Aug. 22, 1961 R. H. WENTORF, JR 2,996,763

DIAMOND MATERIAL

Filed Jan. 31, 1956

Fig. 1.

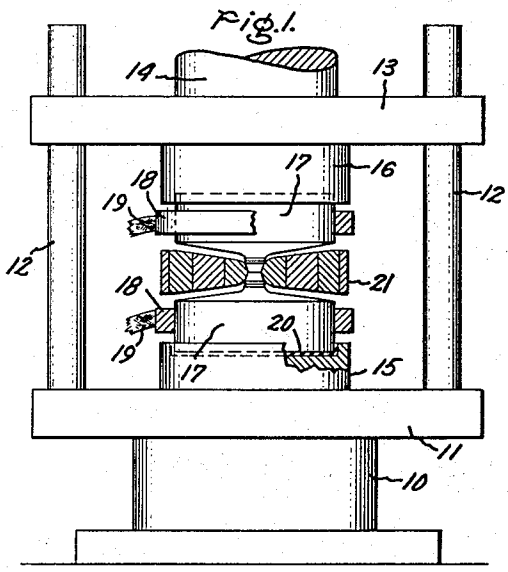


Fig. 3.

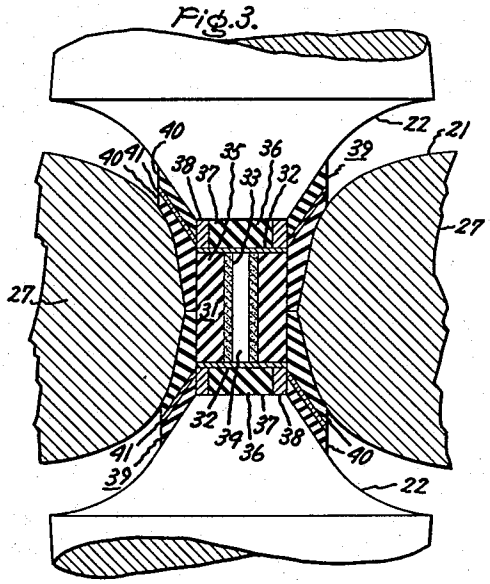
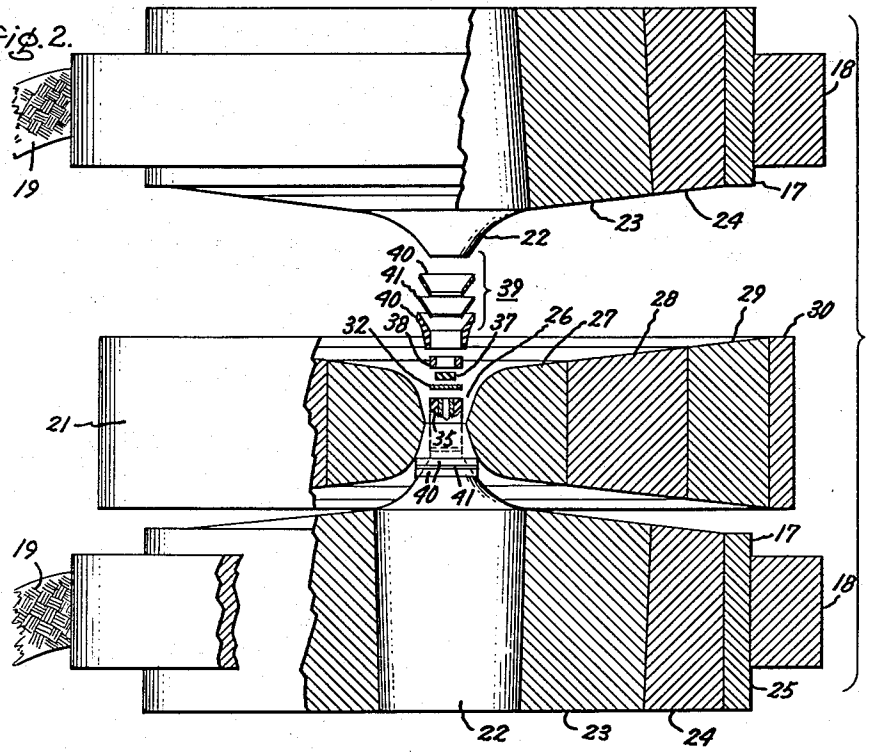


Fig. 2.



