PULSED CATHODIC ARC PLASMA TECHNOLOGY DEPOSITION OF THROMBORESISTANT NANOSTRUCTURAL CARBON COATING ON COMPONENTS OF ARTIFICIAL HEART VALVES

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Abstract

This paper describes the results of applying of pulsed cathodic-arc plasma (PCAP) method formation in vacuum biocompatible and thromboresistant t-a carbon coatings on the components of artificial heart valves (AHV).

The special pulsed arc plasma source was used for evaporation of the graphite cathode to form pulsed accelerated carbon plasma flows and deposited them on polished samples of titanium alloys and components AHV made of the same alloys. Influence of the initial voltage of cathode arc discharge in range of 100÷450V and temperature of substrate during the deposition in the range 293÷773ºK on structure, phase composition, hardness and biomedical properties coatings were investigated .

The results of investigation of biomedical properties t-a carbon films deposited by PCAP method have shown that the best biocompatibility and thromboresistivity have the nanostructural carbon coatings with cluster size 10-20nm and thickness 0,08÷0,1 microns. The artificial heart valves with deposited nanostructural t-a carbon coatings were implanted on ten animals (dogs). The obtained results have shown that the passing of blood through AHV leads to moderate activation of coagulation processes.

[Engineering of Biomaterials, 56-57,(2006),12-16]

Introduction

Nowadays biological and mechanical artificial heart valves (AHV) are successfully used to treat heart valve function disorders. Comparison of there two kinds of AHV has discovered, that biological prosthesis causes less thrombogenity but it has shorter mechanical durability and life period. It wears out rather quickly, valve insufficiency is developed, the valve doesn˘t close property. Mechanical AHV has longer mechanical durability and is resistant to wear [1]. Its life period is more that 30 years. However it injures blood elements shapes, causes blood coagulation process activation, that results in developing of thromboembolitic complications.

The problem of creating biomaterials for artificial human organs, especially artificial heart valves, arises from a number of specific requirements connected to their influence on living body. A material should not be toxic, allergic, traumatizing living tissue, it should be resistant to wear and mechanical destruction and should not cause hemoles and blood coagulation, change its structure and surface configuration, transform chemically or decompose [2, 3]. Such metals and alloys as stainless steel, Ti, Ta, Co-Cr and others have received the greatest spreading as biomaterials for medical purposes. But side by side with such merits of these materials as high strength, long life, good technology of treatment, they have great shortcomings, such as biological incompatibility, not sufficient resistance to the influence of biological environment, and excite allergy. This problem can be solved by protection of an implant surface with special coatings [4, 5].

Methods of preparation and examination DLC coatings

The special pulsed are plasma source was used for evaporation of the graphite cathode to form accelerated pulses carbon plasma flows and to deposit them on polished titanium alloys samples and AHV components. The deposition conditions were as follows: initial voltage of arc discharge 100÷350V, arc current amplitude 2000÷3500A, pulse energy 10÷150J, pulse frequency 5÷8Hz, pressure of the vacuum chamber~10^{-3}Pa, substrate temperature 293÷673ºK.

Transmission electron microscope (TEM) JEM 200CX was used to investigate structure of the DLC films.

Raman and X-ray photoelectron spectroscopies were employed to characterize the chemical atomic bond and composition of the DLC coating. The photoelectron spectrometer EC-2401 with Mg-Ka radiation (hω=1253.6 eV) was used.

Nanohardness was measured by Scanning Probe Microscope and nanotribology was determined by Digital Scan-3100 (both made of Digital Instruments).

Medical tests were carried out with blood of ten dogs which had implanted AHV with DLC coating.

Results and discussion

Contact of an alien material with blood leads to formation of a blood plasma albumen layer on the material surface, the dynamic of composition and structure alteration of which to a great extend determines physicochemical and biocompatible properties of an implant surface. As it is shown by results of scientific research and practical surgery with organs implantation the best biocompatible and medico-biological properties have the surfaces with minimal value of interface free energy of implant's surface and biological habitat which are composed of interchanging hydrophile and hydrophobe domains with size less than 10-50nm (i.e. nanostructural surfaces). If such conditions are observed the surface absorbs a minimal albumen quantity easily interchangeable with blood plasma albumen what results in the rise of bio- and hemocompatibility of the implant. The DLC coatings are the more preferable [6-11]. Carbon coatings don't have general toxic, allergenic and carcinogenic influence and they aren't histotoxic. These coatings are thromboresistive, biocompatible with blood sells don't make influence on blood plasma albumen and don't change activity of plasma enzymes.

