbide layer.

1.3 Growing diamond films by plasma-chemical method

The surface morphology of the film, obtained during its CVD growth, strongly depends on the ratio of the components of the gas mixture and the substrate temperature [38]. Under "slow" growth conditions — a low partial pressure of methane CH_4, and a low substrate temperature — a microcrystalline film is obtained with the most pronounced triangular faces along with well-visible twin boundaries (Figure 3) [39]. With an increase in the relative concentration of CH_4 in the initial gas mixture or with an increase in the temperature of the substrate, the faces of the grains begin to dominate, both square and rectangular in shape. The cross section of such a microcrystalline film shows that growth has a predominantly columnar character (Figure 4) [40]. At even higher CH_4 partial pressures, the crystal morphology completely disappears and the film shown in Figure 2 begins to grow. Figure 5, which is a collection of diamond nanocrystals and disordered graphite.

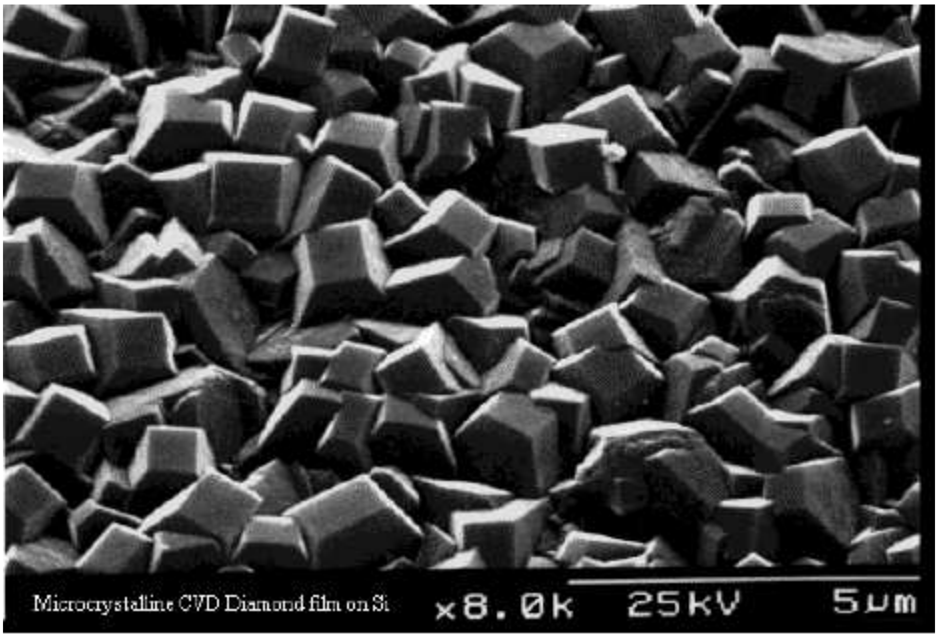


Figure 3 - Typical form of a microcrystalline CVD diamond film on silicon. This film is polycrystalline with the appearance of twinning and numerous crystal defects

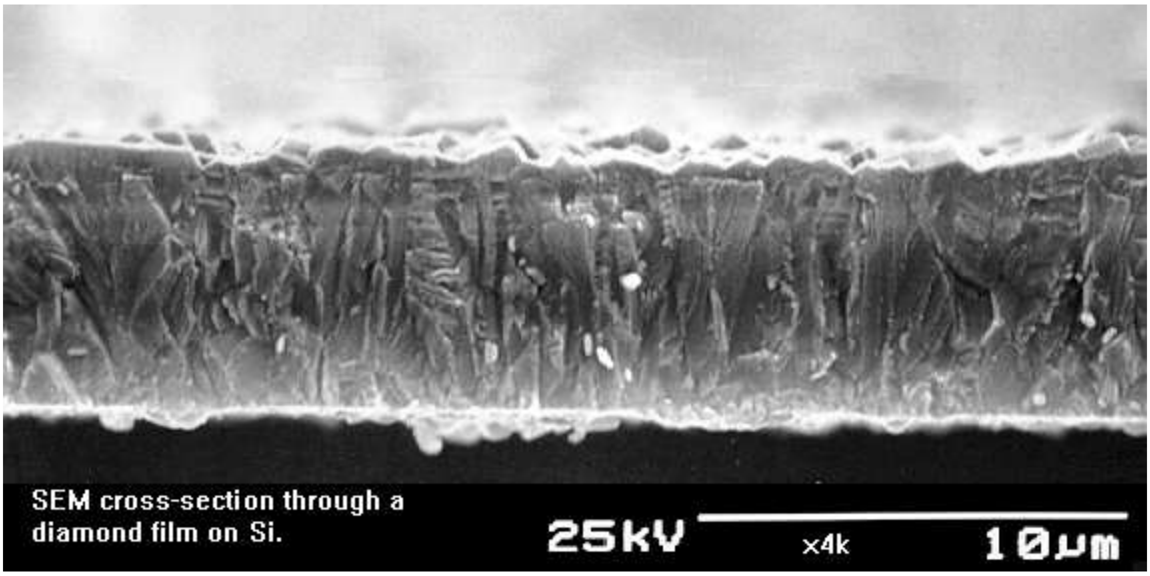


Figure 4 - Cross section of a diamond film with a thickness of 6.7 microns on a silicon substrate, demonstrating the columnar nature of film growth

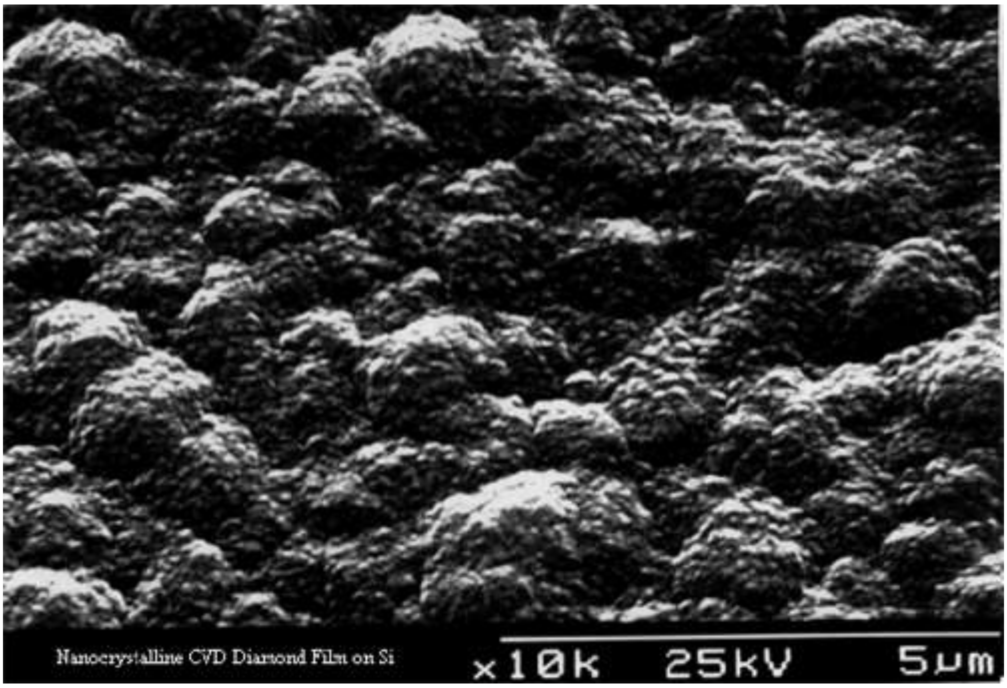


Figure 5 - Nanocrystalline film grown at high (> 2%) methane concentrations. This film has a much smoother surface than a microcrystalline film, but its mechanical and electrical properties are not extreme