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2,996,763

DIAMOND MATERIAL

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This invention relates to radioactive diamond material and to the preparation thereof. More particularly, this invention is concerned with diamonds containing a surface coating of a radioactive metal and to the preparation thereof.

As is well known in the art, natural diamonds, particularly natural diamonds of the industrial variety, readily acquire and retain a charge of static electricity. This generally occurs when natural industrial diamonds come into contact with each other or with some other surface. The static charge on industrial diamonds, results in a number of problems in their use. Thus, when an attempt is being made to sort industrial diamonds, it is found that the static charge seriously interferes with the segregation of the diamonds into various size groups and interferes with the moving of the diamonds from one location to another. In addition, when natural diamonds are applied in uses such as in bearings or in grinding tools, the charge on the surface of the diamond tends to attract dust or other undesirable particles of matter to the surface of the diamond. When diamonds are used as bearing materials such as in time pieces and other delicate mechanisms, the dust or other particles attracted by the static charge on the surface causes abrasion of the surface supported by the bearing and, therefore, reduces or destroys the efficiency of the bearing. Where diamonds are used in industrial grinding tools, it is found that the static charge on the diamond surface causes particles of dust and of metal to adhere to the surface of the diamond and interfere with proper and accurate grinding.

An object of the present invention is to provide a diamond material which does not retain a charge of static electricity for any substantial period of time.

Another object of the present invention is to synthesize diamond material of the type described from non-diamond carbon.

These and other objects of my invention are accomplished by providing a radioactive diamond material comprising a diamond having a radioactive metal in adherent relation to the surface thereof. This radioactive diamond material is formed by a method which comprises subjecting non-diamond carbon to a pressure of at least 75,000 atmospheres, e.g. from 85,000 to 110,000, and preferably about 95,000 atmospheres, at a temperature of from 1200 to 2000° C. in the presence of (1) a radioactive material, preferably a radioactive metal, and (2) at least one metal selected from the class consisting of group VIII metals of the periodic table, and chromium, tantalum, manganese, and compounds of these metals which decompose to a metallic state under the conditions of the reaction.

My invention may be best understood by reference to the following detailed description taken in connection with the drawing in which:

FIG. 1 is a front elevational view of a hydraulic press with a high temperature high pressure apparatus useful in the practice of the present invention;

FIG. 2 is an enlarged, exploded sectional view of high temperature high pressure apparatus which is shown in FIG. 1; and

FIG. 3 is an enlarged sectional view of the reaction vessel and associated parts which are shown in FIGS. 1 and 2.

The term "radioactive" metal is used in the present application in its usual sense to refer to a metal which has the property of spontaneously emitting alpha, beta, or gamma rays by the disintegration of the nuclei of the atoms of the metal. As is well known, the radiation emitted by radioactive elements causes ionization of the medium surrounding the radioactive material or the medium through which the alpha, beta, or gamma rays pass. It is further understood that when, for example, air is ionized by rays from a radioactive material, the ionized air becomes, to some extent, a conductor of electricity. Thus, where the diamond material of the present invention acquires a static charge, the radioactivity of the surface layer of the diamond material causes ionization of the air or other medium surrounding the diamond material and this ionized air or other material carries away the static charge from the surface of the diamond material, rendering the diamond material electrically neutral.

As can be seen from the description of the ionization of the medium surrounding the diamond materials of the present invention and the "discharging" of the static charge on the diamond material, the diamond material of the present invention is free from the problems associated with natural diamonds. Thus, since the diamonds of the present invention do not retain a static charge, they may be sorted without the difficulty caused by any static charge on the material. In addition, when the diamonds of the present invention are used as bearings, they will have no tendency to attract dust or other abrasive particles to their surface. Furthermore, when the diamond material of the present invention is employed as abrasive material in abrasive wheels, there is again no tendency for any static charge to accumulate on the diamonds and therefore there is no tendency for the grinding wheel to attract particles of metal which may interfere with the accurate use of the grinding wheel.

