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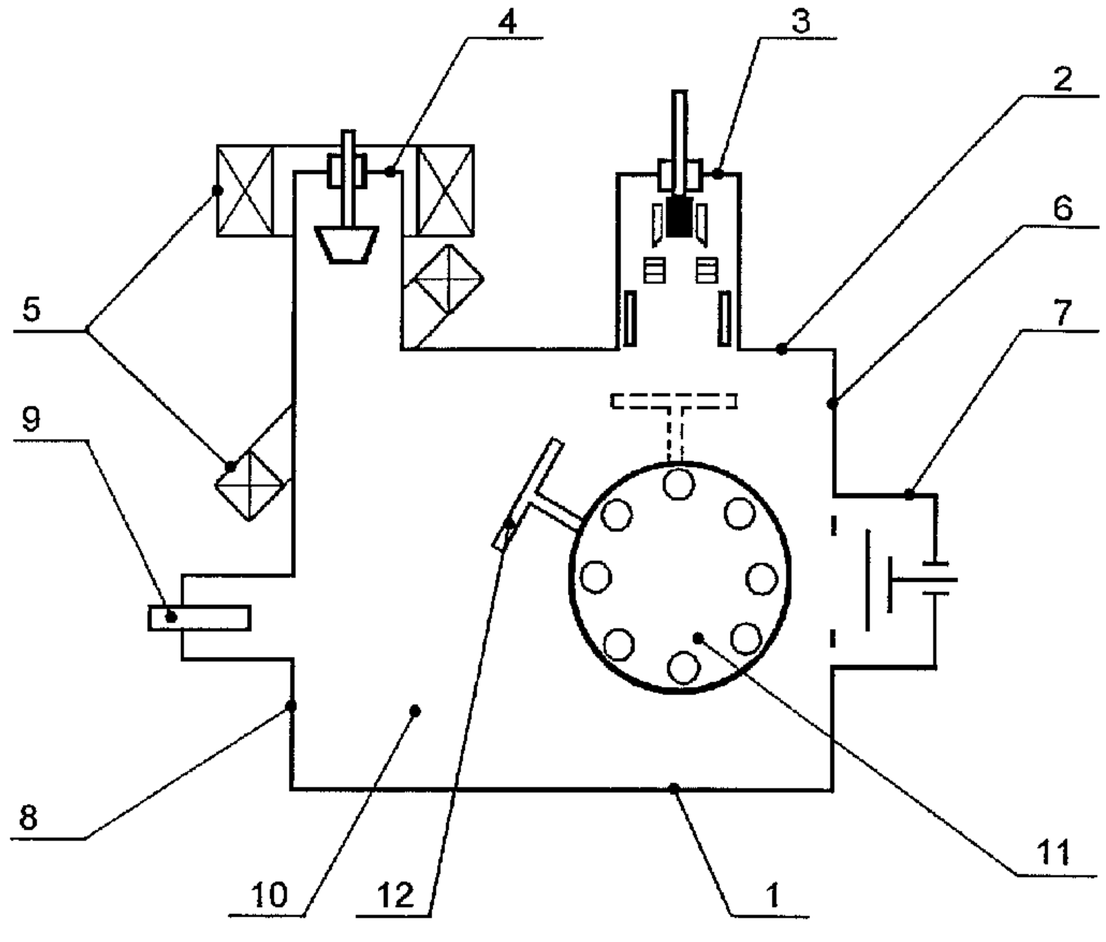
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(54) Titre : REVETEMENT GENRE DIAMANT, METHODE DE PLACAGE ET MEULETTE DE TOUR DENTAIRE POSSEDANT CE REVETEMENT

(54) Title: DIAMOND-LIKE COATING, METHOD OF ITS PLATING AND DENTAL BUR WITH THE SAID DIAMOND-LIKE COATING



(57) Abrégé/Abstract:
Objects of the invention: diamond-like coating, method for its plating on the product surface and dental bur with this coating. The field of the invention: producing wear-resistant diamond-like coatings on metal and non-metal products, in particular, on medical tools. The essence of the invention: a layer of diamond-like substance plated on the substrate by way of carbon deposition in the gas phase under the conditions of reduced pressure is an aggregate of successive sub-layers of diamond-like coating with reduced concentration of contaminants in the sub-layers interfaces areas, the layer is plated in a sealed chamber under pressures lower than atmosphere pressure controlling the product temperature during the deposition process by the method comprising plating a metal sub-layer from a separate stationary source of metal plasma and further plating diamond-like coating on products being under floating voltage, wherein before turning on each of the metal or carbon plasma sources the plasma flow is completely blocked with a shutter, then the corresponding source is turned on, and in the predetermined period of time the shutter is made open. Such a coating is plated on a dental bur. The technical result: increase of the coating thickness and increase in its strength and wear resistance.

