

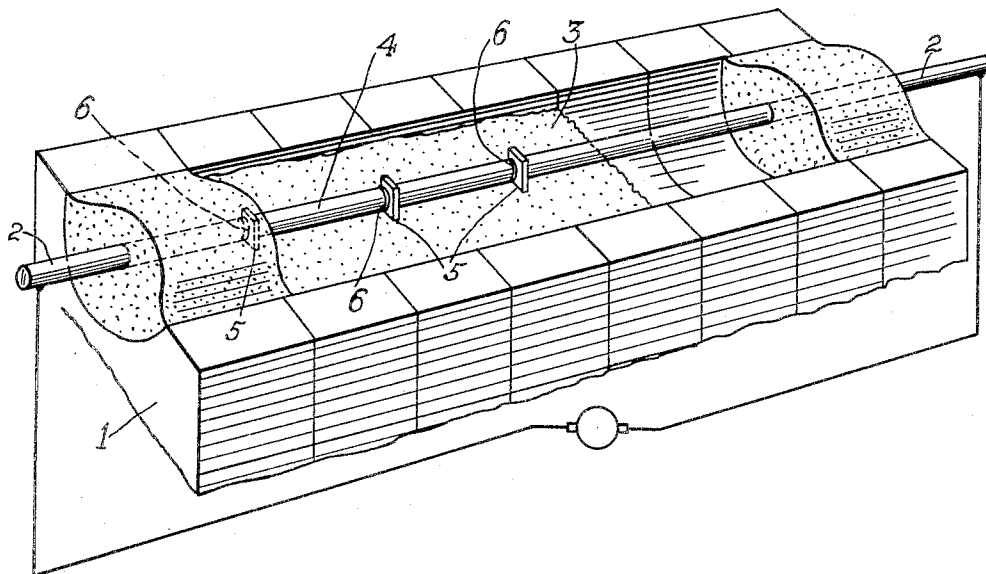
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ELECTRICAL RESISTOR AND MANUFACTURE THEREOF

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ELECTRICAL RESISTOR AND MANUFACTURE THEREOF

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This invention relates to the manufacture of electrical resistors composed essentially of silicon carbide, to which small additions of other materials are added to modify the electrical properties. The present application is a continuation-in-part of my copending application, U. S. Serial No. 676,665, filed November 23, 1923. This copending application discloses a process of manufacturing silicon carbide resistors wherein powdered metallic aluminum is incorporated into the original mix, after which the resistor elements are molded and heated to a temperature to cause a bonding of the silicon carbide grains, under conditions which afford at least a partial oxidation of the aluminum.

In the manufacture of silicon carbide resistors, and especially in the manufacture of domestic and industrial heating elements where the conductivity must be fairly high, the accurate control of the resistance of the element presents a difficult problem. A number of addition agents are known in the art for modifying the electrical properties of the resistor, but these are, in general, non-metallic in nature.

The addition of non-metallic materials which are at the same time non-conducting involves certain difficulties in cases where the resistor must carry considerable current, as in a heating element. It is difficult to obtain a uniform coating of the addition agent over the surface of the silicon carbide grain, and the resistor thus has a tendency to heat unevenly and develop hot spots. If an appreciable quantity of the addition agent is added in an effort to obtain a uniformly distributed film over the surface of the grain, the resistance of the element is usually increased much beyond the value desired. If the resistance is to be accurately controlled, it must be possible to produce comparatively small and consistent increases in the resistance of the element without affecting uniformity of heating. Furthermore, it is often desirable to cure the element by passing the current through the resistor itself, and when non-conducting addition agents are used, the electrical resistance of the unburned element

is so great that such a method of burning is impractical if not impossible.

I have found that the resistance of a silicon carbide resistor can be increased, and that this increase can be accurately controlled by adding powdered metallic aluminum to the mix and subsequently curing the element under conditions which will at least partially oxidize the aluminum. The aluminum is sufficiently unctuous so that even small amounts are uniformly distributed, and there is thus no tendency to produce uneven heating or hot spots. The increase produced is within the range desired, and the method is especially applicable for the control of the resistance of domestic heating elements.

It is well known that powdered aluminum as ordinarily manufactured has a great tendency to "leaf" or spread itself in a very thin film over a surface which is large compared with the amount of powder used. This unctuousness is particularly important when the material is used as an addition agent for a silicon carbide resistor mix. The aluminum powder spreads itself evenly over the surface of the grain, and the covering effect is very different from that obtained by the addition of the solid oxide, which is crystalline and difficult to spread. The unburned element is a better electrical conductor than if the aluminum were not present, so that the resistor may be cured by passing the current directly through it if desired.

