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K. C. DE WALT  
PROCESSING GRAPHITE  
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Fig. 1.

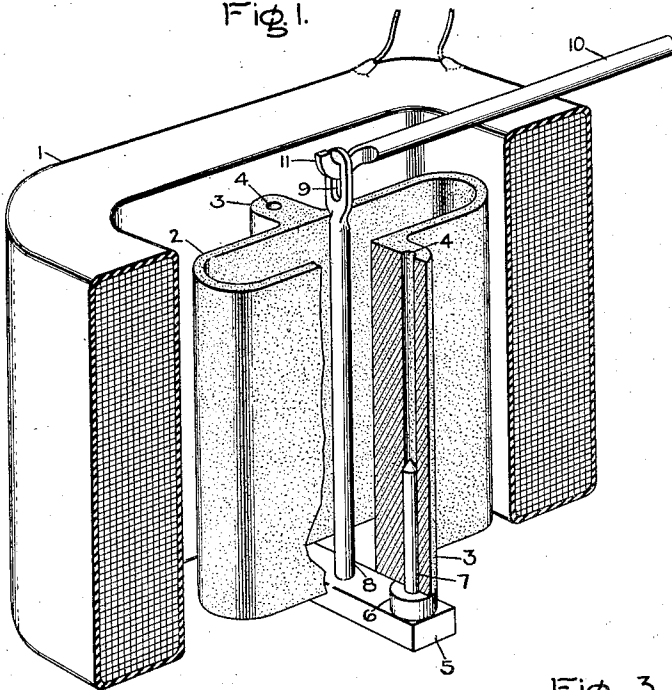


Fig. 2.

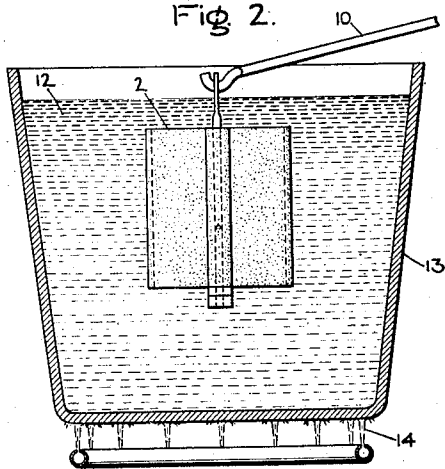
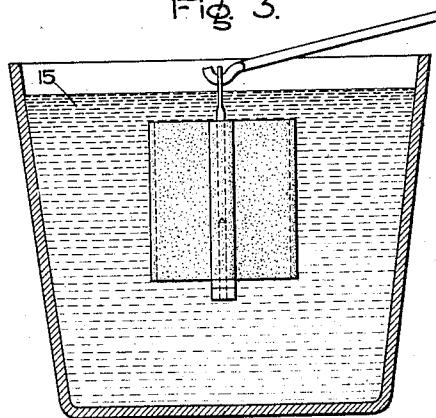


Fig. 3.



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## UNITED STATES PATENT OFFICE

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## PROCESSING GRAPHITE

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7 Claims. (Cl. 250—27.5)

The present invention relates to electric discharge devices and more particularly to the materials of which the electrodes of said devices are made.

As the result of considerable research and developmental work, it has been found that graphite or specially treated carbon offers many advantages as electrode material over metals which have been most commonly employed for this purpose. In the first place, graphite lends itself readily to well-known extrusion methods so that electrodes of simple configuration may be cheaply made. The material is fairly soft so that if desired and necessary, the electrode may be easily machined out of solid blocks of graphite. There is less tendency for the finished graphite electrode which often is constituted of relatively thin material, to warp than is the case with metal electrodes when subjected to heat and consequently, less supporting structure is necessary within the tube. Graphite has a relatively high electrical conductivity so that there is no appreciable energy loss in the electrode and moreover, has a relatively low vapor pressure so that it produces no deposits on insulators in the tube which might give rise to excessive electrode leakage or at least to blackening of the bulb during exhaust. Finally, graphite offers the most desirable advantage over metal of being able to dissipate greater quantities of heat, since it constitutes more nearly an ideal "black body" and in view of its much higher melting point than that of metal, may be operated at higher temperatures, all of which results in providing a tube which gives a higher operating plate dissipation.

In contrast to the many advantages which graphite has over metal for electrode material, it is subject to a most pronounced disadvantage which has tended to restrict its use generally in electron discharge devices. It has been found that there is usually a film or layer of very fine particles collected on the surface of a graphite electrode, in the form of a powder, and these particles are retained for the most part in the porous surface structure. When an anode or a control member, or both, of a high voltage electric discharge device are made of graphite, the electric and thermal forces within the tube tend to detach these fine particles from the surface and the resulting dust migrates within the envelope and often comes to rest on the cathode surface which may be an indirectly heated or a directly heated member coated with thermionically active agent. The result of this deposit is to reduce the electron

emission to a relatively low value and hence to decrease the operating efficiency of the tube.