We produces biocompatible thromboresistant DLC coatings manufactured from pulsed flows of carbon plasma by the method of pulsed cathodic-arc deposition. The results of DLC coatings deposition in vacuum on the AHV components from titanium alloy VT16 are presented. The influence of the initial voltage of cathode arc discharge in the range of 100-450V and the temperature of AHV components during the deposition in the ranges of 293 -773ºK on structure, phase composition, adhesion, tribology and hardness of DLC coatings were investigated. The deposited

particles in our process are usually high energetic ions (carbon is once ionized at 98%). High ionization takes place due to a very high power density presenting at the surface of the feedstock material, i.e. carbon in the case of diamond-like carbon. Deposition by high energetic ions has two advantages. The first advantage is that the ions react more likely with the substrate than with neutral atoms and molecules and the second advantage is that usually at impact of high energy ions, which embed partially into substrate surface. As a result dense, inorganic coatings with an excellent adhesion can be deposited virtually on any material. All our pulsed plasma processes were executed in a vacuum chamber with a base pressure of about 10-3Pa. The pulsed plasma discharge was formed by a short-time electrical arc erosion of cathode material by extremely hot microspots of a vacuum arc discharge. In addition the plasma can be accelerated with a Hall-type plasma accelerator and directed towards the substrate where a coating is deposited.

The generation of the pulsed plasmoids is obtained by short-term electrical erosion of cathodic material by hot cathodic microspots of pulsed vacuum arc, burning on the integral "cool" surface. The plasma flows or plasmoids, periodically generated by the Hall's arc accelerator, were accelerated and directed onto the substrate, fixed in the substrate holder. As a result of condensation of the plasmoid material thin films and coatings are formed on the substrate surfaces.

A new Hall's pulsed accelerator of electroerosive plasma for technological purpose has been designed and developed to realise the pulsed method [12]. The plasma source gives a possibility to obtain the thin films and coatings on the base of metals, their allows and combinations of nitrides, carbides etc, and also of diamond-like carbon.

The scheme of without contact ignition plasma source is presented in the FIG.1. The plasma source has ignition system which to use a localised electrical contact between cathode 1 and the thin film conductor 2, deposited on the surface of an insulator 6. In our plasma source the above localised contact moves from pulse to pulse by the cathode 1. A such technical design provide a high discharge ignition probability and practically avoid the erosion of an insulator, providing a long life of the ignition system.

The plasma source consists from three main units: a cathode unit, an anode unit and a discharge ignition unit. The cathode unit has the main plasmaforming expendable electrode - the cylindrical cathode 1 which is fixed on the end of cooling cathode holder 3. There is a bellows which allow to move the cathode in the plasma generation zone
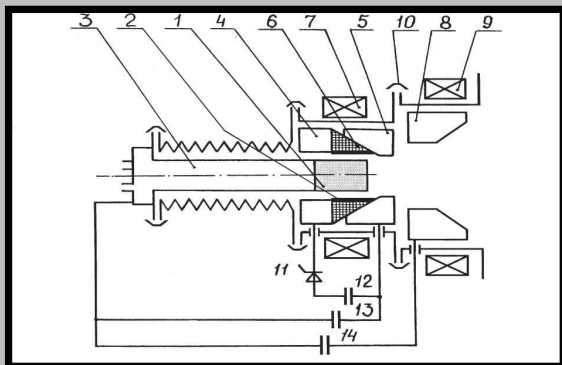
after its partial evaporation. This gives the constant diagram of distribution of accelerated plasma flows. In the plasma source the discharge ignition is executed by a discharge between the ignition electrodes 4 and 5, having the contact with thin film conductor 2 deposited on a dielectric 6. The solenoid 7 is used to increase the discharge stability ignition. The plasma source anode unit is composed by an anode 8 and a focusing solenoid 9. The assembly of generator units is effected by the clamps 10. A control semiconductor valve 11 is used for discharges commutation. The ignition capacitor 12 of 10-20µF is discharged by this valve. The pulse arc discharge is made by energy of capacitors battery 14 having a capacity of 2-4 thousand of microfarads.

