

UNITED STATES PATENT OFFICE

2,167,128

METHOD OF PRODUCING SUPERFICIAL
CARBONIZATION OF METALS AND AL-
LOYSEdward William Skelton, Erdington, Birming-
ham, England, assignor to The International
Nickel Company, Inc., New York, N. Y., a cor-
poration of DelawareNo Drawing. Application March 5, 1937, Serial
No. 129,193. In Great Britain March 27, 1936

12 Claims. (Cl. 250—27.5)

The present invention relates to a method of producing superficial carbonization of metals and alloys, to an electrode constituted of such superficially carbonized metals and alloys, and to an electron tube containing such an electrode.

It is well known that for various reasons, it has often been desirable to blacken a metallic surface. For example, it is known that in order to facilitate radiation from a surface that surface should be blackened. A particular instance is that of an electron discharge tube, the grid and plate electrodes of which tend to heat up in operation and to bring about undesired electronic emission. In order that the heat may be radiated as quickly as possible from such electrodes, it is of advantage to blacken their surfaces and various methods of rendering the surfaces black by carbonization have already been suggested or attempted.

It is an object of the invention to provide an improved process of producing a carbonized surface on a metal or alloy.

It is another object of the invention to provide an improved process of carbonizing metallic strip especially a strip used in thermionic discharge tubes.

The invention also contemplates the provision of a method of carbonizing metallic strip within the envelope of a thermionic discharge tube.

Other objects and advantages will become apparent from the following description of a preferred embodiment of the invention.

According to the present invention a carbonized surface is produced on metals or alloys by employing a carbon-containing metal or alloy and heating it in vacuum or in an atmosphere incapable of reacting rapidly with carbon until a firmly adhering film of carbon is formed on the surface.

The invention may be applied with particular advantage to the treatment of nickel, but is not limited to the treatment of any particular metal or alloy.

For the purpose of giving those skilled in the art a better understanding of the invention, the following description is given of a preferred embodiment of the invention.

In accordance with the principles of the present invention, it is necessary to co-ordinate the carbon content, the general composition of the material, the duration of the heating and the temperature to produce a satisfactory deposit of carbon. In general, as the temperature decreases the time required to reach a given blackness of carbon increases and as the carbon content de-

creases the temperature to be used decreases also. Thus, in the case of nickel the carbon content should be at least 0.1% and if it is as low as this the temperature should not exceed 650° C. on heating in vacuum. If, as is preferred, the carbon content is 0.25% or 0.30% or over, the temperature may advantageously be from 900° C. to 950° C. although it may lie anywhere between 500 and 950° C. If the carbon content is as high as 0.5% temperatures up to 1200° C. may be employed.

The foregoing relationship between maximum allowable temperatures and carbon contents may be approximately expressed algebraically by the equation

$$T=1400C+500$$

where T is the temperature in degrees centigrade and C is the carbon content in percent. The duration of heating varies as stated above, but as an example if a temperature of 850° C. were employed several hours would be needed to produce a substantial carbon deposit even with a fairly high percentage of carbon, (say 0.5%) whereas only a few minutes are necessary if a temperature of 1000° C. is employed. Other factors that affect the duration of the heating are the nature of the heating chamber and the surrounding atmosphere.

If the carbonized material is intended to be used in the construction of electrodes for electron discharge tubes, it may be treated in accordance with the invention when in the form of strip, rod or wire, or after it has been formed into an electrode, either before or after assembly in the discharge tube. This discharge tube may have the structure of a conventional thermionic discharge tube.

By way of example a suitable material which may be carbonized in accordance with the invention and used for the electrodes of a discharge tube is nickel wire or strip comprising 0.35% carbon, 0.05% silicon, 0.13% iron, 0.04% copper, 0.04% magnesium, a trace of manganese, and the rest nickel. After heating this material in vacuum (for example, in an exhausted silica tube placed in a furnace) at a temperature of about 900° C. for 30 minutes, it will be found to have acquired a firmly adhering deposit of carbon.

Instead of employing a vacuum during the blackening process, an atmosphere of nitrogen, argon, helium or other gas incapable of reacting rapidly with carbon may be employed.

In the manufacture of a discharge tube, the electrodes may be mounted in the tube prior to the formation of the film of carbon. The en-

velope is then exhausted and the carbonizing operation carried out. Owing to the high temperature and the vacuum, the usual degassing will proceed simultaneously, thus rendering unnecessary separate heating for degassing purposes. Advantageously all the electrodes of a tube except the cathode may be made of carbon-containing metal, assembled in the tube and then heated as described.