Obviously, the crystalline morphology of CVD diamond films is particularly important when the film is made for specific possible applications. A film similar to the one shown in figure 3, can be used as a purely abrasive coating, but most applications of diamond films relate to optics, in particular, to devices for heat removal, and microelectronic devices require that the surface of the films be as smooth as possible. At least two ways of achieving this goal can be foreseen: either the researcher must determine the growth conditions that naturally lead to the formation of smooth films, or optimize the methods of “polishing” the surface irregularities of the film, which is obtained in the form in which it grows. Both concepts are currently the subject of an intensive research program.

Using a technique called “nucleation at substrate displacement,” in which an offset of -100 is applied during microwave deposition of a diamond film between the substrate and the plasma; it is possible to grow diamond films showing some degree of correlation with the crystalline orientation of the silicon substrate. This procedure makes it possible to grow "oriented" or "textured" films in which diamond crystallites grow mainly along the (100) direction. This makes it possible to get closer to the ultimate goal of epitaxial growth of single-crystal diamond films necessary for high-quality electronics.

1.4 Current and future applications of diamond films

Gradually, a large number of applications for synthetic CVD diamonds are beginning to emerge.

Materials for heat sinks. Natural diamond has a thermal conductivity of about four times greater than that of copper, while being a dielectric. Therefore, to our small surprise, the CVD diamond is currently positioned on the market as a material for the heat sinks of laser diodes and for small microwave integrated circuits.

A natural extrapolation of this application is to manufacture chips with great speed, since active devices mounted on a diamond substrate can be better packaged without overheating. You can also expect an increase in reliability, since the temperature of the thermal transition when installing the chip on a diamond substrate will be lower [41]. CVD diamond heat sinks are shown in figure 6.

Figure 6 – CVD diamond heat sinks

Cutting tools. CVD diamonds are also used as an abrasive or coating on the inserts of the cutting tool. Various cutting and drilling tools such as drill heads, reamers, countersinks with synthetic CVD-diamond coatings, etc. is now commercially available for machining non-ferrous metals, plastics and composite materials. Initial tests have shown that tools with CVD diamond coatings have a longer service life, provide higher cutting speed and better finishing than conventional tungsten carbide inserts [42]. CVD diamond on cutting tools is shown in figure 7.

Figure 7 – CVD diamond on cutting tools

Wear resistant coating. In both previous applications, CVD diamonds perform a task that could be performed equally well with natural diamonds, if you ignore economic considerations. However, there are many other applications, both market and near-market, in which CVD diamond coating offers completely new possibilities. Wear resistant coatings are one such application. The ability to protect mechanical parts using superhard coatings, for example, in a gearbox, engine, and transmission, can allow to greatly increase the service life of the nodes with reduced lubricant consumption [43].

Optics. Because of its optical properties, diamond begins to find application in optical nodes, especially as a protective coating for infrared (IR) optics in adverse environmental conditions. Most IR windows are currently manufactured from materials like ZnS, ZnSe, and Ge, which, with their excellent IR transmittance characteristics, have the disadvantage of their increased brittleness and ease of damage. A thin protective barrier from a CVD diamond film may be a solution to this problem, although it is more likely that in the future IR windows will consist entirely of diamond films that will be made up to several millimeters in thickness using improved high-speed growing methods [44]. Optical grade polycrystalline CVD diamond is shown in figure 8.

Figure 8 - Optical grade polycrystalline CVD diamond

However, the main issue with using polycrystalline CVD diamond films for optics is the flatness of their surface, since the rough surface causes weakening and scattering of the transmitted IR signal with subsequent loss of image resolution. Consequently, there is interest in the methods of smoothing the surface of a diamond film, which were very briefly mentioned earlier.

Electronic devices. The possibility of doping a diamond by introducing impurities into it and, thus, changing its properties from a dielectric to a semiconductor opens up a whole range of potential applications of diamond films in electronics. However, there are many serious problems that require solutions to create electronic circuits based on diamond films. The principal thing here is the fact that CVD diamond films are polycrystalline and, therefore, contain grain boundaries, twins, packaging defects and other defects that reduce the service life and mobility of carriers. Active devices made using homoepitaxially grown diamond films on substrates of natural or synthetic diamonds have already been demonstrated, but to date there have been no confirmed reports of successful growth of instrument-quality heteroepitaxial diamond films on non-diamond substrates. This circumstance remains the main limiting factor in the development of microelectronic devices on diamond films [45]. Nevertheless, the degree of influence of grain boundaries and defects on electronic carriers in the best polycrystalline diamond films is to be determined and it is clear that this still does not exclude finding a method for manufacturing active diamond devices.

Composite materials. Another interesting development in diamond technology is the possibility of deposition of CVD diamond films on the outer surfaces of metallic wires or non-metallic fibers (Figure 9). The fibers with diamond coating had elastic modulus values close to those expected for diamond, which makes such fibers extremely rigid for their weight. If growth rates can reach commercially viable levels, such diamond fibers can be used as reinforcing components in metal matrix composites, making it possible to create stronger, more rigid and lighter elements of supporting structures, produced, for example, for space applications [46].