In the copending application of H. T. Hall et al., Serial No. 488,116, filed February 14, 1955, now abandoned, and assigned to the same assignee as the present invention, there is described a method of making diamonds in which non-diamond carbon is subjected to a pressure of at least 75,000 atmospheres and preferably from 85,000 to 110,000 atmopheres, at a temperature of from 1200 to 2000° C. in the presence of a catalyst which is a metal selected from the class consisting of group VIII metals of the periodic table, and chromium, sodium, tantalum, aluminum, manganese, and compounds of these metals which decompose to a metallic state under the conditions of the reaction. The process of the present invention is carried out by the same method as that described in the aforementioned Hall et al. application except that the reaction is carried out also in the presence of a radioactive material.

The process of the present invention may be carried out in any type of apparatus capable of producing the pressures required at the temperature required. However, I prefer to employ apparatus of the type described in the application of H. T. Hall, Serial No. 488,050, filed February 14, 1955, now abandoned, and assigned to the same assignee as the present invention. The disclosure of this Hall application is hereby incorporated by reference into the present application. The apparatus disclosed in the aforementioned Hall application is a high pressure device for insertion between the platens of a hydraulic press. The high pressure device consists of an annular member defining a substantially cylindrical reaction area, and two conical, piston-type members designed to fit into the substantially cylindrical portion of the annular member from either side of said annular member. A reaction vessel which fits into the annular

member may be compressed by the two piston members to reach the pressures required in the practice of the present invention. The temperature required is obtained by any suitable means, such as, for example, by induction heating, by passing an electrical current through the reaction vessel, or by winding heating coils around the reaction vessel.

The apparatus of the aforementioned Hall application, Serial No. 488,050, is best described by reference to the accompanying drawing. In FIG. 1 of the drawing, a hydraulic press comprises a base 10 with a press bed 11 on which are mounted a plurality of vertical shafts 12 to support a carriage 13 with a hydraulic shaft 14. A pair of opposed, recessed pistons 15 and 16 on bed 11 and carriage 13 are recessed to partially position members 17 therein, each of which is provided with an electrical connection in the form of an annular conducting ring 18 with a connector 19 to supply electric current from a source of power (not shown) through member 17 to the high pressure high temperature reaction vessel which is described below. A layer of electrical insulation 20 is provided between at least one member 17 and its associated pistons 15 to prevent conduction of electrical current through the press. A pressure resisting member or belt 21 is positioned between opposed members 17 to provide a multi-staging pressure effect.

As is best shown in FIG. 2, each member 17 comprises a die 22 with surrounding binding rings 23 and 24. If desired, a soft steel safety ring 25 is located around binding ring 24. Conducting ring 18 is mounted around the periphery of safety ring 25 to conduct current through rings 25, 24 and 23 to die 22. Pressure resisting member 21, which is positioned between opposed members 17, tapers inwardly towards its center to provide an aperture 26 in axial alignment with opposed dies 22. Such a tapering effect produces greater strength in member 21 to resist pressure. Member 21 comprises an inner ring 27 surrounded by one or more binding rings 28, 29 and a soft steel safety ring 30.