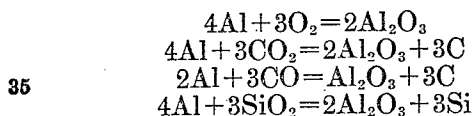
In carrying out my invention, I mix a small quantity of aluminum powder, usually from 1 to 5 per cent, with the silicon carbide grain, the type and grit size of which will be further specified. The dry ingredients, after thorough mixing, are tempered with a small amount of silicate of soda in water to produce a temporary bonding of the particles, and the mix is then molded and cured.

Any of the ordinary methods of molding may be employed. I have found that if the material is introduced into the mold in small portions and hand tamped as is well known in the art, the resulting resistor, when satisfactorily cured, will possess a uniformity of heating which is greatly desired.

In curing the resistor, the article is heated

to a sufficiently high temperature to cause a bonding of the crystal grains. The temperature may be as high as approximately 2200° C., and depends upon the process used for curing.

I have found that even when an atmosphere which is non-oxidizing with respect to silicon carbide is used for curing the resistors, the addition of aluminum powder produces a substantial increase in the specific electrical resistance of the finished element. For example, if the resistor is embedded in a sand carbon mixture, the atmosphere generated at high temperatures contains a large proportion of carbon monoxide, and would ordinarily be regarded as highly reducing, but it affects the aluminum so as to produce an increase rather than a decrease in the specific electrical resistance of the resistor. The reactions which take place with aluminum in the ordinary reducing atmosphere make possible the production of a resistor of relatively high specific resistance in which the silicon carbide is retained in a non-oxidized condition, even when the resistor is cured at an extremely high temperature. In practice there is evidence that aluminum is oxidized in practically any atmosphere which would be encountered in curing a silicon carbide resistor in accordance with one or more of the following equations:



Moreover, any conditions which will convert the aluminum to a non-conductor are in general of a character to be effective. As an example, it is well known that nitrogen will convert the metallic aluminum to a nitride.

The actual mechanism of the above reactions is not so important as the results obtained since the increase in resistance, even when the element has been cured under conditions which would ordinarily be considered reducing, as for example in a sand-carbon mix, has been verified experimentally.

In curing the resistors, I have found that a process described and claimed in my co-pending application, U. S. Serial No. 386,518, filed August 17, 1929, gives results which are satisfactory and reproducible. In this process, the molded elements are burned at a fairly low temperature (approximately 600° C.) before removal from the mold, in order to attain sufficient mechanical strength for handling. After removal from the mold, the resistors are dipped in a slip or slurry containing fine sand, carbon and water, and are subsequently dried, and placed in a burning furnace of the type shown in Figure 1, which consists of a refractory bottom or trough 1, with electrodes at either end for conducting

the current. The refractory bottom is covered to a depth of about two inches with a mixture 3 of fine silica and carbon, in the proportion of 75 parts of silica and 25 parts of carbon by weight. The relative amounts of silica and carbon are of importance in controlling the vapor of the silicon in the atmosphere generated about the resistor in the subsequent heating, and the proportion just given approximates the molecular proportion required to give maximum reduction of silicon dioxide to silicon according to the equation $\text{SiO}_2 + 2\text{C} = \text{Si} + 2\text{CO}$.

The coated resistors 4 are then placed carefully upon the bedding material described above in end-to-end relationship extending from one electrode to the other and electrically joined to each other and to the electrode by means of graphite blocks 5 and a paste 6 comprising graphite, silicon and water. When the rods have been laid down and joined to form a continuous path for the current they are covered with a silica and carbon mixture like that used for the bottom or bedding mix and are ready then for burning or curing. Burning or curing is then effected by applying current to the electrodes 2.

It will be seen from Figure 1 and from the above description that the resistors are so positioned in the furnace that their longitudinal axes lie within the path taken by the current in traversing the furnace from electrode to electrode and that their axes coincide with the axis of the path of the heating current. In fact the resistors carry the current after the first few minutes of the burning.

Because of the resistance of the uncured resistor, a voltage of approximately 500 volts per linear foot of furnace charge between electrodes may be required at the start to send sufficient current through the furnace to heat up the bedding material and the resistors. However, as the resistors approach the maximum temperature they approach the normal resistance for which they were designed and the applied voltage is cut down to avoid further substantial change. For example, a furnace load 6 feet long, comprising six resistors designed to operate at 110 volts may require approximately 6 times 110 volts for the final applied voltage, but at the start will require approximately 6 times 500 volts to force the heating current through the furnace.