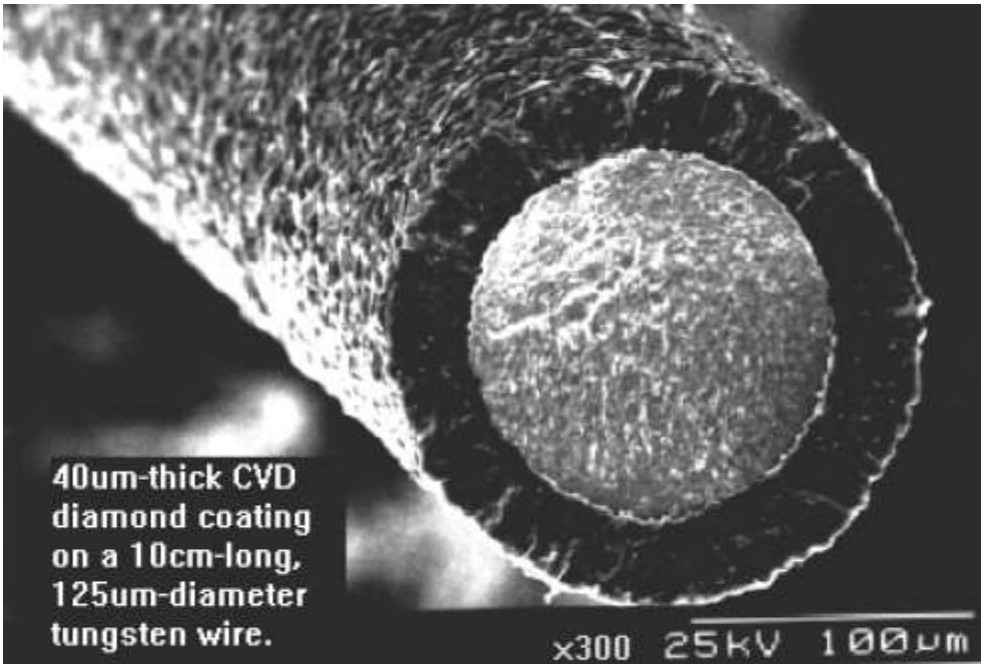


Figure 9 - Diamond coated tungsten wire. The metal core has a diameter of 125 microns with a thickness of diamond coating of approximately 40 microns

In addition, the etching of the metal core coated with diamond wire using appropriate chemical reagents makes it possible to produce all-diamond tubes or hollow diamond fibers. They also have potential applications for increasing the strength of supporting structures, since such hollow diamond tubes can act as pipelines for the supply of fillers, refrigerants, or sensors placed in a reinforced casing. Two-dimensional braided coatings or plexuses of diamond-coated fibers have also been proposed as reinforcing elements.

1.5 Wear-resistance coatings on cutting tools

A Cutting Tool or Cutter is a tool used to create inserts or remove any remaining material of a workpiece by clearing out the excessive deformity. Cutting Tools are pointed and are mounted on various machines tools to be used in the process of cutting. Cutting Tools are always harder than the material they are used to cutting. The Cutting Tools used for work should also withstand the heat released in the process. Cutting Tools are of classified according to two types:

Single Point Tools - The tools which make use of a single sharp cutting edge to remove deformities are known as Single Point Tools. The act of turning, the exact opposite of boring, makes use of Single Point Tools as it works on the exterior diameter of a cast hole. The sharp tip of a Single Point Tool is usually round, forming a nose radius [47].

Multiple Point Cutting Tools - Multiple Cutting Tools have more than one cutting edge to them and are used for various different purposes. A Multiple Cutting Tool is mounted on a machine and used by utilizing the tool in a rotation motion. The activities of drilling and mining make use of Multiple Cutting Tools. Types of cutting tools are shown in figure 10.

Figure 10 – Types of cutting tools

Tools classified by their material properties:

- **Carbon Steel**: This material is amongst the lowest grade of tools and falls in the family of low-grade alloys. A tool made from Carbon Steel has is hard, tough and has strength when it is hardened at a certain temperature. Carbon Steel Tools are suitable to be cut with at a lower cutting speed as above a temperature of 180^{o}C, the Carbon starts melting [48]. This limits this type of tool to lower speed machines in turn, rendering them unable for metal cutting. The materials it is composed of are cheap, easily available and comfortably forged. Carbon Steel tools have a hardness of about 62 RC and usually opted for working with wood;

- **High-Speed Steel**: HSS has a higher resistance and hot hardness than Carbon Steel because of its material composition. Tools made from High-Speed Steel can be used for Metal cutting at a speed of about 2 to 3 times faster than Carbon Steel Tools. HSS tools can cut through a metal comfortably and can perform high-speed cut with faster metal removal rate. The melting point of this steel is about 900^{o}C [49]. Cutting Tools made with High-Speed Steel are suitable for interrupted cuts on metals using different machines and processes. One of the most significant indicators of using of the cutting tool is ability to maintain its functional parameters for a long time. By ensuring an increase in the efficiency of the tool it is possible to significantly increase the productivity of mechanized labor, thereby reducing the cost of purchasing a new tool and saving on other accompanying technological components;
- **Cast Alloys**: Cast Alloys were introduced in the early 1900's and were in regular use since then. They have a maximum hardness value of about 55 – 64 Rc. Cast Alloys have a better resistance than its toughness, lower comparatively to HSS. Cast Alloys can be used at a slightly higher speed than High-Speed Steels [50]. These tools maintain their hardness from up-to 760^{o}C and they are highly alloyed, which makes them brittle and susceptible to damage. Tools made from Cast Alloys are now limited in use;
- **Carbides**: Carbides or Cemented Carbides are materials that have a high Hot Hardness over various temperatures, high thermal conductivity and a high Young's Modulus making them a suitable material for manufacturing cutting tools. Usually, Carbides are made of Tungsten powder and Carbon, mixed in a ratio of weight- 94: 6. Then the next step involves sintering it with Cobalt at high temperatures. There is a wide range of grade of Tungsten Carbide available in the Market. The addition of Cobalt gives

increased hardness, depending on the amount present in the mixture. Tungsten Carbide has a higher wear resistance than Tungsten [51]. Typical cutting speed is of - 30 – 150 mm; when coated it is about 100 – 250 mm. There are three main types of Carbides- Straight Tungsten Carbide, Titanium Carbide or Tantalum Carbide & Composite Carbides. Straight Tungsten Carbide is strong and has a high resistance. Titanium Carbides help in reducing the chip rate of a tool and help in improving the hot hardness.

The Hardest Cutting Tool is Diamond. Diamonds are the hardest substance known to man yet they are comparatively brittle. Traditionally, Single Diamond Tools used for the purpose of cutting. Nowadays, machines make use of Poly-crystalline Diamonds as a replacement. Though Diamonds are costly and are hence not used a lot to make tools, other than specific industries.

During operation of the cutting tool, the main load is transferred to its working part, this leads to partial wear or complete destruction of the planes and cutting edges. There are a number of technologies for the treatment of working surfaces, which gives them additional hardening, the most effective is the method of applying special coatings to the surface of the cutting tool. Improving the performance of the cutting tool can be achieved by improving the properties of the surface layer of the tool material, in which the working surface of the tool most effectively resists of characteristic types of wear. Such a material should have a significant margin of strength in bending, compression and withstand shock loads. One of the most important problems of tool durability is that after deformation of the tool it is sent to sharpen, before sharpening, the coating layer is removed.

This leads to reduced tool life. The solution to this problem: secondary coating. Coatings are available in two methods:

- Chemical deposition method (Chemical Vapor Deposition - CVD)
- Physical Vapor Deposition method (PVD).

Industrial applications received PVD-methods of applying protective coatings on the cutting tool. This is due to the fact that the deposition of PVD coatings using an arc or glow discharge (magnetron) has a higher productivity and is not so sensitive to minor deviations of the technological parameters. CVD technologies involve the use of expensive high-purity chemicals ($TiCl_4$, NH_3 etc.) and precise control of the products of chemical reactions in the working chamber [52].