As is best shown in FIGS. 2 and 3, a specimen holder 31, which is positioned in aperture 26 between dies 22, comprises a pair of spaced conductive disks 32 with a hollow conductive cylinder or reaction vessel 33 therebetween adapted to contain a specimen 34 to be subjected to high temperature high pressure conditions. A washer 35 of electrically insulating material is positioned around cylinder 33 between disks 32 to complete the assembly of specimen holder 31. If desired, specimen holder 31 may be in the form of a hollow casing which is in electrical contact with dies 22 but which is thermally insulated from inner ring 27. A washer 36 is positioned between each die 22 and its associated disk 32 to provide a heat insulating core 37 with a surrounding outer conductive ring 38 in electrical contact with the die. A laminated conical gasket assembly 39 surrounds each die 22 and comprises a pair of thermally and electrically insulating and pressure resisting conical washers 40 with a metallic washer 41 between adjacent washers 40. The outer pressure resisting washer 40 is tapered inwardly to be engaged on its exterior surface by the tapered surface of inner ring 27 of member 21 and on its interior surface by washer 35 of specimen holder 31. While only a pair of washers 40 with a single separating washer 41 are illustrated in the drawing, it has been found that a plurality of alternate washers further increases the size of specimen holder 31, the permissible motion between dies 22, and the pressure. Relatively high pressures are obtained when inner washer 40 and metallic washer 41 are eliminated and specimen holder 31 is shortened this corresponding thickness along the center line. However, pressures of the order of 40,000 to more than 100,000 atmospheres are produced when gasket assembly 39 is employed through additional increases in both relative motion and compression. Examples of suitable materials from which core 37 and washers 35 and 40 may be made are pyrophyllite and catlinite.

In the operation of the high temperature high pressure apparatus shown in FIGS. 1–3, each member 17 with associated conducting ring 18 and connector 19 is positioned partially within the recesses of its associated piston in the press. Specimen 34 which is to be subjected to a high temperature high pressure environment is placed in reaction vessel 33 within washer 35 between disks 32 to complete the assembly of specimen holder 31. Pressure resisting member 21 is positioned between opposing members 17 to locate specimen holder 31 in aperture 26 between dies 22.

Pressure is applied to specimen 34 by shaft 14 of the press. At the same time electrical current is supplied from one electrical connector, such as upper connector 19 to upper conducting ring 13, rings 25, 24 and 23, die 22, rim 38, and disk 32 to generate heat and pressure simultaneously in cylinder 33 of specimen holder 31. The current path continues from cylinder 33 through lower disk 32, rim 38, die 22, rings 23, 24 and 25, conducting ring 18, and connector 19 to the electrical source. Pressures in excess of 95,000 atmospheres at temperatures higher than 2000° C. have been maintained in such apparatus for periods of hours.

The reaction vessel or cylinder 33, described in Hall application Serial No. 488,050 referred to above, may be formed of any of the conventional materials of construction or of graphite. Where the reaction vessel is constructed of a metal, it is convenient to employ one of the metals which acts as a catalyst in the present invention. This vessel may then be filled with non-diamond carbon and a radioactive material and compressed so that the metal present in the vessel will serve as a catalyst for the transformation to diamond. When the reaction chamber or vessel is formed of graphite, it may be filled with a mixture of the catalyst material and the radioactive material and the compression of the graphite vessel with the catalyst and radioactive material at the pressures and temperatures required by the present invention results in the transformation of the non-diamond carbon into the radioactive material of the present invention. The reaction vessel or chamber may also be formed of a radioactive metal. Where this is the case, the reaction vessel is filled with non-diamond carbon and catalyst material and compression of the reaction vessel at the proper pressure and temperature causes transformation of the non-diamond carbon into the radioactive material of the present invention. Regardless of the material of construction of the reaction vessel, the non-diamond carbon, the catalyst, and the radioactive material may be admixed inside the vessel. Thus, mixtures of powdered graphite, powdered catalyst, and powdered radioactive material may be employed as a charge in the reaction vessel and the compression of the vessel and charge at the required temperature effects transformation to radioactive diamond material.

In the preferred embodiment of my invention, I employ a reaction vessel 33 comprising a cylinder of graphite having a hollowed out cylindrical center portion, the axis of the center portion being coaxial with the axis of the reaction vessel. Into this graphite reaction vessel is placed cylinders or disks of graphite, the catalyst, and the radioactive material. This reaction vessel is sealed at its ends by metallic disks which may be formed of any material of construction inert under the conditions of the reaction or it may be formed of a catalyst metal or a radioactive material. If desired, plugs of non-diamond carbon or metal may be placed in the ends of the reaction vessel before sealing. This reaction vessel is then placed in the apparatus described in the above-mentioned Hall application Serial No. 488,050 and subjected to the elevated temperature and pressure required to effect the transformation to radioactive diamond ma-