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ABSTRACT OF THE DISCLOSURE

Objects of the invention: diamond-like coating, method for its plating on the product surface and dental bur with this coating. The field of the invention: producing wear-resistant diamond-like coatings on metal and non-metal products, in particular, on medical tools. The essence of the invention: a layer of diamond-like substance plated on the substrate by way of carbon deposition in the gas phase under the conditions of reduced pressure is an aggregate of successive sub-layers of diamond-like coating with reduced concentration of contaminants in the sub-layers interfaces areas, the layer is plated in a sealed chamber under pressures lower than atmosphere pressure controlling the product temperature during the deposition process by the method comprising plating a metal sub-layer from a separate stationary source of metal plasma and further plating diamond-like coating on products being under floating voltage, wherein before turning on each of the metal or carbon plasma sources the plasma flow is completely blocked with a shutter, then the corresponding source is turned on, and in the predetermined period of time the shutter is made open. Such a coating is plated on a dental bur. The technical result: increase of the coating thickness and increase in its strength and wear resistance.

DIAMOND-LIKE COATING, METHOD OF ITS PLATING AND DENTAL BUR WITH THE SAID DIAMOND-LIKE COATING

The invention is related to the field of producing highly rigid wear-resistant coatings on metal and non-metal goods, in particular on medical cutting tools, for example, on dental burs.

Background of the invention

Diamond-like coatings (DLC) are widely used in various branches of science and technology. Main advantages of DLCs are their hardness, wear-resistance, purity, biological inertness, etc. Due to such properties DLCs are plated on medical tools, medical implants, cutting tools and are used in other branches.

There is known a DLC plated by vacuum arc method disclosed in the USSR Certificate of Authorship No. 1070949 comprising a DLC layer on a substrate, DLC being plated by a pulse stream of compensated no-current carbon plasma with the density of 10^{18}-10^{19} $cm^{-2}c^{-1}$.

However, the disclosed coating does not have a necessary thickness due to poor DLC adhesion to the substrate.

The closest prior art for the coating according to the invention claimed is a coating disclosed in the USSR Certificate of Authorship No. 1494554 published on March 15, 1994, Bulletin No. 5. The coating has an increased strength of the sub-layer adhesion to the substrate and to the following layer of DLC. Yet strength provided by this method is not sufficient for producing a DLC with high wear resistance due to high concentration of contaminants in DLC.

There is known a method for producing highly rigid DLCs on metal and dielectric substrates by way of cathode spraying of graphite in a magnetic field at low pressure of the inert gas – krypton (10^{-5}-10^{-2} Pa) on the substrate being cooled and having the temperature lower than 100 K (the USSR Certificate of Authorship No. 411037 published in 1974, Bulletin No. 29). A drawback of

this method is poor quality of the coating, low productiveness and high cost of the products.

Main efforts of the researchers and manufacturers of DLCs are aimed at increasing their wear resistance defined both by adhesion to the substrate and the structure of the coating itself. One of the most important parameters of the DLC deposition process is the substrate (product) temperature that is not to exceed the critical temperature 135-150° C during the process, since in case it is higher coatings start losing their physical and mechanical properties and adhesion to the substrate due to thermal graphitization. Furthermore, necessary is high purity of the source material, high density of the carbon flow, high purity of the substrate surface, etc. As a result, a pulse ion-plasma vacuum arc (further – vacuum arc) method of plating DLC was developed, the essence of which lies in forming an intensive pulse flow of carbon ions in an electric arc in a vacuum.

One of the known vacuum arc methods of DLC plating is a method disclosed in the USSR Certificate of Authorship No. 1070949 that involves DLC condensation on the substrate produced by a pulse flow of compensated no-current carbon plasma with density of 10^{18}-10^{19} $cm^{-2}c^{-1}$.

However, the said method does not also provide for necessary thickness of the coating due to poor DLC adhesion to the substrate.