The variety of methods of physical deposition of wear-resistant coatings used at present is reduced to evaporation or ion sputtering of titanium or its alloys, ionization and heterogeneous reaction of atoms and metal ions and reaction gas on the instrument surface, leading to the formation of nitride, carbide, carbonitride. The structure and adhesion of the tool coating, and their cutting properties predetermine many parameters: optimization of the coating temperature, increasing the degree of ionization, the velocity and flux density of the sprayed particles, the configuration of the tooling, the use of ionic cleaning of the substrate, accelerating stresses, various modes of deposition etching or doping and many other features determine the structure of the coatings themselves and the structure of the interfacial boundary "coating - substrate."

It should be noted that the concept of "multi-layered" in many cases is quite arbitrary, since sputtering methods allow to achieve the absence of clearly defined interphase boundaries between the layers, as well as between the coating and the substrate. Multilayer wear-resistant hard coatings have increased crack resistance, improved adhesion, high impact strength, a lower level of internal stresses and stresses at the “coating - substrate” boundary due to equalization of thermal expansion coefficients. For example, a TiCN coating has a multi-layer two-phase structure TiN – TiCN, which increases the strength and viscosity characteristics compared to the coating. In the last decade, various combinations of coatings using thin external solid lubricating coatings (for example, TiAlN / MoS_2) have been developed and are widely adapted to ensure good drainage. shavings. High hard diamond-like coatings (diamond like carbon coatings - DLC) have a low coefficient of friction and high wear resistance. However, they have a serious drawback: a very high level of internal stresses, leading to embrittlement and flaking at high contact loads, which limits the thickness of coatings to 1 micron.

Another problem of such a coating is low thermal conductivity, which can lead to their local graphitization and subsequent leaching. The upper operating temperature limit is limited to 250 ° C, and lubricating coolants (coolant) are required. Coatings of cubic boron nitride (CBN) also have a high level of internal stresses and a coating thickness of not more than 0.1 μm. When machining, maximum efficiency is achieved with the integrated use of high-tech equipment equipped with CNC, and modern cutting tools that provide higher cutting speeds and productivity. The most versatile group of cutting materials that allows the processing of the vast majority of metallic and non-metallic materials. The carbide tool is designed to work with cutting speeds up to 300 m/min (drills 50–70 m/min, the latest developments up to 90–180 m/min); it is used, in the bulk, on machines of foreign manufacturers with a spindle speed of an average of up to 10,000 rpm. To improve the properties (increase in hardness, reduce the radius of rounding of the cutting edge and, consequently, increase the durability of the tool), manufacturers strive to reduce the grain of the alloys [53]. In dry processing, TiAlN coating has proven itself well. This coating allows you to improve adhesion, improve impact strength, reduce friction coefficient and have a high crack resistance. At the same time, coatings increase the radius of rounding of the cutting edge, which adversely affects, for example, when removing a small allowance. Different types of coatings are shown in figure 11.

Figure 11 – Different types of coatings on cutting tools

Some coatings are characterized by high internal stresses resulting in peeling of the coatings. The most applicable coatings on non-refillable tools and plates, because with a regrinding of the coating in areas subjected to sharpening, are completely destroyed. The main types of PVD coatings (characterized by a thickness of 1–3.5 μm, therefore, are used for cutting edges with a small rounding radius, allowing to reduce cutting forces, improve chip breaking, prevent vibrations) and CVD coatings with a thickness of up to 20 μm (Al_2O_3).

The main difference between these coatings consists in the deposition method: physical deposition is used for PVD coatings (400–500 ° C), chemical deposition for CVDs (1000–1100 ° C). Impact mechanical destruction of the tool also adversely affects the operation of coatings, so their effectiveness may be reduced when using imperfect equipment. In addition to wear-resistant coatings (the most common nitride TiAlN, TiAlCrN, TiN) solid lubricating coatings are used, which have a very low coefficient of friction and provide a reduction in cutting forces and chip removal (TiO_2, $WO_3V_2O_5$).

Tools with soldered carbide inserts, or with replaceable carbide knives for face mills, are most common in Russia. Its advantages: simplicity, low cost. The main disadvantage is low productivity, the need for high-quality sharpening. Cutting speed rarely exceeds 100 m / min. Thus, the improvement of coating technology for cutting tools, the development of new modifications of protective coatings can significantly improve the performance of the tool and expand the scope of its effective use.

The application of innovative coatings on the cutting tool is a strategic task. For a number of requirements, the process coating, ultimately, must meet a high degree of wear resistance. Therefore, it must be thermally resistant and firmly adhere to the tool body. The coating is selected based on the type of material being processed and the technology of using a specific tool [54].

More detailed information about types and characteristics of wear-resistance coatings is shown in table 1.

Table 1 – Characteristics of wear–resistance coatings.

Coating	Color	Hardness, HV	Thickness μm	Friction coefficient	Max temperature of using ºC
TiN	Golden-yellow	2400	1-7	0.25	600
CrN-Multi	Silver metallic	2400-2800	1-10	0.2-0.25	600
TiSi	Dark gold	3000	1-3	0.25	600
TiCN	Blue-silver	3200	1-4	0.3	400
TiCN-MP	Copper red	3200	1-4	0.25	400
TiAlN	Purple black	3400	1-4	0.3	700
AlTiN	Black anthracite	3500	1-5	0.3	900
ZrN	White gold	2400	0.5-3	0.3	550
CrN	spectral	2500	0.5-3	0.15-0.2	500
AlTiSi	Brown	3800-4000	1-4	0.4	1100
nACo	Blue	3800-4200	1-5	0.35-0.4	1200
nano DLC	Black	1400-2600	1-5	0.1	400

2. Equipment and methods

Obtaining of the coating

2.1 Plasma - chemical reactor

The main part of the installation is a plasma-chemical reactor, which includes gas distribution system, gas discharge electrode system and temperature-controlled substrate holder with a precise positioning mechanism height. All components of the reactor in the process are under the action of high temperature, so each of them is intensively cooled with running water. Scheme of the installation for the deposition of diamond films in a glow discharge is shown in figure 12.