The invention includes a discharge tube embodying an electrode with a blackened surface produced in any of the ways described above, and particularly a discharge tube having the surfaces of all the electrodes except the cathode blackened as just described.

It is to be observed that the present invention provides a process in which a firmly adhering film of carbon is produced on metals or alloys by employing a carbon-containing metal or alloy and heating it in vacuum or in an inert atmosphere until a proportion of the carbon is expelled. In the claims where the expression "in an atmosphere incapable of reacting rapidly with carbon" is used, it is to be understood as including within its scope not only inert gaseous atmospheres such as nitrogen, argon, helium, etc., but rarified atmospheres or vacuum as well. In the manufacture of a discharge tube, the electrodes may be mounted prior to the formation of the carbon film and the carbonizing and degassing operations carried out simultaneously.

I claim:

1. The method of producing a carbonized surface on nickel and nickel alloys which comprises employing a carbon-containing metal, containing sufficient carbon to become carbonized, and heating it within the temperature range of about 500° C. to about 500° C. plus 1400 times the carbon content in percent, said heating being carried out in an atmosphere incapable of reacting rapidly with carbon until a firmly adhering film of carbon is formed on the surface.

2. The method of producing a carbonized surface on nickel, which comprises heating nickel containing at least 0.1% carbon at a temperature within the range of about 500° C. to 1200° C. in an atmosphere incapable of reacting rapidly with carbon until a firmly adhering film of carbon is formed on the surface.

3. A method according to claim 2 in which the carbon content of the nickel is 0.25% or over and the heating is conducted at a temperature lying between 500 and 950° C.

4. The method of producing a carbonized surface on a nickel alloy for the manufacture of electrodes for electron discharge tubes which comprises employing a carbon-containing nickel alloy containing sufficient carbon to become carbonized, and heating it within the temperature range of about 500° C. to about 500° C. plus 1400 times the carbon content in percent, said heating being carried out in an atmosphere incapable

of reacting rapidly with carbon until a firmly adhering film of carbon is formed on the surface.

5. The method of producing a carbonized surface on a nickel alloy which comprises employing a carbon-containing nickel alloy containing sufficient carbon to become carbonized, and heating it at a temperature within the range of about 500° C. to 1200° C. within the envelope of the tube after exhaustion thereof and simultaneously with the degassing of the electrodes until a firmly adhering film of carbon is formed on the surface.

6. The process of manufacturing an electron discharge tube comprising mounting in the tube an electrode made of a nickel alloy containing sufficient carbon to become carbonized, and heating the mounted electrode at a temperature within the range of about 500° C. to 1200° C. in an atmosphere incapable of reacting rapidly with carbon until a firmly adhering film of carbon is formed on the surface.

7. A process as set forth in claim 6 in which the electrode is made of nickel containing at least 0.1% carbon.

8. A process as set forth in claim 6 in which the electrode is made of nickel containing at least 0.25% carbon and the heating is conducted at a temperature between about 500 and about 950° C.

9. A process as set forth in claim 6 in which the electrode is made of an alloy containing about 0.35% carbon, about 0.05% silicon, about 0.13% iron, about 0.04% copper, about 0.04% magnesium, a trace of manganese and the rest substantially nickel and the heating is conducted at about 900° C. for about 30 minutes.

10. The method of producing a carbonized surface on nickel alloy, which comprises heating an alloy containing about 0.35% carbon, about 0.05% silicon, about 0.13% iron, about 0.04% copper, about 0.04% magnesium, a trace of manganese and the rest substantially nickel in vacuum at a temperature of about 900° C. for about 30 minutes to form a firmly adhering film of carbon on the surface.

11. An electrode element having relatively thin cross-section composed of heat treated nickel or nickel alloy containing carbon throughout the cross-section and having uncombined carbon occurring immediately under the surface of said element and at the exterior thereof.

12. An electrode element having relatively thin cross-section composed of a heat treated alloy containing about 0.35% carbon, about 0.05 silicon, about 0.13% iron, about 0.04% copper, about 0.04% magnesium, a trace of manganese and the rest nickel, the carbon being at least in part uncombined and occurring immediately under the surface of said element and at the exterior thereof.

EDWARD WILLIAM SKELTON.