The closest prior art for the method according to the claimed invention is a method disclosed also in the USSR Certificate of Authorship No. 1494554 published on March 15, 1994, Bulletin No. 5, the essence of which lies in the fact that before vacuum arc DLC plating on the substrate surface the said surface is coated by a sub-layer of metal, e.g. titanium having increased strength of adhesion to the substrate and the following layer of DLC, the product being under floating voltage during the process of plating the sub-layer of metal and DLC.

However, adhesion strength obtained by this method is still insufficient for producing a DLC with high wear resistance. Besides, it is reasonable to

plate the titanium sub-layer only on metal substrates whereas DLC may be plated on non-metal substrates as well.

One of the applications of strengthening coatings is their plating on medical cutting tools, in particular on dental burs. Known are dental burs made of steel or hard-alloyed materials (for example, see the State Standard of Ukraine – 22090-89). Yet these burs have a short durability due to insufficient hardness and wear resistance of cutting edges.

The closest prior art for dental burs claimed according to this invention is dental burs of Romidan Dental Ltd. presented on the Internet at the address: http:/www.romidan.com/fgburs.htm. The heads of these burs made of stainless steel are coated by electrostatic gluing with a coating of diamond powder made of natural diamonds. Drawbacks of these burs are their high cost and relatively short durability due to the fact that strength of the bond between the powder and metal is determined by the glue adhesion to the powder and metal.

Summary of the invention

As far as the DLC and method of its plating are concerned, this invention is based on the objectives of improving wear resistance of the product due to the increase in the level of the coating adhesion to the substrate and increasing the coating thickness due to the change of its structure.

As far as dental burs are concerned, this invention is based on the objective of increasing their wear resistance by introducing new materials and elements.

The set objective as to the coating is solved as follows: the known coating comprises a diamond-like substance layer plated on the substrate by carbon deposition in the gas phase under reduced pressure conditions, said layer being an aggregate of successive sub-layers of diamond-like coating with the reduced concentration of contaminants in the areas of the sub-layers interface.

The decrease in the concentration of contaminants is obtained as follows: during the process of plating the coating on the product surface by deposition

of a carbon plasma flow from a pulse arc source in a sealed chamber at pressures below atmosphere pressure, before turning on the carbon plasma source the plasma flow is blocked with a shutter, then the source is turned on, and then in the predetermined period of time the shutter is made open. Then a relatively thin DLC sub-layer is plated, the source is turned off, the shutter is closed, and the process is repeated.

The improvement claimed in this invention consists in the creation of the coating structure out of a number of successive sub-layers with a sharp decrease in concentration of contaminants both at the interface of the successive sub-layers and within one sub-layer. This increases adhesion of sub-layers to each other and to the substrate and so allows to increase the DLC quality and thickness that in turn increases the coating strength and wear resistance.

A further improvement of the coating according to the invention is an additional titanium sub-layer plated by titanium deposition in the gas phase before plating the diamond-like coating on the steel substrate. Adhesion of the whole coating to the substrate is increased due to the fact that titanium has increased adhesion to metal, especially to steel, as a metal. A particular effect is obtained when before plating titanium the titanium plasma flow is blocked with a shutter for a certain period of time. This also leads to decrease in amount of contaminants at the interface and to increase in the DLC adhesion to the substrate and wear resistance of the coating.

In specific embodiments of the invention as a result of the research performed the following parameters of the coating were optimised:

thickness of the diamond-like coating sub-layer is not less than 0.1 μm;

amount of the diamond-like coating sub-layers is within the range of 1 – 20.

The objective set as to the method is solved by adding new operations to the known method of DLC plating on the product surface comprising plating a

diamond-like coating on the product surface by deposition of a carbon plasma flow from a pulse arc source, the said process taking place in a sealed chamber under pressures lower than atmosphere pressure, the temperature of the product being controlled during the deposition process, and the product being under floating voltage, said new operations being as follows: before turning on the carbon plasma source this source is completely blocked with a shutter, then the source is turned on, and in the predetermined period of time the shutter is made open.

The blocking of the carbon plasma source with a shutter before turning it on does not allow the carbon plasma flow to be deposited on the products, and it is these initial periods when a great amount of contaminants, which impregnate the cathode during free air entering into the chamber and during the process of pumping out by oil pumps, are penetrating into the plasma when leaving the surface of the carbon plasma source cathode made of graphite and being a porous material with a well-developed surface. Contaminants present in initial layers of coatings reduce significantly the adhesion of one layer to another one and impair the coating quality. Therefore, the improvement claimed in this invention reduces sharply the quantity of contaminants getting on the product surface and hereby increases the metal adhesion to the product surface and to the DLC, which in turn increases wear resistance of the DLC.