terial. Alternatively, instead of employing a reaction vessel, a cylinder of carbonaceous material, such as graphite, may be sandwiched between two disks, each formed of a radioactive metal, this sandwich in turn being sandwiched between two other disks formed of a metal which acts as a catalyst for the transformation. The sandwich is then placed in the high pressure apparatus and subjected to the conditions required to cause the transformation to radioactive diamond material. As a further alternative, a metallic reaction vessel may be filled with carbonaceous material in powder or solid form and the catalyst and radioactive material for the reaction may be supplied by admixing it with the powdered carbon or by forming end disks of either of these materials to seal the reaction vessel. This assembly is then subjected to the pressures and temperatures required. A reaction "vessel" may also be formed by compressing a mixture of non-diamond carbon, catalyst, and radioactive material until a cylinder is formed which fits into the substantially cylindrical aperture described in the above-mentioned Hall apparatus. Again, this latter apparatus may be employed in the usual manner at elevated temperatures and pressures to effect the transformation to radioactive diamond material.

In carrying out the process of the present invention, the temperature in the reaction vessel may be measured by a thermocouple located adjacent the reaction vessel previously mentioned. In carrying out this process, the time of reaction required to convert the reaction mixture into radioactive diamond material may vary from a few seconds up to several minutes or more depending on the particular charge to the reaction vessel. However, regardless of the charge to the reaction vessel, I have found that the radioactive diamond material of the present invention is formed within two to five minutes. Instead of observing the time which the reaction vessel is maintained at the reaction temperature, I may alternatively observe the progress of the reaction by the method described in the application of H. T. Hall, Serial No. 530,935, filed August 29, 1955, now U.S. Patent 2,947,608, issued August 2, 1960, and assigned to the same assignee as the present invention. This Hall application is hereby incorporated by reference into the present application. By the process of this Hall application, the mixture of non-diamond carbon, catalyst, and radioactive material, is first subjected to a pressure of at least 75,000 atmospheres and subsequently sufficient heat is applied to the mixture for sufficient time to cause an inflection in the electrical resistance of the mixture. When the inflection in the electrical resistance of the mixture occurs, the transformation of the mixture to the radioactive diamond material of the present invention has occurred.

The radioactive materials which may be employed in the practice of the present invention include all of the naturally occurring radioactive metals such as uranium, thorium, bismuth, rhenuium, lutecium, samarium, indium, rubidium and potassium. Also included within the radioactive metals employed in the practice of the present invention may be mentioned the decay products of naturally radioactive materials as well as all of the well known radioactive isotopes of metals. Included within these latter two groups of radioactive materials may be mentioned, for example, the radioactive isotopes of protactinium, actinium, neptunium, plutonium, radium, francium, radon, cobalt, etc.

In addition to the radioactive isotopes specifically mentioned above, it should be understood that other artificial metallic isotopes are also included within the scope of the present invention. As an illustration of other isotopes within the scope of the present invention, reference is made to all of those radioactive isotopes of metals listed in appendix 6 at page 464 of "Nuclear Radiation Physics" by Lapp and Andrews, Prentice-Hall Inc., New York (1948).

In preparing the radioactive diamond material of the present invention, the proportions of the three ingredients employed are not critical, and I have found that suitable radioactive diamond material has been formed regardless of the relative concentrations of the non-diamond carbon, the catalyst, and the radioactive metal. However, in the preferred embodiment of this invention, it is preferred to have the ingredients present in the proportions of about six parts by volume of non-diamond carbon, six parts by volume of catalyst, and one part by volume of the radioactive metal. Regardless of the proportions of ingredients and the physical location of the various ingredients in the reaction vessel, it has been found that the resulting product comprises diamond having in adherent relation to the surface thereof a radioactive metal.