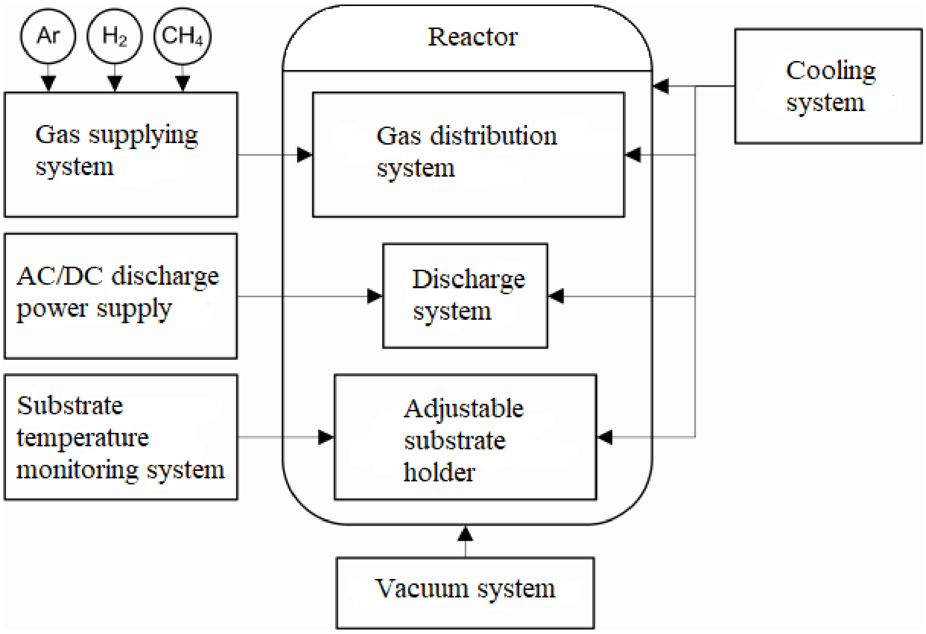


Figure 12 – Scheme of the installation for the deposition of diamond films in a glow discharge

To create the necessary gas environment in the reactor, a system is used in which the gases Ar, H2, CH4 (and, if necessary, others) are mixed in strictly defined proportions and fed into the reactor with strictly defined flow rates. This is ensured by the Bronkhorst EL-FLOW precision mass flow regulators. Volumetric gas flow through such regulators is controlled with an accuracy of 0.01 ml / min.

To evacuate the reactor and maintain the required working pressure in it, a conventional fore vacuum pump is used, providing a residual pressure of not more than $5 \cdot 10^{-2}$ Torr. To adjust the pumping speed, a controlled valve with a digital position indication is used, which ensures constant pressure in the reactor. Pressure is measured by two types of sensors: a convection sensor for pressures below 1 Torr, and a membrane-capacitive sensor for precision pressure control during the deposition process with an accuracy of 0.2 Torr in the range from 1 to 760 Torr.

Discharge burning is provided by a power source that allows you to work as an alternating voltage with a frequency of about 40 kHz, and in constant voltage mode. To control the temperature of the substrate during diamond growth, infrared pyrometry is used. We used an infrared pyrometer Kelvin KB Dipole (measured temperature range: 321-2405^{o} C; resolution - 1^{o} C; measurement time - no more than 50 ms.) [55]. The temperature was measured through a window of zinc selenide. Figure 13 shows a scheme of a plasma-chemical reactor based on a glow discharge.

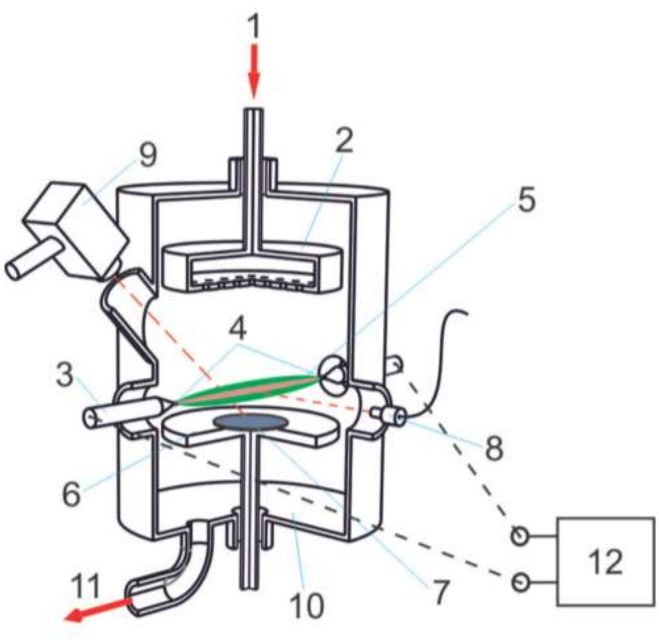


Figure 13 – Scheme of a plasma-chemical reactor based on a glow discharge: 1 – gas mixture supply (Ar, CH_4, H_2), 2 – gas distribution system, 3 – electrode holders, 4 – electrodes, 5 – plasma, 6 – substrate holder, 7 – substrate, 8 – fiber optic receiver of optical emission spectrometer, 9 – optical pyrometer, 10 – vacuum chamber, 11 – pumping out, 12 – AC/DC power supply

The discharge in this system burns between two tungsten electrodes fixed in cooled holders. The interelectrode distance can vary by moving the electrode holders and can reach 20 cm or more. The gas mixture of Ar, H_2 and CH_4 enters the reactor through the gas distribution "shower" system, which ensures a uniform flow of gas over the entire area of the substrate. The substrate is located under the plasma on a water-cooled substrate holder. Both the substrate holder and the gas distribution system can move vertically during the discharge process, and their position is controlled with an accuracy of 0.2 mm. The composition of the plasma is controlled through a fiber-optic receiver of the optical emission spectrometer [56].

In the process of mining the diamond deposition process, various features of the discharge behavior, gas distribution and heat distribution in the reactor were identified. In this regard, during the research 3 modifications of plasma-chemical reactors were created. Figure 14 shows all of these modifications.

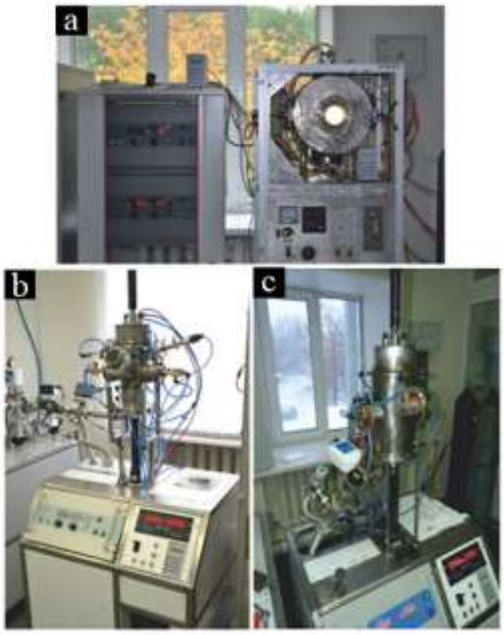


Figure 14 - Photos of machines for the deposition of diamond films with different reactor modifications: a - a reactor with an open discharge system fixed the position of the substrate holder and gas distribution system, b - reactor with open discharge system, the ability to change the height of the substrate holder and gas distribution system, c - a reactor with a closed discharge system, possibility of changing the height of the substrate holder and gas distribution system

Table 2.1 – Main parameters of various reactor modifications

Parameters	Reactor modifications		
	a	b	c
Max. discharge power, kW	3	6	9
Max. discharge current, A	10	20	30
The position of the substrate holder during deposition	Fixed	Changeable	Changeable
The position of the gas distribution system during deposition	Fixed	Changeable	Changeable
Max. interelectrode distance, cm	22	14	12
Max. deposition area, cm^2	60	28	28
The average growth rate of polycrystalline diamond film, micron/h	2	2,5	4
Argon electrode protection	Missed	Missed	Attended

Preparing of the surface

2.2 Sandblasting surface preparation

During abrasive blasting, abrasive particles are accelerated from the abrasive blasting apparatus with the help of compressed air energy. When cleaning, unnecessary materials are removed, the surface of the material is hardened and becomes prepared for coating. With the help of abrasive blasting from metal structures remove old paint, rust and other contaminants. In addition, when jet cleaning, secondary dross is removed, this is formed on new steel. Corner abrasive particles impart surface roughness and create a profile, or notch. New, softer types of abrasive (including plastic and wheat starch), as well as special abrasive blasting equipment with low pressure used for the dry method of removing coatings from modern composite materials. This allows you to clean planes, helicopters, cars, trucks and boats without using abrasive blasting, which can disrupt the surface structure. In addition, the transition to the dry method of cleaning the upper layers eliminates the possibility of exposure to working toxic chemicals used in cleaning, and eliminates the costs associated with the disposal of hazardous waste. Sandblasting machine is shown in figure 15.