A further improvement of the method according to the invention is that before plating a diamond-like coating on metal products a sub-layer of metal is plated on the product surface from a separate stationary source of metal plasma, the said source being blocked with a shutter before turning it on, then this source is turned on, and in the predetermined period of time the shutter is made open.

As it is known, a sub-layer of metal, e.g. titanium has an enhanced adhesion to the substrate metal, and on the other hand, a sub-layer of metal plated by the aforesaid method has an increased purity and therefore has an enhanced adhesion to the following sub-layer of DLC.

Besides, similar to the case with the carbon plasma source, the shutter blocking the metal plasma flow being formed after turning on the source does not allow this metal plasma flow to be deposited on the substrate during initial periods of the source operation when the amount of contaminants in the flow is the highest.

Thus, this improvement further increases the DLC adhesion to the substrate.

In a further improvement of the method according to the invention before plating a metal sub-layer on the product surface a separate stationary ion source of argon is turned on, and ion bombardment of products with argon ions is performed during the predetermined period of time. Such a bombardment heats up at least the surface layer of the products and contributes to cleaning the surface from contaminants entering the external space. This further increases the metal adhesion to the product surface.

In a specific embodiment of the bombardment of products with argon ions the energy of argon ions is established within the range of 5 – 6 keV, and a positive potential is applied to the products within the range of 800 – 1,500 V.

In a specific embodiment of the method, which comprises plating a metal sub-layer, after turning on the metal plasma source with the closed shutter the arc current is maintained within the range of 90 – 100 A, and the duration of the predetermined period of time, in which the shutter of the metal plasma source is made open, is established within the range of 50 – 60 seconds. These ranges were determined experimentally to be the most appropriate ones for the maximum removal of contaminants from the metal plasma source cathode.

A further improvement of the method according to the invention, which comprises plating a metal sub-layer, is that during the said predetermined period of time a separate stationary ion source of argon is turned on, and ion bombardment of products with argon ions is performed with the energy 5 – 6 keV, while a potential is applied to products within the range of 1,400 – 1,600 V. The effect of the bombardment on the degree of the metal sub-layer

adhesion to the product surface is described above, and performing the bombardment during the metal plasma source preparation for operation reduces the period of plating the coating. The aforesaid ranges of physical values are also determined experimentally and are the most appropriate ones for the said operation.

In a specific embodiment of the method, which comprises plating a metal sub-layer, this sub-layer is plated with the positive potential on the products being within the range of 180 – 200 V. The said range is optimum for plating a metal sub-layer and is also determined experimentally.

A further improvement of the method according to the invention is to repeat the process of the diamond-like coating plating several times with plating a sub-layer of the coating each time, wherein each time before turning on the source of carbon plasma the plasma flow is blocked completed with a shutter, then the source is turned on, and in the predetermined period of time the shutter is made open. This allows to obtain reduced concentration of contaminants in every sub-layer interface area, which decreases stresses in this area and enables to increase the coating strength and wear resistance to further extent.

In a specific variant of any of the aforesaid embodiments of the method according to the invention the duration of the predetermined period of time, in which the shutter is made open, is within the range of 40 – 80 seconds. This experimentally determined period of time is sufficient for contaminants to be removed to the utmost from the surface of the carbon plasma source cathode.

By experimental research of conditions of DLC plating on products without exceeding the critical temperatures specific values of the process parameters were chosen, namely:

- energy of carbon ions not exceeding 0.4 keV;

- duration of the carbon source pulses being 10 μsec with the pulse period-to-pulse duration ratio not less than 30,000;

which may be used in the method according to the invention both separately and in the aggregate.

A further improvement of the method according to the invention is that before opening the pulse carbon source with a shutter it is turned off, and the ion source of argon is turned on for the predetermined period of time. This allows to clean the product surface before plating the next sub-layer on products due to the increase in their temperature and enhance the adhesion of the following DLC layer to the preceding one.

In specific embodiments of the method, wherein a number of sub-layers of the coating are plated, the thickness of each sub-layer of the diamond-like coating is not less than 0.1 μm and/or the amount of sub-layers is within the range of 1 – 20.