The generator works in the next consecution. After obtaining in the vacuum chamber on which is fixed the generator the pressure less than 10-3 Pa, the capacitor bank 12 is charged up to a voltage of 500 - 800V, and the capacitors batteries 13 and 14 is charged up to 150-400V. The control signal going on the valve 11 perform the discharge of the capacitor bank 12. The appearing current pulse evaporates the film conductor 2 and creates the appearance of initiating plasma in the area of the localised electrical contact on the electrode 5 and cathode 1. The plasmoid creates a conductivity channel between the cathode 1 and the ignition electrode 5. This is excite the discharge of the capacitor13 and forming of the cathode spots. The material of evaporated in the cathode spot is almost fully ionised. The plasma jets expend in all directions and some fraction of the plasma makes contact with anode.

The further development of the initiating discharge bring to filling of the space between the cathode 1 and the anode 8 by the plasma what due to the beginning of a main discharge by the energy accumulated in the capacitors battery 14. The solenoid 9 create a magnetic field to focus and accelerate plasma. It is generate plasma flows of cathode material. The necessary coating is formed from deposited plasma flows on the surface of manufacturing articles.

The FIG.2 presents the oscillograms of alteration of discharge current between the plasma source electrodes in the process of one pulse. The curve 1 shows the discharge current alteration between the ignition electrodes (unit scale 100 A), the curve 2 shows the main discharge current alteration between the cathode and the anode (unit scale 1000A). The time of the ignition pulse is 25 µs, the time of
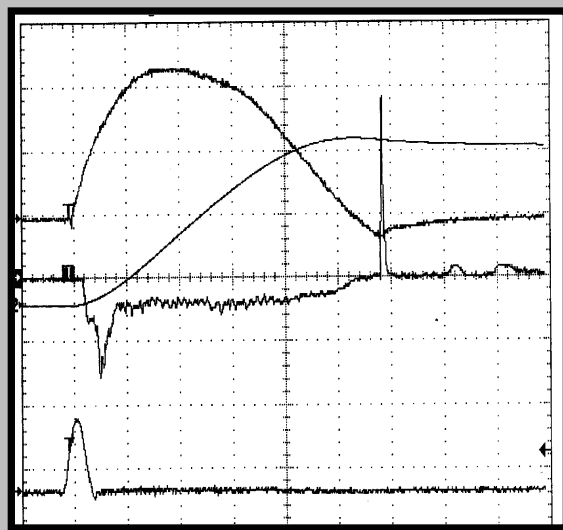

FIG. 1. Schematic illustration of the Hall's plasma accelerator used for pulsed arc deposition DLC coatings.

FIG. 2. Oscillogram of the parameters disharge

the main erosion pulse is 200 µs. The integral current between the cathode and the anode is determined by the curve 3.

The current of plasma going through Longmur probe sensor (the curve 4) is measured during the discharge. The contribution of ion current in the plasma flow is approximately 10% of the arc current. Electrons of plasma reach the probe in 8 µs after main pulse beginning and ions of plasma reach the probe only after 240µs. The electrical current is provided by a flow of electrons from the cathode to the anode which is significantly faster than the velocity of the ions. The electron velocity was 1.5×10^7 cm/sec and the ion velocity was 0.5×10^6 cm/sec, difference 30 times.

the deposited DLC films have the same chemical composition as the graphite cathode. The DLC films have carbon 98% and oxygen 2%, but the graphite cathode has a little more oxygen.

The films have a low content of impurities. The total amount of impurities is ~3% on layers surface of a thickness of 5nm, and in deeper layers it is less than 2%. The main impurity was oxygen and while secondary impurity ion mass spectroscopy analysis showed the presence of traces of Cr,Fe,Ni, which probably concerns the sputtering of vacuum chamber parts.

The results of electron microscopy and diffraction analysis indicate that the quasiamorphous disordered phase con-

Properties	Diamond films CVD	Hydrogenized films α-C:H	Arc plasma deposition films tα-C	Diamond (Type II)
Microhardness (GPa)	70-90	18-40	40-100	57-100
Young's modulus (GPa)	986-1050	50-150	450-600	1079
Density (g/cm^3)	3.5	1.8-2.4	2.4-3.4	3.52
Friction coefficient, up to	0.05	-	0.05	0.05
Resistivity (Ohm·cm)	up to 10^{12}	$10\text{-}10^{14}$	$10^4\text{-}10^9$	10^{14}
Thermal conductivity (W/m·°K)	600-1800	0.2-0.5	3-9	2000
Refraction index (λ=583nm)	2.4-2.45	1.7-2.3	2.4-2.7	2.44

TABLE 1. Properties of diamond and DLC films.