In case only the anode is made of graphite and the grid fabricated of metal, the graphite particles attracted from the anode may lodge on the grid or any other electrode in the tube and cause arcing and subsequent breakdown resulting from the cold emission produced by the intense electric fields at the points of the respective particles.

In order to eliminate this condition, it has been proposed to brush the electrode with a steel bristle brush, and to wipe the member with a soft cloth prior to assembly. While this has done much to relieve some of the difficulties attending the use of graphite, there still remains a considerable amount of trouble from the same cause even though special treatments on exhaust and added aging schedules of manufacture have been employed. It seems that as the voltage between the cathode and anode is increased, the deleterious effects of the graphite powder are multiplied manifold so that any process or technique of ridding the graphite surface still further of these dust particles is highly desirable, particularly in the field of high voltage devices.

An object of the present invention is to improve the technique of treating graphite so that the electrode material will be even less subject to the production of graphite dust than heretofore, and to provide such a process as may be readily performed without necessitating expensive and elaborate apparatus.

In carrying out this object, the improved process consists essentially of heating the finished graphite electrode, i. e. one that is ready to be mounted within the tube, in air by induction or in any other suitable manner to about 1000° C. A high frequency induction furnace is preferred for this purpose since its heating effect may be readily controlled and reproduced. After the heat treatment for about one minute, or until substantially all residue has been burned from the electrode, as evidenced by the disappearance of smoke or vapors, the graphite may be removed from the induction coil or other type of furnace and while red-hot quenched in a hot non-inflammable liquid such as carbon tetrachloride, acetic acid (28% solution), or a glycerin-water solution (25% glycerin), or even tap or distilled water. It is preferred to have the water maintained at a somewhat elevated temperature, approximately 90° C. rather than at room temperature because the higher the temperature of the liquid, the less sudden will be the quenching effect of the graphite electrode and

the less tendency will there be to break or otherwise distort the form of the electrode during the cooling process. For practical reasons, it is preferred to employ distilled water for this purpose so as not to introduce any foreign material such as mineral salts, etc. into the electrode.

Shortly after immersing the graphite electrode in the water, a violent reaction takes place which in effect, blasts the surface free of all impurities, including the dust particles referred to hereinbefore. The particles tend to settle in the water and are permanently removed from the electrode. As stated hereinbefore, the lower the temperature of the water or the higher the temperature of the electrode at the time the quenching takes place, the more violent will be the blasting action. Consequently, these temperatures must be predetermined to give the optimum results, i. e. by producing as violent a reaction as possible without breaking or otherwise distorting the electrode.

The manner in which the electrode surface is freed of impurities is not known completely at present but in general, I believe that the reaction is produced by a rapid cooling of the surface which permits the water to penetrate the surface. The interior of the graphite is still hot enough to vaporize the water and produce steam at a pressure sufficient to create the blasting effect. The action literally detaches in a violent manner the loose dust and impurities from the graphite.

After the reaction is completed, the graphite may be removed from the quenching bath and rinsed in a liquid bath maintained at a somewhat lower temperature, for example, 60° to 70° C. For this purpose, I prefer to use distilled water, since it is non-inflammable and does not leave deposits which are undesirable and is easy to remove in the manner described hereinafter.

The electrode may then be removed from the rinsing bath and allowed to dry in air for a short time, i. e. for approximately ½ hour, to evaporate the surface water, after which the water absorbed within the interior of the electrode may be completely removed by firing the anode by induction or in any other suitable manner in vacuum or by firing for approximately 10 minutes at approximately 1200° C. in a hydrogen atmosphere. The electrode is then ready to be mounted on its supporting structure and sealed within the tube. It is found that after having been given the treatment above described, it is no longer necessary to rub or brush the graphite. In case the electrode is not to be used for a time, it is preferable to wrap the same in cellophane or other protective covering until ready for use.

The apparatus employed in the dust-removing treatment described hereinbefore is quite simple and inexpensive. Reference should now be made to the drawing in which Fig. 1 shows in perspective and partly in section, an induction coil and a graphite electrode mounted in position to receive a heat treatment; Fig. 2 shows the position of the electrode in the quenching bath, while Fig. 3 shows the next step in the process in which the electrode is rinsed in warm water.