A further improvement of the method according to the invention is to introduce an additional new operation of argon ion bombardment of the product surface from a separate ion source during the process of the DLC deposition from a pulse source of carbon plasma.

In the known methods of producing DLC with the help of a pulse source of carbon plasma it was impossible to produce a coating more than 1.5 μm thick due to poor adhesion and large internal stresses in the coating, while many practical applications require coatings up to 5 μm thick and more.

As it is known, the DLCs produced by the known methods have a columnar structure having large internal stress when the coating is sufficiently thick, which results in the cracking of coating at the border of grains and in its detachment from the substrate with the great thickness of the coating.

Furthermore, one of the causes explaining the quality impairment of the DLC plated by the said method on small-sized products with sharp cutting edges and poor thermal conductivity is the local heating of products to temperatures higher than critical temperature, which results in graphitization of metastable diamond structure of the coating and disastrous reduction of wear

resistance and strength of the coating. During the bombardment of the product surface with argon ions obtained from a separate ion source, which takes place simultaneously with the DLC deposition from the pulse carbon source, the argon ions irritate the columnar structure, which contributes to more uniform stress distribution in the coating and reduces the internal stress appearing in the coating during the deposition, whereby graphitization of the structure is avoided. Thus increased quality of the coating is provided, and it is possible to increase its thickness.

By experimental research of conditions of DLC plating on products without exceeding the critical temperatures the energy of the doping argon gas ions was chosen to be within the range of 4 – 6 keV in the specific embodiment of the invention.

The objective set in this invention as to the dental bur improvement is solved as follows: in a dental bur comprising a wear-resistant coating layer the coating is a diamond-like coating comprising a number of successive sub-layers with reduced concentration of contaminants in the sub-layers interface areas.

The technical result obtained thereby lies in the increase in wear resistance of burs due to the plating continuous and homogeneous coating and to the increase in the coating adhesion to the bur material.

The improvements and specific embodiments of the dental bur according to the invention are similar to those described above for the coating.

THE EMBODIMENTS OF THE INVENTION IN WHICH AN EXCLUSIVE PROPERTY OR PRIVILEGE IS CLAIMED ARE DEFINED AS FOLLOWS:

1. A diamond-like coating comprising a layer of diamond-like substance plated on the substrate by way of carbon deposition in the gas phase under the conditions of reduced pressure, characterized in that the layer is an aggregate of successive sub-layers of diamond-like coating with reduced concentration of contaminants in the sub-layers interface areas.

2. The diamond-like coating as set forth in claim 1, characterized in that it further comprises an additional titanium sub-layer plated by way of titanium deposition in the gas phase before the diamond-like coating plating on steel cutting tools.

3. The diamond-like coating as set forth in claim 1 or 2, characterized in that the thickness of each sub-layer of diamond-like coating is not less than 0.1 μm.

4. The diamond-like coating as set forth in claim 1, 2 or 3, characterized in that the amount of sub-layers of diamond-like coating is within the range of 1 – 20.

5. A method for plating the diamond-like coating on the product surface comprising plating a metal sub-layer on the product surface from a separate stationary metal plasma source, after which the product surface is coated with a diamond-like coating by way of deposition of a carbon plasma flow from a pulse arc source, the process taking place in a vacuum chamber with pressures lower than atmosphere pressure, and the product temperature is controlled during the deposition process, the product being under floating voltage, characterized in that before turning on each of the sources of metal or carbon plasma the plasma flow is completely blocked with a

shutter, then the corresponding source is turned on, and in the predetermined period of time the shutter is made open.

6. The method as set forth in claim 5, characterized in that the process of the diamond-like coating plating is repeated several times by plating a sub-layer of the coating each time, blocking the plasma flow each time with a shutter before turning on the carbon flow source, after which the source is turned on, and in the predetermined period of time the shutter is made open.

7. The method as set forth in claim 6, characterized in that the thickness of each sub-layer of diamond-like coating is not less than 0.1 μm.

8. The method as set forth in claim 6, 7, characterized in that the amount of sub-layers of diamond-like coating is within the range of 1 – 20.

9. The method as set forth in any one of claims 5 – 8, characterized in that the duration of the said predetermined period of time is within 40 – 80 seconds.