Our experiments have shown that the deposited DLC films have a good adhesion with different materials of substrate. The DLC films exhibited a density mass up to 3.4 g/cm^3, electrical specific resistance at different conditions is from 10^4 up to 10^9 Ohm×cm, the temperature of structural transformation to graphite was higher than 673°K [13]. TABLE 1 shows the results of comparison of some properties of DLC films with natural diamond and carbon films prepared by using other methods.

For explain the film properties we have conducted complex studies of their structure, chemical and phase composition by electron microscopy, Raman and photoelectron spectroscopies, nanohardness and nanotribology [13].

DLC films deposited on surface AHV components have thickness 0.5÷0.2µm. therefore for measurement of hardness it's necessary to use nanohardness. The result of measurements were shown that DLC films hardness depends from condition of deposition and achieves hardness of natural diamond up to 100GPa.

Chemical composition investigation performed by means of X-ray photoelectron spectroscopy method has shown that

tains crystalline inclusions of different carbon modifications, with size 5÷15 nm (FIG. 3). At the same time one can see larger crystalline inclusions with diamond structure. Crystalline inclusions have a comparatively small volume in films, therefore the quasiamorphous phase has the main influence on DLC-films properties.

The electron diffraction patterns of the quasiamorphous phase in carbon films exhibits from two to six diffusive rings which may differ by their position, intensity and shape in various samples and can be described by that of small cluster models. It was found that DLC films deposited from pulsed vacuum plasma flows represent a nanostructural condensate with ordered nanoclasters of a size of about 0.5-2.5 nm with coherently dispersed electrons (FIG. 4) and have a diamond structure which organize clusters more larder sizes 5÷15 nm. Between these nanoregions there are carbon atoms located in disorder.

Different DLC films properties are due to different correlation of sp, sp^2 and sp^3 atomic bonds [14-16]. It was shown that possible changing the deposition conditions to obtain
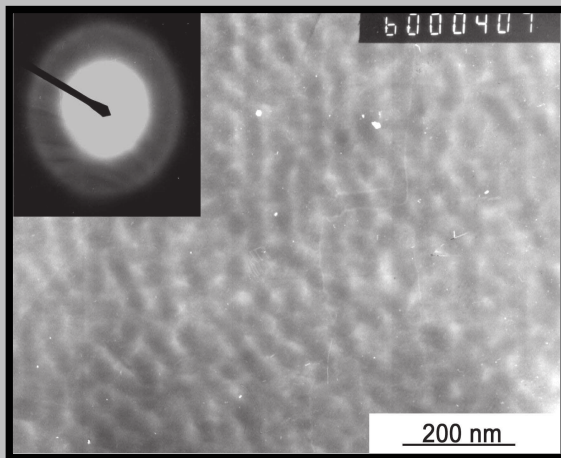

FIG. 3. TEM image and a diffraction pattern of nanostructure of the DLC film.
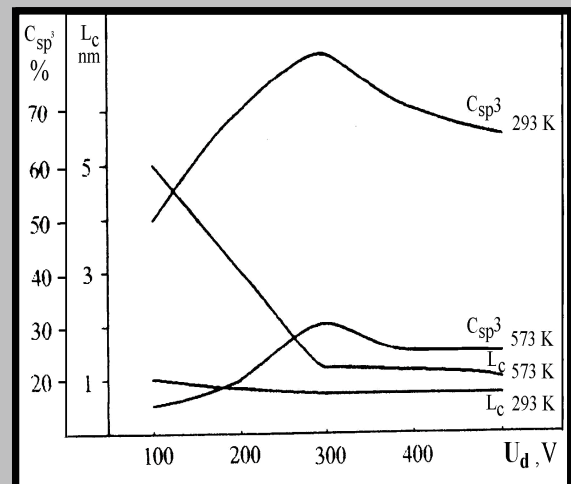

FIG. 4. The dependence of Csp^3 atomic bonds and Lc area size of coherent electron scattering on main discharge voltage Ud for substrate temperature of 293°K and 573°K.