In the drawing, numeral 1 represents a coil having a configuration which approximates that of the electrode 2. Oscillatory current obtained from a source not shown flows through the coil and sets up electro-magnetic lines which tend to heat any conducting material of a hollow and perimetrical closed configuration contained within the coil, as is well known in the art. The

electrode 2 is illustrated as taking the form of a well-known type of anode, which has a general box-like configuration and a thickness in the thinner parts of approximately ¼". Intermediate the ends of the box there are two oppositely directed flanges 3 constituted of solid material, both of which contain a longitudinal opening 4 which is used for mounting purposes. In order to apply the high frequency coil or furnace to the graphite electrode or vice versa, it is preferable to support the electrode on a metal cradle so that the hands of the operator never have to come in contact with the electrode either before or after the heat treatment and thereby contaminate the surface thereof. While any suitable form of cradle will suffice, I prefer to employ a frame which includes a rectangular bar member 5 of metal on which at each end there is secured a heavy metal washer 6. Projecting upwardly from these washers, there is a pair of pins 7 tapered at their uppermost ends, which are so positioned along the bar member as to fit snugly within the openings 4 already provided in the electrode. It is apparent that these rigidly positioned pins provide lateral support to the thin walls of the hollow electrode. The metal bar 5 carries at the center a rod upright member 8 of somewhat longer length than the electrode and which terminates at the top in an eyelet provided with a slot 9. There is a rod member 10, which terminates at one end, in a handle adapted to be grasped by an operator and at the other end, in a flattened hook 11 which engages the slot 9.

Obviously, by grasping the handle of the rod 10, an operator may lift the entire electrode 2 which rests on the metal bar 5. After having been given the high frequency heat treatment described hereinbefore and illustrated in Fig. 1, the electrode may be removed from the coil and then plunged in the heated liquid 12 contained within a receptacle 13 of suitable shape and size, maintained in a heated condition by means of gas jets 14. While in the liquid 12, the graphite electrode undergoes a violent reaction which removes the dust particles and foreign material from the surface so that the electrode comes from the quenching bath in an extremely clean condition. The blasting effect is so severe and the stresses set up in the electrode so great that the thin walls would tend to fracture in the absence of the lateral support offered by the cradle.

The electrode may then be lifted from the quenching bath after the reaction has been completed and immersed in the rinsing bath which preferably contains distilled water 15, maintained at a lower temperature than the water 12 in the quenching bath, after which the electrode is allowed to dry in air for a short time in order to evaporate the surface water and finally, is fired either in a vacuum or in a hydrogen atmosphere as described hereinbefore.

The electrode is ready to be mounted in a vacuum tube and after the treatment described hereinbefore, is substantially devoid of graphite dust particles or other source of contamination which might give rise to discharge or other irregularities of operation in the tube.

While I have illustrated and to some extent described my invention in connection with a graphite anode of an electron discharge device, it will be understood that the invention is not limited to the illustrated form or type of electrode but has application to the treatment in general of graphite or other specially prepared

carbon in which it is desired to remove the fine particles which inevitably form on the surface thereof. Obviously, grids or control members for electric discharge tubes may be treated in precisely the same manner as the illustrated anode, in which case, a specially designed cradle or supporting frame may be provided, as will readily occur to those skilled in the art.

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

1. The method of treating graphite which consists in heating the graphite to a temperature at which substantially all residue is removed by volatilization, then immersing the heated graphite in a non-inflammable liquid so as to cause a violent reaction within the graphite and to blast the surface free of loose particles.

2. The method of treating a graphite electrode which consists in heating the electrode to a temperature at which substantially all residue is removed by volatilization, then plunging the heated electrode into water to cause a violent reaction within the electrode and to blast the surface free of loose particles.

3. The method of treating a graphite electrode which consists in heating the electrode to approximately 1000° C., then quenching the heated electrode in water to cause a violent reaction within the electrode and to blast the surface free of loose particles.

4. The method of treating a graphite electrode which consists in heating the electrode by induction to approximately 1000° C., then quenching the heated electrode in water at a temperature of approximately 90° C. to cause a violent reaction within the electrode and to blast the surface free of loose particles.

5. The method of treating a graphite electrode which consists in heating the electrode by induction to approximately 1000° C., then quenching the heated electrode in water at a temperature of approximately 90° C. to cause a violent reaction within the electrode and to blast the surface free of loose particles, then rinsing the cooled electrode in water at a temperature of approximately 60° to 70° C.

6. The method of treating a graphite electrode which consists in heating the electrode by induction to approximately 1000° C., then quenching the heated electrode in water at a temperature of approximately 90° C., then rinsing the cooled electrode in distilled water at a temperature of approximately 60° to 70° C., drying the electrode, and finally firing the electrode in a hydrogen atmosphere whereby the absorbed water is completely removed.

7. The method of treating graphite which consists in heating the graphite solely in a gaseous atmosphere, then immersing the heated graphite in a non-inflammable liquid to cause a violent reaction within the graphite and to blast the surface free of loose particles.

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