10. The method as set forth in any one of claims 5 – 9, characterized in that the deposition of the DLC from a pulse source of carbon plasma is performed with the simultaneous bombardment of the product surface with argon ions from a separate stationary ion source.

11. The method as set forth in claim 10, characterized in that the energy of argon ions is within the range of 4 – 6 keV.

12. The method as set forth in any one of claims 5 – 11, characterized in that during the coating deposition the energy of carbon ions does not exceed 0.4 keV.

13. The method as set forth in any one of claims 5 – 12, characterized in that the duration of the carbon source pulses does not exceed 10 μsec with the pulse period-to-pulse duration ratio not less than 30,000.

14. A dental bur comprising a layer of wear resistant coating, characterized in that the coating is a diamond-like coating comprising an aggregate of successive sub-layers with reduced concentration of contaminants in the sub-layers interface area.

15. The dental bur as set forth in claim 14, characterized in that it comprises an additional titanium sub-layer plated by way of titanium deposition in the gas phase before the diamond-like coating plating.

16. The dental bur as set forth in claim 14 or 15, characterized in that the thickness of each sub-layer of the diamond-like coating is not less than 0.1 μm.

17. The dental bur as set forth in claim 14, 15 or 16, characterized in that the amount of sub-layers of the diamond-like coating is within the range of 1 – 20.

18. A method for plating the diamond-like coating on the product surface comprising plating a diamond-like coating on the product surface by way of deposition of a carbon plasma flow from a pulse arc source, the process taking place in a vacuum chamber with pressures lower than atmosphere pressure, and the product temperature is controlled during the deposition process, the product being under floating voltage, characterized in that before turning on the carbon plasma source the plasma flow is completely blocked with a shutter, then the source is turned on, and in the predetermined period of time the shutter is made open.

19. The method as set forth in claim 18, characterized in that before plating a diamond-like coating on metal products a sub-layer of metal is plated on the product surface from a separate stationary source of metal plasma, the said source being blocked with a shutter before turning it on, then this source is turned on, and in the predetermined period of time the shutter is made open.

20. The method as set forth in claim 19, characterized in that before plating a metal sub-layer on the product surface a separate stationary ion source of argon is turned on, and ion bombardment of products with argon ions is performed during the predetermined period of time.

21. The method as set forth in claim 20, characterized in that the energy of argon ions is established within the range of 5 – 6 keV, and a positive potential is applied to the products within the range of 800 – 1,500 V.

22. The method as set forth in claim 19, 20 or 21, characterized in that after turning on the metal plasma source with the closed shutter the arc current is maintained within the range of 90 – 100 A, and the duration of the predetermined period of time, in which the shutter of the metal plasma source is made open, is established within the range of 50 – 60 seconds.

23. The method as set forth in claim 19, 20, or 21, characterized in that during the predetermined period of time a separate stationary ion source of argon is turned on, and ion bombardment of products with argon ions is performed with the energy 5 – 6 keV, while a potential is applied to products within the range of 1,400 – 1,600 V.

24. The method as set forth in any one of claims 19 through 23, characterized in that the sub-layer of metal is plated with the positive potential on the

products being within the range of 180 – 200 V.

25. The method as set forth in any one of claims 18 through 24, characterized in that the process of the diamond-like coating plating is repeated several times by plating a sub-layer of the coating each time, blocking the plasma flow each time with a shutter before turning on the carbon flow source, after which the source is turned on, and in the predetermined period of time the shutter is made open.

26. The method as set forth in any one of claims 18 through 25, characterized in that the duration of the said predetermined period of time, in which the shutter of the carbon plasma source is made open, is within 40 – 80 seconds.

27. The method as set forth in any one of claims 18 through 26, characterized in that during the coating deposition the energy of carbon ions does not exceed 0.4 keV.

28. The method as set forth in any one of claims 18 through 27, characterized in that the duration of the carbon source pulses does not exceed 10 μsec with the pulse period-to-pulse duration ratio not less than 30,000.

29. The method as set forth in any one of claims 18 through 28, characterized in that before opening the pulse carbon source with a shutter it is turned off, and the ion source of argon is turned on for the predetermined period of time.

30. The method as set forth in any one of claims 25 through 29, characterized in that the thickness of each sub-layer of the diamond-like coating does not exceed 0.1 μm.

31. The method as set forth in any one of claims 24 through 30, characterized in that the amount of sub-layers does not exceed 20.