Indices	Control group n=10	After an AHV implantation						
		1-3 days		P	1 month		P₁ <	P₂<
		1 group (n=14)	2 group (n=10)		1 group (n=9)	2 group (n=7)		
Time of coagulation in non siliconized test-tube (sec)	491.0±12.l	347.2±13.8	660.0±II.6	0.001	366.0±23.2	432.0±6.7	0.05	0.001
Time of coagulation in siliconized test-tube (sec)	688.5±12.5	412.2±80.8	780.0±8.9	0.05	532.0±66.2	794.0±18.5	0.05	0.5
Autocoagulogramme 8 min	13.8±0.3	15.2±0.9	13.0±0.6	0.5	12.0±0	20.6+0.6	0.001	0.001
10 min	16.7±0.3	15.6±1.2	14.0±l.2	0.05	12.3±0.8	24.6±0.8	0.001	0.001
PTI %	86.3±3.2	94.4±3.7	96.5+14.1	0.5	89.3±4.9	78.2±4.6	0.001	0.06
Fibrinogen A (mg)	96.6±4.7	296.0±35.9	126.6±l.0	0.05	371.6±35.6	123.0±4.7	0.05	0.001
Tolerance of plasma to heparine (sec)	231.0±3.2	112.0±10.8	188.0±22.0	0.1	100.0±56.3	147.4±6.2	0.008	0.06
Thrombus time (sec)	28.3±1.9	27.4±0.9	29.0±l.2	0.5	21.8±1.4	25.0±1.7	0.05	0.005
Fibronolisis euglobuline (sec)	3324±33	3442±48	3090±17	0.001	3412±41	2620±101	0.001	0.003
Hematocryte	45.0±0.9	47.8±5.2	41.0±0.6	0.05	48.3±4.2	40.4±l.0	0.05	0.5
where: P – truth of data comparison after 1-3 days and data of control group P₁ - truth of data comparison after 1 month and data of control group P₂- truth of data comparison after 1 month and 1-3 days after operation								

TABLE 2. Coagulation data of the peripheral blood of dogs after implantation AHV with the DLC coating.

DLC films with the predominance of tetrahedral (sp³-diamond) types of bonds between carbon atoms. Determination of bonds between carbon atoms C-C sp³ and C-C sp² is realized using X-ray photoelectron and Raman spectroscopy. For example FIG. 4 shows the correlation of the number of sp³ bonds on main discharge voltages for different substrate temperatures. One can see that this dependence has a non-linear behavior. Diamond type sp³ bonds are most prevalent with main discharge voltage of 300V. It was ascertained that the properties of both DLC-films prepared by pulsed vacuum arc plasma deposition and all DLC film-titanium substrate samples have high chemically inert and biocompatible.

On the base of investigations of DLC coatings property dependence on deposition conditions there were chosen optimal parameters of cathodic-arc evaporation of graphite target with formation of carbon plasma flows and their condensation on surface of substrates from titanium alloys VT-16 and on components of AHV made of VT-16 alloy. The investigation results have shown that the deposition of DLC coatings increase considerably wear resistance and chemical fastness of researching alloys. DLC coatings of 0.08-0.1µm thickness were deposited on AHV components at different deposition modes and than was executed a medico-biological testing of AHV to determine their thrombogenity and biocompatible.

Comparative experiments with dogs were executed to discover the effectiveness of DLC-coating details AHV [17]. The AHV "PLANEX" was implanted to 14 dogs (1 group) and AHV "PLANEX" with DLC coatings was implanted to 10 dogs (2 group). For both groups autocoagulography coagulation period was defined in 8 and 10 minutes to determine the first hemostasia phase. The protrombin index, fibrinogen A level, the prothtrombin period of spontaneous and euglobulin fibripoliosis were defined to determine the second hemostasia phase.

The data of venous and aortic coagulography peripheric blood as well upper and bellow the AHV implantation were analyzed to expose the AHV influence on hemostasia system. TABLE 2 demonstrates the results of the investigations.

A month later after the implantation a well-noticeable hypercoagulation and increased level of fibrinogen A were observed in the peripheric blood of the dogs (group 1). Coagulation indexes approached the normal in the dogs (group 2) which were implanted AHV with DLC coatings. The coagulation period was increased, PTI index came back to initial state, the number of fibrinogen A and hematocritis figures were decreased. Thus brightly expressed thrombogenios influence of AHV "PLANEX" without DLC coatings on the blood coagulation system on both local and systematic levels was preserved for a month.