Figure 15 – Sandblasting machine T06304

Uninhabited sandblasting chambers have the form of boxes that have an opening for placing hands in it and a window with which the cleaning process is controlled. In this case, it is not possible to hire additional staff to stay in the cell. All work is carried out automatically. Sandblasting chambers resemble an injector; to ensure their efficiency, compressed air is needed, which is preliminarily cleaned from oil content.

Sandblasting sand gun is an equipment with the help of which manual cleaning is performed with the help of sand. The scope of its use is most often limited to the construction and automotive industry.

Sandblasting machine consists of:

- Source of compressed air;
- Tanks in which sand is located;
- Handles, which are based on high-strength material;
- A hose or tube that connects all components into a single structure.

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

2.3 Ultrasonic cleaning

Ultrasonic cleaning (US-bath) is a device for disinfection and sterilization of a wide range of objects in certain solvents (or distilled water) using high-frequency ultrasonic waves (from 20 to 400 kHz). Solvents, unlike water, make the cleaning process more efficient. Duration of cleaning varies within 3-6 minutes. Ultrasonic bath is shown in figure 16.

Figure 16 – 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 ... 65 ° C). However, when sterilizing medical devices, it is necessary to maintain the temperature below 38 ° 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.

Analyses of the obtained coating

2.4 Raman spectroscopy

Microspectrometers are usually equipped with high-precision motorized scanning tables for performing two-dimensional (and in the case of a confocal microspectrometer and three-dimensional) pointwise shooting of spectra from a given area (volume) sample. This mode of operation is also called Raman mapping. The result sample measurement in this mode is the Raman atlas or Raman sample image.

Raman spectral imaging is a powerful technique for creating detailed chemical images based on the Raman spectra of the sample. At each point of the sample is taken full spectrum, then on the basis of the array of spectra is pseudo color image containing information on the composition and structure of the material:

- The intensity of the Raman peak allows visualization of the concentration and substance distribution;
- The position of the Raman peak gives an image of the molecular structure and phase, internal stress.

The width of the Raman peak reveals the crystal structure and phase Raman spectral images provide information about a sample that is not can be obtained using traditional optical microscopy. In particular, they can be used to determine:

- Component distributions and grain or particle sizing.
- Changes in the crystal structure or phase in the sample
- Size and shape of particles of impurities
- Interaction and mixing of components at the phase boundary
- Distribution of internal stresses and deformations on the sample.

Thus, based on a single data set, various spectral Raman images that allow the researcher to substantially move beyond what a conventional microscope can see. InVia Basis Raman spectrometer represented in figure 17.

Figure 17 – InVia Basis Raman spectrometer

2.5 Nanohardness determination

Mechanical testing is used to determine properties such as hardness, modulus, fracture toughness or yield strength. Bulk samples typically are examined using uniaxial compression and tensile testing to acquire elastic modulus data which requires days of sample preparation and testing. Hardness test methods use an indenter probe that is displaced into a surface under a specific load. In traditional testing, the size or depth of indentation is measured to determine hardness leading to user bias in the data. Microhardness testing is an industry standard for quality and process control for hardness data. Microhardness testing, with applied loads under 10 N, is typically used for smaller samples, thin specimens, plated surfaces or coatings. Measuring of micro and nanohardness were obtained by MTS nanoindentor G200 which shown in figure 18.

Figure 18 – MTS nanoindentor G200

Nanoindentation has advantages over traditional mechanical testing, providing both elastic modulus and hardness data from a single test. Integrated indentation testing (IIT) automates the indentation process so hundreds of tests with micron spatial resolution can be performed on a small sample size. The nanoindentation tests can be performed as fast as each indent in less than a second making it the fastest mechanical characterization technique. The analyses of the data are automated removing any user bias. Some materials like composites and devices are too complex to apply traditional test methods where nanoindentation has a unique advantage. As dimensions shrink, mechanical properties change as the scale changes from bulk to micron to nanometer and nanoindentation can provide size-dependent properties while accommodating any sample geometry.

Wear resistance research

2.6 CNC machine

Work on the machine can be represented in the form of several stages:

1. Acquaintance with the drawing and the technological map of a detail;
2. Preparation of the workpiece, fixtures, tools and other tooling;
3. Installation, alignment and fixing on the machine workpiece. Drawing up a note;
4. Installation and fixing tool on a machine;
5. Installation in the initial position of the moving parts of the machine, including - parts of the cooling system, fencing etc;
6. The choice of cutting modes: spindle speeds, working feed rates;
7. Turn on the machine and monitor the cutting process;
8. Turn off the machine. Check the dimensions of the part.

Familiarization with the drawing is necessary in order to clearly imagine what needs to be done, from what material, what requirements are imposed on the finished product.

The flow sheet contains all the information on how to process the workpiece on the machine. This includes instructions on what surface should be the reference (base) when securing the workpiece, what surface should be machined, what technological equipment is used (fixture, vice, mandrel, etc.), what tool should be used and with which modes to work and also what sizes and what roughness of a surface have to be reached when processing.

Technological map should be drawn up in detail. It may contain indications of the measuring instrument used, the composition of the coolant, calculated data for processing complex parts, etc.

However, in practice there may be cases when the worker is given a blank or a pre-processed part with a drawing and only the name of the operation is formulated. In this case, the worker himself must determine with which device to fasten the part on the machine, which tool to use, with which measurers to reconcile the part in the clamping device, etc. Once all the issues have been achieved, it is completely clear and as should be done, the worker proceeds to the next stage of work. CNC machine is shown in figure 19.

Figure 19 – CNC machine

Preparing everything you need for work - it means not only collecting all the accessories and tools required for the machine, but also checking whether everything prepared is suitable for work, everything corresponds to the chosen processing method and the expected quality of the part. Used on universal milling and boring machines, a set of fastening bolts, clamping strips, studs and crackers in case of use on coordinate-boring and tool-milling machines is complemented by measuring stands, precise prisms, angles, clamps and some other devices and parts.