The thickness of the radioactive layer on the outside of the diamond varies to a minor extent with the proportions of ingredients employed in the reaction. However, regardless of the proportions employed, I have found that the thickness of the radioactive metal layer lies within the range of about 0.5 to 5.0 microns. The relative weight of the diamond to the radioactive layer is dependent upon a number of factors, including the density of the radioactive material, the concentration of the reactants employed in forming the diamond material, and the size and shape of the diamonds. Because of these variables, it is impossible to specify an exact ratio of the weight of the carbon in the diamond to the weight of the radioactive metal. However, I have obtained satisfactory products where the ratio of the weight of diamond to the weight of radioactive metal varied from about 10 to 20.

The type and intensity of radiation emitted from the radioactive diamond material of the present invention is, of course, dependent upon the type of radioactive material employed. Thus, where the radioactive material employed is thorium, the radiation from the radioactive diamond material is that well known radiation emitted by thorium. Similarly, where the radioactive material is a material other than thorium, the radioactivity is that characteristic of that other radioactive material. The half life of the radiation from the radioactive diamond materials is also dependent on the radioactive metal employed and is the same as the half life of the radioactivity commonly observed from said radioactive metal.

Since the radioactive materials of the present invention are similar to other radioactive materials, some degree of care is necessary in handling the products of the present invention. The degree of care required in handling the radioactive diamond materials is similar to that degree required in handling the radioactive metals themselves.

The following examples are illustrative of the practice of my invention and are not intended for purposes of limitation.

In the following examples, the reaction vessel consisted of a cylindrical graphite member having a cylindrical aperture therethrough, the axis of the aperture being coaxial with that of the cylinder. The walls of the cylindrical graphite member were approximately one-sixth as thick as its diameter and the length of the cylinder was approximately three-fifths the times of its outside diameter.

Example 1

Into the reaction vessel described were placed two nickel cylinders to act as a catalyst for the reaction, two thorium disks as the radioactive metal, and one graphite cylinder which was to be converted to diamond. These reactants were assembled in the reaction vessel in the following order: a nickel cylinder, a thorium disk, the carbon cylinder, the second thorium disk, and the second nickel cylinder. The diameters of each of these rods were substantially equal to the internal diameter of the reaction vessel and the length of each rod was selected so that there was present in the reaction vessel about 12 parts by volume of carbon, 12 parts by volume of nickel, and one

part by volume of thorium. The reaction vessel was then sealed at each end with a tantalum disk and subjected to a pressure of about 95,000 atmospheres at a temperature of about 1500° C. for about six minutes. At the end of this time the reaction vessel was allowed to cool, the pressure was released, and the radioactive diamond material was separated from the matrix in which it was formed by solution of the matrix in fuming red nitric acid. From a measurement of the radioactivity of the diamond material, it was determined that the thorium content of the radioactive diamond material was approximately 5 percent by weight based on the total weight of the diamond material. This is equivalent to about 2.5 thorium atoms per thousand carbon atoms and represents a film about 0.75 micron thick of thorium on the surface of the diamond. The fact that the thorium was present as a surface layer on the diamond was established by boiling the radioactive diamond material just described in aqua regia for 24 hours, after which time the diamond material exhibited no further radioactivity despite the fact that the appearance of the diamond material was unchanged.

Example 2

The procedure of Example 1 was repeated except that the ratio of the volume of the various reactants was as follows: 6 parts by volume of carbon, 6 parts by volume of nickel, and one part by volume of thorium. After the reaction vessel had been exposed to a pressure of about 95,000 atmospheres at a temperature of about 1500° C. for five minutes, the resulting radioactive diamond material was isolated and found to have a radioactivity corresponding to 10 percent thorium by weight, which is equivalent to 5 thorium atoms per thousand atoms and represents a film of thorium about 1.5 microns thick.