Conclusion

According to results of thrombogenity investigations there were optimized the conditions of DLC- coatings deposition on AHV components and there were prepared AHV which were implanted to animals. For determine the thrombogenity of AHV the blood for analysis was taken before and after implanted AHV. Coagulogramme was studied during one month. The obtained results have shown that the passing of blood through AHV leads to moderate activation of coagulation processes. The AHV with DLC coatings which prepared by pulsed cathodic arc plasma deposition have vastly less thrombogenity than the AHV without DLC coatings.

AHV with DLC coatings influenced much less expressively hypercoagulating on the central and peripherical blood stream starting with the first month in the body. Taking into consideration, all the above mentioned should be recommended preferably to apply in clinical practice.

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THE CHLORINATED SYNTHETIC DIAMOND SURFACE INTERACTION WITH C-NUCLEOPHILES

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Abstract

Several reactions of C-nucleophiles with the chlorinated surface of two types of diamond were implemented. Detonating nanodiamond "UDA-SF" and synthetic diamond "DALAN" have been employed in the above procedures. The incorporation of butyl and nitrile groups has been achieved. However the incorporating of phenyl groups via reaction with PhLi is still a problem. For the first time NMR-H1 spectroscopy of suspension was used for elucidating structure of grafting compound was proposed.

Keywords: nanodiamonds; grafting compounds; surface chemistry; chemical functionalization.

[Engineering of Biomaterials, 56-57,(2006),16-19]

Introduction

There is an escalating interest in diamond chemistry to nanoscale diamonds in recent years [1]. It is due to several reasons. The first is that nanodiamonds (ND) became commercially available only in last ten years [2]. The second and obviously the most significant ones are their unique properties which combine mechanical, termic, radio and chemical stability of diamond as well as lability of functional cover of surface. The latter is determined by large value of specific surface area in ND. In various patterns of NDs specific surface area is about 300 m^2/g or less whereas in the case of natural or synthetic diamonds powders the value of specific surface area usually is about several metres per gram. Such large value of specific surface area of NDs substantially increases possibilities of their further chemical modification and moreover enlarges the field of potential application of diamond materials. Among the most attractive utilizations of NDs are biomedical applications (nanoparticles for drug carry in vivo) and development of new chromatographic materials, especially for HPLC.

Also stable hydro- and organosoles of NDs are required for many technological applications. Therefore the problem of chemical modification of ND particle surface is of current importance. Besides modification of ND surface is attractive as a technique for unification of surface chemical state.

Notwithstanding the fact that NDs is a promising material the number of works concerning chemical modification of its surface is rather modest [3,4,5]. Therefore investigations on controlled chemical creation of grafting compounds on the ND's surface are of fundamental and technological importance.

Experimental

In the present work two types of diamond were employed: detonation nanodiamond "UDA-SF" and synthetic diamond powder "DALAN". "UDA-SF" is typical nanodiamond with the size of aggre-gates no more then 0.04 μm and specific surface area 245 m^2/g (BET measurement). Synthetic diamond powder "Dalan" used represents itself a polydisperse powder with the size of particles from 5 nm up to 10 microns with a specific surface area of 22 m^2/g (BET measurement).

Chemical state of diamond particles was characterized using Fourier-transform spectroscopy (IR200 Thermonicolet), NMR-H1 - spectroscopy (Bruker-Avance-400).

Results and discussions

It is necessary to mention that in native state the surface of diamond powder is covered by different oxygen containing groups (for ex. alcohol, carboxyl, carbonyl and others that is firmly confirmed by infrared spectra [6,7]), i.e. the surface is polyfunctional. And this fact complicates any further chemical reactions on the diamond surface due to nonselectivity of their passing. Consequently the polyfunctionality arises the problem of unification of diamond surface in order to make reactions that are carried out on it to be selective.

The most common procedure of unification, and moreover the only one, consists in hydrogen stream treatment of diamond at 1073 or 1173 K for 4 or 5 hours. These conditions are sufficient to modify the diamond surface in a way of hydride group's layer formation (Scheme, FIG.1).

It should be noted that specific surface area of diamond powder is slightly affected by this treatment as determined by BET analysis. So, after such treatment we obtained a fully hydrogenated diamond surface that is confirmed by IR-spectra presented (FIG.2).

As we can see from spectra, after such treatment stretch-