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

To student:

Group	Full name
4BM7E	Khalafov Rustam

School	School of Advanced Manufacturing Technologies	**Department**	Material 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":	
1. The cost of scientific research (NI) resources: material, technical, energy, financial, informational and human	*1.Materials costs -12950 rub.;* *Costs for salaries of performers – 180269 rub.;* *Extrabudgetary funds deductions – 54080 rub.;* *Overhead charges – 57327 rub.*
2. Norms and standards of resource use	*2. This research is carried out for the first time, therefore there are no norms and standards for the use of resources.*
3. Used tax system, rates of taxes, deductions, discounting and lending	*3. The coefficient of deductions for payment to extra-budgetary funds - 30%*
The list of issues to be investigated, designed and developed:	
1. Assessment of the commercial potential of engineering solutions (IR)	*1. Analysis of competitive technical solutions*
2. Formation of the plan and development schedule and introduction of IR	*2. Determination of work stages; the definition of labor-intensive work; Gantt graphics development*
3. Justification of the necessary investments for the development and implementation of R & D	*3. Project costing*
4. Budgeting engineering project	*4. This research is conducted within Federal project, so there is no need in investments from others.*
5. Assessment of resource, financial, social, budgetary efficiency of R & D and potential risks	*5. This research is carrying out without any exploitation costs.*
The list of graphic material (with the exact indication of the required drawings)**:**	
1. Evaluation map for comparison of competitive technical solutions *2. Calendar schedule for conducting scientific research*	

Date of assignment for the section on a linear schedule	

Task issued by a consultant:

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

The task was accepted by the student:

Group	Full name	Signature	Date
4BM7E	Khalafov R. D.		

4. Financial management, resource efficiency and resource saving

4.1 Analysis of technical competitive 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).

Polycrystalline diamond coating on cutting tools is analysis object.

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

Today, diamond-coated cutting tools are used primarily for machining non-ferrous materials such as aluminium-silicon alloys, copper alloys, fibre-reinforced polymers, green ceramics and graphite. The tool life of cemented carbide cutting tools is greatly improved by diamond coating, and typically more than 10 times the tool life is obtained. In this report we will present cutting performances of diamond-coated inserts, twist drills, square end mills and ball nose end mills.

Diamond films are usually deposited more than 10 pm thick to make tool life longer, since tool life is directly related to film thickness. However, increased film thickness caused many problems with cutting performance. The most severe problem was the decrease in the transverse rupture strength of the diamond-coated substrates. Because of this effect, the diamond-coated insert used under high speed and intermittent cutting conditions.

Table 4.1 - Evaluation chart for comparison of technical competitive solutions.

Evaluation criterion	**Weight of criterion**	**Mark**			**Competitiveness**		
		B_f	B_{k1}	B_{k2}	C_f	C_{k1}	C_{k2}
1	2						
Technical criterion for resource saving evaluation							
1. Labor productivity increase	0.3	3	2	1	0.9	0.6	0.3
2. Machining accuracy increase	0.1	5	4	1	0.5	0.4	0.1
3. Reduced electricity costs	0.2	4	4	1	0.8	0.8	0.2
4. Reliability	0.3	5	4	2	1.5	1.2	0.6
5. Durability	0.1	3	2	2	0.3	0.2	0.2
6. Ecologically safe	0.1	1	1	2	0.1	0.1	0.2
In total	1	21	17	9	4.1	3.3	1.6
Economical criterion of efficiency evaluation							
1. Competitiveness	0.3	3	2	1	0.9	0.6	0.3
2. Patented technology	0.2	3	5	1	0.6	1	0.2
3. Price	0.1	5	3	1	0.5	0.3	0.1
4. Exploitation period	0.2	4	2	2	0.8	0.4	0.4
5. Funding of scientific research	0.2	5	3	1	0.4	0.2	1
In total	1	20	15	6	3.2	2.5	2

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

$$C = \sum W_i \times B_i \tag{4.1}$$

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».

Labor productivity is one of the main aspects of evaluation of cutting tools. Polycrystalline diamond coating (PDC) obtains better physical and mechanical properties than others, so the price of such cutting tools is much bigger than with

DLC or without coating tool. Durability of such tool a bit more than analogs, however accuracy is not decreasing with time, which allows using them more. There is an opportunity to use diamond cutting tools several times even when the coating is broken dawn.

4.2 Planning of researching of wear resistance of PDC

There are two persons conducting research: scientific supervisor and engineer. Planning of research allows dividing duties between performers, to evaluate employee's salary; moreover it guarantees realization in time. Order and content are shown in table 4.2.

Table 4.2 – Stage's order, division and distribution of performers

Main stages	№	Work content	Performer position
Assignment	1	Drafting and affirming of technical task	S. S.
Research direction	2	Selecting and investigation of them material	Eng.
	3	Choice of research direction	S. S.
Theoretical and experimental research	4	Research of wear resistance of polycrystalline diamond coating on base o WC-Co	Eng.
	5	Selecting materials and methods for research	S. S.
	6	Obtaining diamond coating on cutting tool	Eng.
	7	Wear resistance experiments	Eng.
	8	SEM & REM analysis	S.S.
Results discussion	9	Treatment of obtaining results	Eng.
	10	Results and conclusion scientific explanation	S.S.
Report compilation	1	Development plan of making out Researcl	Eng.

		work	
	2	Research work making out report	Eng.
Report protection	3	Protection of Research work	Eng.

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. The following formula is used to determine the expected (average) value of labor intensity.

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

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

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

$t_{max\ i}$ = the maximum possible complexity of the implementation of a given i-th work (pessimistic assessment: assuming the most unfavorable set of circumstances), pers.-day.

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%.

$$T_{w_i} = \frac{t_{\exp i}}{N_i} \qquad (4.4)$$

where $T_{w\,i}$ - the duration of one work, *days*;

$t_{exp\ i}$ - 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 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. To do this, use the following formula:

$$T_{c\,i} = T_{w\,i} \cdot k_{\mathrm{cal}} \tag{4.5}$$

Where $T_{c\,i}$ - the duration of the i-th work in calendar days;

$T_{w\,i}$ - the duration of the i-th job in working days;

k_{cal} - calendar factor.

The calendar factor is determined by the following formula:

$$k_{cal} = \frac{T_{cal}}{T_{cal} - T_{off} - T_{hol}} \tag{4.6}$$

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):

$$t_{\mathrm{exp\ i}} = \frac{3t_{min\,i} + 2t_{max\,i}}{5} = \frac{3 * 2 + 2 * 7}{5} = 4 \text{ pers.} - \text{days}$$

$$T_{w_i} = \frac{4}{1} = 4 \text{ days}$$

For a six-day working week (for scientific supervisor and engineer) the calendar factor:

$$k_{cal} = \frac{T_{cal}}{T_{cal} - T_{off} - T_{hol}} = \frac{365}{365 - 52 - 14} = 1.22$$

$$T_{cal} = T_w * k_{cal} = 4 * 1,22 = 4,88 \approx 5 \text{ дней}$$

The calculation results are shown in table 4.4.