32. The method as set forth in any one of claims 18 through 31, characterized in that the deposition of the DLC from a pulse source of carbon plasma is performed with the simultaneous bombardment of the product surface with argon ions.

33. The method as set forth in claim 32, characterized in that the energy of argon ions is within the range of 4 – 6 keV.

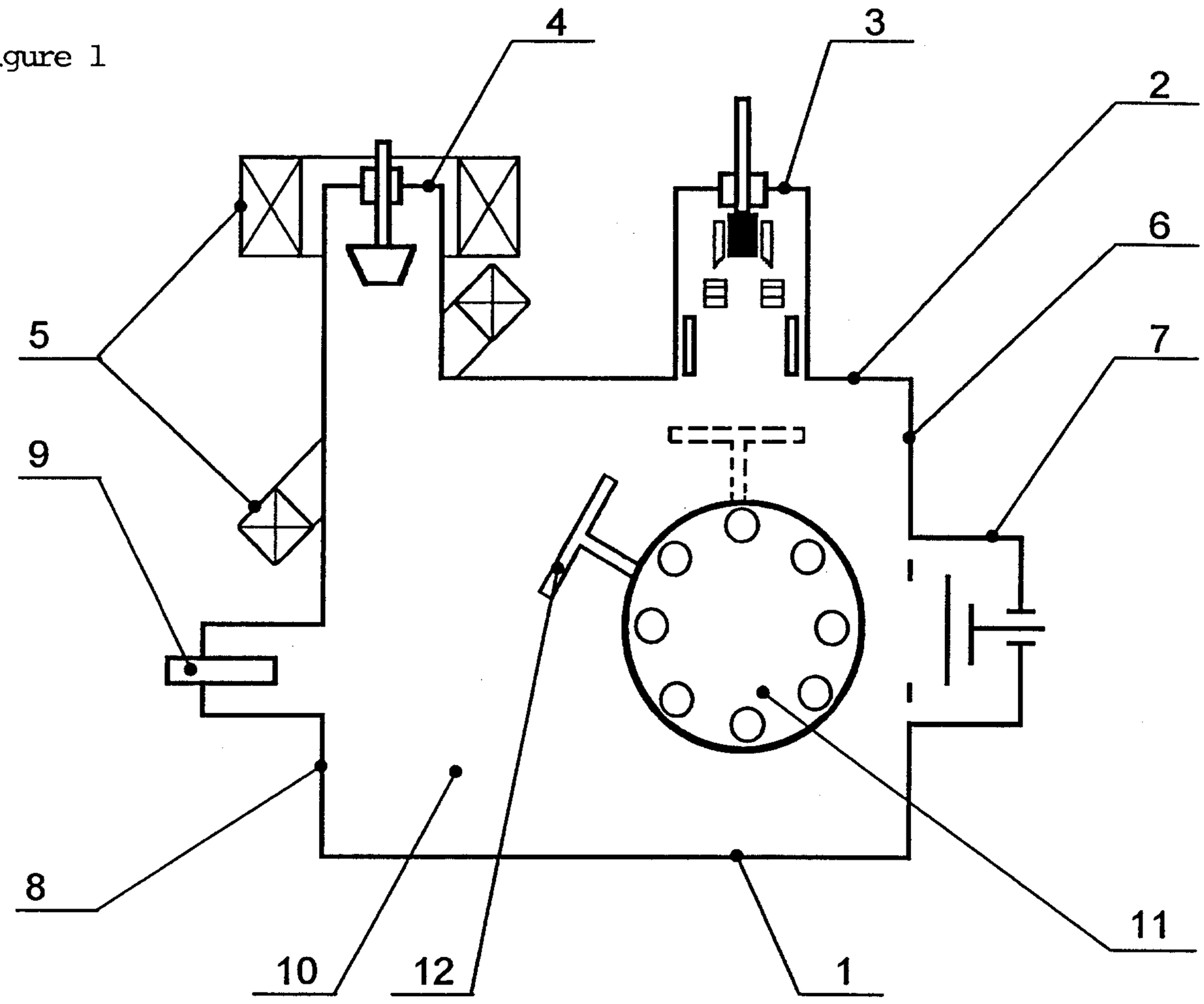
34. A dental bur comprising a layer of wear resistant coating, characterized in that the coating is a diamond-like coating comprising an aggregate of successive sub-layers with reduced concentration of contaminants in the sub-layers interface area.