Although the catalyst and the radioactive materials which may be employed in the practice of the present invention have been described only in terms of pure metals, it should be understood that compounds of these metals which decompose under the conditions of the reaction may also be employed. Thus, compounds of the catalyst and of the radioactive metal which decompose into pure metal under the conditions of the reaction include, for example, the carbides, sulfides, carbonyls, cyanides, ferrotungstates, ferritungstates, oxides, nitrides, nitrates, hydrides, chlorides, molybdates, arsenates, acetates, oxalates, carbonates, borates, chromates, phosphates, phosphides, permanganates, silicates, sulfates, tungstates, etc. Specific examples of decomposable compounds usable as catalysts in the present invention include ferrous sulfide, iron carbonyls, palladium chloride, chromium carbide, tantalum hydride, sodium fluoride, nickel permanganate, cobalt acetate, nickel sulfate, etc. Examples of radioactive compounds which may be employed include, for example, uranium fluorides, thorium carbides, radium sulfates, etc.

Although the above examples have described the reaction of the present invention only at a pressure of 95,000 atmospheres and a temperature of about 1500° C., it should be understood that any pressure inexcess of 75,000 atmospheres is satisfactory for the practice of this invention. My preferred pressure range is from about 80,000 to 110,000 atmospheres. Similarly, my temperature range may vary from 1200° to 2000° C., and preferably from about 1400° C. to 1800° C.

The radioactive diamond material of the present invention is useful for all of those uses to which diamonds are commonly put. In addition, this radioactive diamond material is particularly useful as the abrasive medium in an abrasive wheel. In addition, the radioactive diamond material of the present invention is particularly suitable for use as a bearing surface in applications where the bearing must withstand an extremely high load and where the dust which would be attracted by an ordinary diamond bearing would ruin the effectiveness of the bearing action. Thus, the radioactive diamond materials of the present invention are particularly suitable as "jewels" for use in clocks, timers, and the like where dust might interfere with the accuracy of the device.

What I claim as new and desire to secure by Letters Patent of the United States is:

1. The method of synthetically making a radioactive diamond having a radioactivity only on the surface layer thereof, which comprises (1) combining non-diamond carbon with a radioactive metal and at least one metal catalyst selected from the class consisting of group VIII metals of the periodic table, chromium, tantalum, manganese, and compounds of these metals which decompose to the metallic state under the following conditions of reaction, (2) subjecting such mixture of non-diamond carbon, radioactive metal, and metal catalyst to a pressure of at least about 75,000 atmospheres at a temperature of from about 1200° to 2000° C., and (3) recovering the formed radioactive diamond having radioactivity only on its surface layer.

2. The method of synthetically making a radioactive diamond having a radioactivity only on the surface layer thereof, which comprises (1) combining non-diamond carbon with thorium and at least one metal catalyst selected from the class consisting of group VIII metals of the periodic table and chromium, tantalum, manganese, and compounds of these metals which decompose to the metallic state under the following conditions of reaction, (2) subjecting such non-diamond carbon, thorium, and metal catalyst to a pressure of at least about 75,000 atmospheres at a temperature of from about 1200° to 2000° C., and (3) recovering the formed radioactive diamond having radioactivity only on its surface layer.

3. The method of synthetically making radioactive diamond having a radioactivity only on the surface layer thereof, which comprises (1) combining non-diamond carbon with a radioactive metal and nickel, (2) subjecting the said non-diamond carbon, radioactive metal, and nickel to a pressure of at least about 75,000 atmospheres at a temperature of from about 1200° to 2000° C., and (3) recovering the formed radioactive diamond having a radioactivity only on its surface layer.

4. The method of synthetically making radioactive diamond having a radioactivity only on the its surface layer thereof, which comprises (1) combining non-diamond carbon with thorium and nickel, (2) subjecting the combined non-diamond carbon, thorium, and nickel to a pressure of at least about 75,000 atmospheres, at a temperature of from about 1200° to 2000° C., and (3) isolating the radioactive diamond so formed having a radioactivity only on its surface layer.

5. The method of synthetically making radioactive diamond having a radioactivity only on the surface layer thereof, which comprises (1) combining non-diamond carbon with thorium and nickel, (2) subjecting this aforesaid non-diamond carbon, thorium and nickel to a pressure of about 95,000 atmospheres at a temperature of about 1500° C., and (3) thereafter recovering the radioactive diamond material so formed having a radioactivity only on its surface layer.

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