Table 4.4 – Timing indicators of scientific research

Title of work	Complexity of work						Duration of work in working days $T_{w\ i}$		Duration of work in calendar days $T_{cal\ i}$	
	t_{min}, *pers.-days*		t_{max}, *pers.-days*		$t_{exp\ i}$, *pers.-days*					
	S.	E.	S.	E.	S.	E.	S.	E.	S.	E.
1. Drafting and affirming of technical task	2	-	5	-	7	-	5	-	6	-
2. Selecting and investigation of theme material	-	10	-	15	-	12	-	9	-	10
3. Choice of research direction	6	-	9	-	10	-	8	-	7	-
4. Research of wear resistance of polycrystalline diamond coating on base of WC-Co	-	5	-	9	-	15	-	14	-	18
5. Selecting materials and methods for research	4	-	5	-	4	-	4	-	5	-
6. Obtaining diamond coating on cutting tools	3	-	4	-	5	-	5	-	6	-
7. Wear resistance experiments	-	11	-	23	-	16	-	15	-	16
8. SEM & REM analysis	7	-	4	-	8	-	5	-	8	-
9. Treatment of obtaining results	-	2	-	5	-	3.8	-	4.8	-	4
10. Results and conclusion scientific explanation	-	20	-	22	-	24	-	20	-	24
11. Development plan of making out Research work	-	4	-	2	-	6.2	-	4.2	-	4
12. Research work making out report	-	7	-	6	-	8	-	7	-	6
13. Protection of Research work	-	1	-	2	-	2	-	2	-	2

On the basis of table 4.4 build a calendar schedule (for the maximum duration of the execution of works), table 4.5.

Table 4.5 – Calendar schedule

№	Perf.	$T_{cal\ I}$, cal.d.л.	Duration of work																	
			Dec.		Jan.		February			March			April			May			June	
			2	3	2	3	1	2	3	1	2	3	1	2	3	1	2	3	1	
1	S.S.	6																		
2	E.	10																		
3	S. S.	7																		
4	E.	18																		
5	S.S.	5																		
6	S.S.	6																		
7	E.	16																		
8	S.S.	8																		
9	E.	4																		
100	E.	24																		
11	E.	4																		
12	E.	6																		
13	E.	2																		

- Scientific supervisor - Engineer

Based on the schedule data, table 4.5, it can be concluded that the duration of work of research of wear resistance of polycrystalline diamond coating will take 14.5 decades. The beginning of the project development will be in the second decade of December and will end in the last decade of June.

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

The duration of the project in calendar days is equal to:

– 32 *days* (the duration of the scientific supervisor);

– 84 *days* (the duration of the project by an engineer).

4.5 Cost budget for scientific research

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 group of costs.

– Material costs of project;
– basic salary of the project performers;
– additional salary of project executors;
– extrabudgetary funds deductions (insurance deductions)
– Overhead charges.

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 4.6 – Material costs

Name	Unit of measurement	Amount	Price per one, *rub*	Materials costs C_m, *rub*
Mills	Piece	20	10000	200000
Graphite	Kilogram	5	1000	5000
CH_4 gas	m^3	5000	20	10000
A4 paper (500 sheets)	package	1	200	200
Tungsten wires	Coil	3	4200	12600
Acetone	Littre	20	80	1600
Pen	piece	5	30	150
Printer Cartridg	piece	2	600	1200
Total, *rub*				**230750**

In the amount of material costs amounted to 230750 rubles. Prices are average in the city of Tomsk.

4.5.2 The basic salary of the project performers

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}):

$$S_s = S_b + S_{ext} \quad (4.7)$$

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

The basic salary of the head (engineer):

$$S_b = S_d * T_w \quad (4.8)$$

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

S_d - average daily wage of an employee, rub.

$$S_d = \frac{S_m \cdot M}{F_d} \quad (4.9)$$

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:

$$S_m = S_{tr} \cdot (1 + k_{pr} + k_s) \cdot k_d \quad (4.10)$$

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:

$$S_{ext} = k_{ex} \cdot S_b \quad (4.11)$$

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):

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

$$S_d = \frac{S_m \cdot M}{F_d} = \frac{51285 \cdot 10}{365 - 66 - 56} = 2110{,}5 \text{ rub.};$$

$$S_b = S_d * T_w = 2110{,}5 \cdot 27 = 56983.5 \text{ rub.};$$

$$S_{ext} = k_{ex} \cdot S_b = 0{,}135 \cdot 56983.5 = 7692.8 \text{ rub.}$$

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

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

$$S_d = \frac{S_m \cdot M}{F_d} = \frac{33150 \cdot 10}{365 - 117 - 28} = 1506{,}8 \text{ rub.};$$

$$S_b = S_d * T_w = 1506{,}8 \cdot 76 = 114517 \text{ rub.};$$

$$S_{ext} = k_{ex} \cdot S_b = 0{,}135 \cdot 114517 = 15460 \text{ rub.}$$

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_{ex}	S_{ext}, rub.	Total, rub
Scientific supervisor	26300	0.3	0.2	1.3	51285	2110.5	27	56984	0.135	7693	64677
Engineer	17000				33150	1506.8	76	114517		15460	129977

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:

$$S_{eb} = k_{eb} \cdot (S_b + S_{ext}) \qquad (4.12)$$

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	56984	7693
Engineer	114517	15460
Coefficient of deductions for payment to extra-budgetary fund	0.3	
Total		
Scientific supervisor	19403	
Engineer	38993	

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:

$$S_{over} = (\text{sum of articles } 1 \div 4) \cdot k_{over} \qquad (4.13)$$

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

$$S_{over} = (C_m + S_b + S_{ext} + S_{eb}) \cdot 0{,}16;$$
$$S_{over} = (230750 + 171501 + 23153 + 58396) \cdot 0{,}16 = 77408\, rub.$$

4.5.6 Formation of costs for the project

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

Table 4.9 – Budget of the project

Article name	Sum, *rub*	Total in %
1. Material costs of project	230750	41.2
2. Costs for basic salaries	171501	30.5
3. Costs for additional salaries	23153	4.1
4. Extrabudgetary funds deductions	58396	10.4
5. Overhead charges	77408	13.8
Budget of the project	561208	100

The budget of all project costs is 561208 rubles. The largest percentage of the budget is the material costs for project (41.2 %).

List of publications

1. S.A. Linnik,* , A.V. Gaydaychuk , V.V. Okhotnikov , R. Khalafov , S.E. Kunashenko High-speed deposition of uniform diamond coatings on WC-Co milling cutters in AC glow discharge plasma\\ Elsevier journal \\ Surface and Coatings Technology\\ on processing in Elsevier Editorial System.

2. Khalafov R. \\ Investigation of thermal treatment influence on the structure and properties of PIM-products \\ Moderntechnologies and materials of new generations: a collection of proceedings of the International Conference with elements of a scientific school for youth, Tomsk, October 9- 13, 2017-Tomsk, 2017. - 2017. - P. 251-252.