35. The dental bur as set forth in claim 34, characterized in that it comprises an additional titanium sub-layer plated by way of titanium deposition in the gas phase before the diamond-like coating plating.

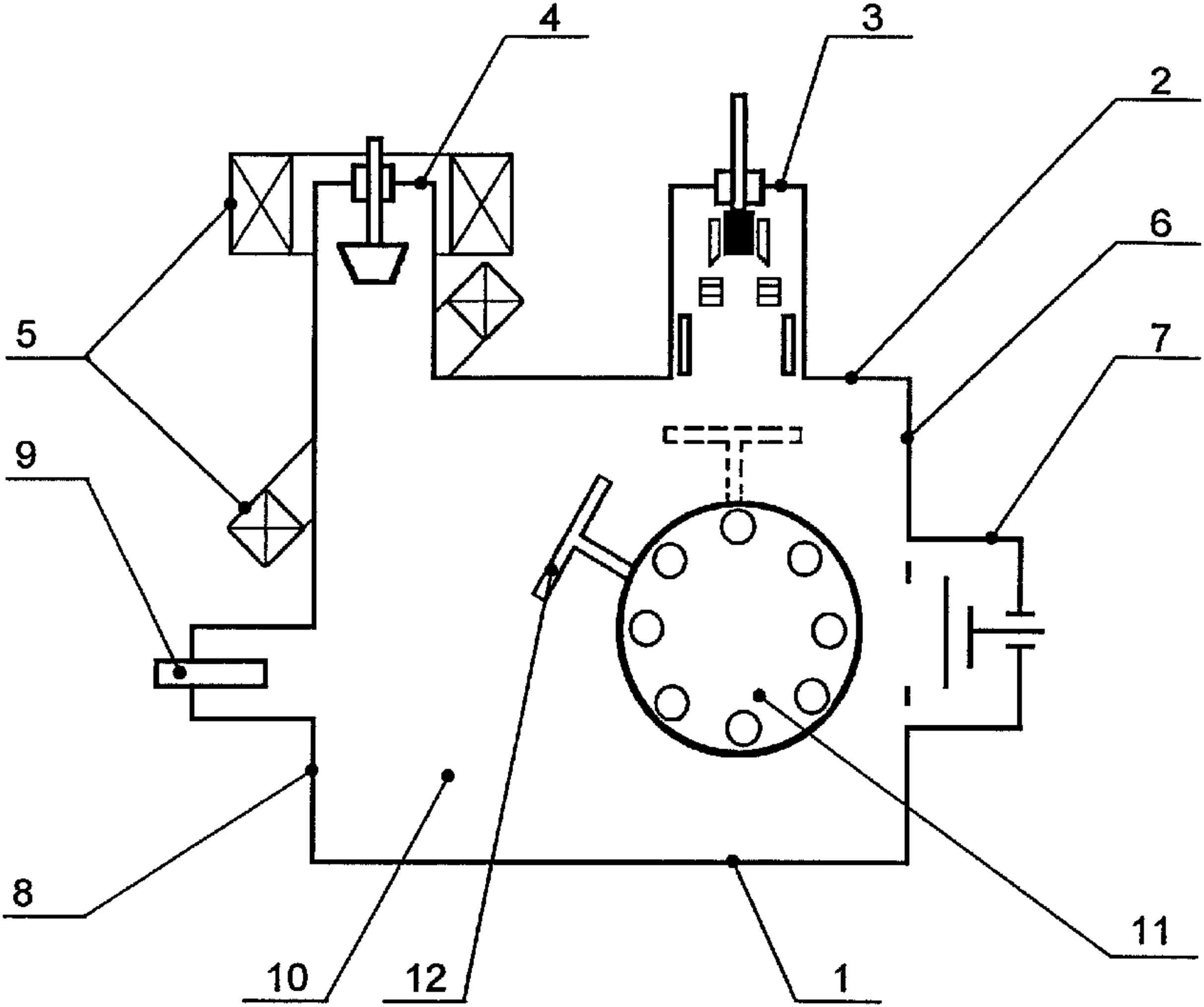
36. The dental bur as set forth in claim 34 or 35, characterized in that the thickness of each sub-layer of the diamond-like coating is not less than 0.1 μm.

37. The dental bur as set forth in claim 34, 35 or 36, characterized in that the amount of sub-layers of the diamond-like coating is within the range of 1 – 20.

Figure 1



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