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PROCESS OF TREATING CARBONACEOUS ARTICLES

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This invention relates to carbonaceous articles having a hard non-oxidizing surface and to a method of treating carbonaceous articles by a method analogous to cementation to obtain such a surface.

An object of the invention is to provide carbonaceous articles, more particularly electric resistors, tubes, crucibles, etc., made of graphite, clay graphite, or carbon, and adapted to be exposed to high temperatures, with a dense homogeneous surface layer which is substantially non-oxidizing and resistant to acids. Such surface layer is extremely hard and dense, possesses substantially the same low coefficient of expansion as graphite, and forms substantially an integral part of the article and increases the resistance of the article to fracture.

Another object of the invention is to provide an economical and practical method of treating carbonaceous articles of all shapes as above described and which method allows control of the depth of the coating.

Heretofore, articles of this character have not been produced with a hard, dense, non-oxidizing surface, nor was any method known whereby the depth of the treatment could be controlled with any accuracy.

According to the present invention, carbonaceous articles are packed in a mixture of finely divided flint and carbon and the temperature raised sufficiently high to produce a surface on the articles treated consisting substantially of silicon carbide. By flint is meant the purer forms of silica used in the glass and ceramic industries, but I do not restrict myself thereto, as other silicious materials such as sand give equivalent results when used as herein disclosed. By carbonaceous materials is meant graphite, coke, gas carbon, lamp black, and mixtures predominantly of carbon with clay etc., such as used in crucibles and other refractories. Carbonaceous materials as herein used, are also free of hydrocarbons or other volatiles.

This treatment may be carried out by embedding the articles in a mixture of flint and finely divided carbon or coke and then heating the mass to a temperature sufficient to effect the combination with the carbon of the articles, temperatures between about 3180° F. to 6000° F. being suitable for this. The depth of the coating is dependent upon the temperature and length of time of treatment.

The treatment is carried out in an induction furnace of commercial, intermediate, or high frequency, as articles of all shapes can be more uniformly heated and the treatment better controlled. The articles to be treated are placed in a container together with a mixture of flint and finely divided coke, the proportion of flint to coke preferably being about 63 parts by weight to 26 parts by weight, and the container, usually a tube or crucible, is fitted into the coil of an induction furnace and the current turned on.

By using an induction furnace the articles to be treated are uniformly heated and the temperature and period of heating can be accurately controlled to give the desired depth of coating. The size and shape of the article to be coated presents no difficulty as the heating is uniform throughout as contrasted with the irregular heating effected by a furnace heated from outside or one having a central core resistor through which the current is passed. Furthermore, in treating carbonaceous articles as described in an induction furnace, a uniform homogeneous dense layer of sufficient depth to adequately protect the article can be attained with ease. In the case of large articles this layer may extend to a depth of ¼″ or more. A uniform layer of such depth has not heretofore been commercially possible, and such articles as I have produced have not been commercially known.

Carbonaceous articles treated by my process have an integral dense surface which seems to be very pure silicon carbon, is homogeneous throughout, has substantially the same low coefficient of expansion as the material of the article, has a hardness of upwards of 7 on the Mohs scale, in most instances a hardness of 9 on the Mohs scale, is non-oxidizing even at very high temperatures, and is chemically resistant to acids, alkalies, etc. The layer is also free of fluxes or slags.

The proportion of flint to coke in the mixture in which the articles are embedded may

be varied but to produce a uniform, homogeneous and hard surface layer, a mixture of about 63 parts by weight of flint to 26 parts by weight of coke is most suitable.

The process is applicable to articles of carbon, graphite, graphite-clay mixtures and the like.

The following example is given to specifically illustrate the invention.

A crucible made of "Acheson" graphite and measuring 4" outside diameter, 3" inside diameter and 8" in length, was coated as follows: A quartz cylinder with a closed bottom, 10" inside diameter and 15" long, was packed with the crucible embedded in a mixture of flint and coke of the following composition, 445 ounces of comminuted flint and finely divided coke, the mixture being in the proportion of 63 parts by weight of flint to 26 parts by weight of coke. The flint, such as used for fine porcelain, had the following analysis:

	Per cent
Silica	99.43
Alumina	0.19
Titanium	0.03
Calcium	0.11
Magnesia	0.02
Iron	trace

Both the flint and coke were of greater fineness than one hundred mesh. The crucible was centrally located with respect to the walls and ends of the quartz cylinder. The cylinder was then placed into an induction coil having 40 turns and an inside diameter of 11" and 13" in height, and the induction coil was then connected in the circuit of an "Ajax-Northrup" 60,000 cycle converter, the current turned on and the converter adjusted to supply 11 k. w. to the induction coil holding the cylinder. This was permitted to run 2 hours at a total consumption of 22 k. w. h. The temperature by pyrometer was above 3500° F. The current was then turned off and the mass permitted to cool and the crucible removed.

The crucible was uniformly coated throughout with a uniform, homogeneous, and hard coating of silicon carbide. The coating showed a hardness of 9 on the Mohs scale and was about 1/8" deep.

It has been found advantageous to add a small amount, approximately 1 part, of high grade beach sand to the mixture of flint and carbon. By the use of this small proportion of sand, a coating is produced which is more resistant to cracking that might be caused through rough handling of the coated article. This advantage is less pronounced in a rigid article, such as a crucible where there is little likelihood of cracks developing under usual conditions.

Carbonaceous articles containing up to 40% of inert material such as clay have been successfully coated by the process herein described.

Electric furnace resistors made according to this process have been used substantially as produced from the furnace in heat treating, ceramic testing furnaces, kilns, etc., without requiring packing or other protective covering. Also, by this process the complete length of the resistor up to the metallic terminals can be protected so that special fittings are not necessary where connections are made with terminals, bus bars and the like. Further, resistors made according to this process only have their ohmic resistance increased up to about 20% when cold; when heated the conductivity is the same as graphite or carbon, but the saving effected by elimination of packing, etc., with my electrodes is much greater than the cost of slightly more cross section to obtain the desired conductivity. Also, by reason of eliminating packings, a hot resistor of this invention can be pulled out of the furnace and another put in without cooling the furnace which so far as I am aware has not heretofore been possible.

The invention claimed is

1. Process of treating carbonaceous articles comprising embedding the same in a mixture of flint and carbon, placing the mass in an induction furnace, and passing a current through said furnace to raise the temperature sufficiently above the vaporization point of flint and below the vaporization point of carbon to form an adherent surface layer of uniform, homogeneous and hard silicon carbide.

2. Process for treating hollow carbonaceous articles comprising embedding the same in a mixture of flint and carbon, placing the mass in an induction furnace centrally of the flux, and passing a current through said furnace to raise the temperature sufficiently above the vaporization point of flint and below the vaporization point of carbon to form an adherent surface layer of uniform, homogeneous and hard silicon carbide.

3. Process of treating carbonaceous articles comprising embedding same in a mixture of 63 parts by weight of flint to 26 parts by weight finely divided coke, placing the mass in an induction furnace, and passing a current through said furnace to raise the temperature sufficiently above the vaporization point of flint and below the vaporization point of carbon to form an adherent surface layer of uniform, homogeneous and hard silicon carbide.

Signed at New York, in the county of New York and State of New York this 28th day of August A. D. 1930.

MICHAEL J. MARCIN.