Resistors of different cross-sectional areas require different current values for the production and maintenance of the maximum temperature and also require maintenance of the maximum temperature for somewhat different periods of time. Obviously resistors of large cross-sectional area require higher current values for the production of maximum temperatures than do smaller resistors of the same general nature. It may be stated,

however, by way of example, that a rod of circular cross-section approximately $\frac{1}{8}$ of an inch in diameter will require approximately 15 amperes for a total time of approximately 8 minutes.

The temperature of the process is believed to be between approximately 2000 and 2200° C., although exact measurement is difficult, owing to the fact that the material must be covered, and that silica fumes are evolved which interfere with the optical pyrometer readings. It is not necessary to regulate the temperature directly, since the temperature is usually controlled by regulating either the power input or amperage, the proper current being determined experimentally for a given diameter of element and a given amount of covering material. It has been found that the conditions above stated (i. e. 15 amperes for 8 minutes with a $\frac{1}{8}$ inch diameter element) will produce a satisfactory resistor.

The aluminum powder may be used either with or without other addition agents, depending upon the resistance desired. In the following table three examples of mixes containing aluminum are given, together with a similar mix containing no aluminum. The approximate specific resistance of the element, when burned in accordance with the process described above, is also given. These data illustrate the accurate control offered by a variation in the addition agents present. The remainder of the mix is practically identical in the three cases, and the conditions of curing were also identical;

	I	II	III	IV
120 mesh silicon carbide-----	30	30	30	30
280 mesh silicon carbide-----	70	70	70	70
Carbon flour-----	2.5	2.5	2.5	none
Aluminum powder-----	none	1	2	2.5
Sodium silicate solution, 28% Be-----	2.5	2.5	2.5	2.5
Specific resistance—approximate (ohms/cm ³)-----	.19	.39	.65	.96

The resistance values given are for an operating temperature of 1000° C. The actual resistance obtained and the increase produced by the aluminum added will depend upon the grit sizes and character of the silicon carbide grain used. The usual silicon carbide resistor made in accordance with the process described above has a specific resistance of from about .06 to .20 at an operating temperature of 1000° C. when no addition agent is added to the mix to increase the specific resistance. It will be observed from the above table that a resistor having a specific resistance as high as .96 can be obtained by adding 2.5 percent aluminum to the mix, and that the resistances can be varied by comparatively uniform steps over a range of several hundred percent. By using an intermediate quantity of aluminum practically any resistance between the limits produced by mixes I and IV can be obtained.

Although I prefer to use aluminum powder, other metallic materials may be used, providing they possess the desired physical and chemical properties. The unctuousness of powdered aluminum is to a certain degree a function of its method of preparation, and as a result of the process of manufacture the powder is usually flaky and contains an appreciable proportion of lubricant. These properties may be duplicated to some extent with powders made from other oxidizable metallic materials, such as magnesium or the alloys of magnesium and aluminum. The addition of a small quantity of stearic acid to metallic powders will increase the spreading power of the material when used in connection with the process herein described.

While I have described certain preferred and specific methods of practicing the invention, it will be understood that the invention is not confined to the specific operations herein disclosed or to the particular manner of burning, and that various changes and modifications may be made within the scope of the following claims.

I claim:

1. The method of controlling the resistance of a silicon carbide resistor which comprises adding powdered aluminum to the mix from which the resistor is formed, and curing said resistor under conditions oxidizing to the aluminum.

2. The method of making a silicon carbide resistor which comprises incorporating powdered aluminum into a mix from which the resistor is to be formed, forming the resistor, and curing said resistor by heating in a sand carbon mix.

3. In the manufacture of silicon carbide resistors from a granular mix, the steps comprising incorporating finely divided metallic aluminum particles in the mix, forming the resistor, and converting the metallic particles in situ into compounds having a low electrical conductivity as compared with silicon carbide.

4. In the manufacture of silicon carbide resistors from particles of silicon carbide, the steps of increasing the resistance of the silicon carbide, which comprise initially applying adhering aluminum particles to some of the silicon carbide particles, and subsequently converting the adhering aluminum to an oxide which adheres to the surface of the silicon carbide particles.

5. The method of making a silicon carbide resistor which comprises forming the resistor from a mix of silicon carbide grains and powdered aluminum, embedding the formed resistor in a mixture containing sand and carbon, and curing the resistor by the passage of an electric current through the said resistor.

6. The method of making a series of silicon carbide resistors of different specific resist-

ances which comprises forming the resistors from a mix of silicon carbide grains and powdered aluminum, embedding the formed resistors in a mixture containing sand and carbon, curing the resistors by the passage of an electric current through the said resistors and varying the specific resistance of the finished resistors by varying the amount of aluminum added to the resistor mix.

- 10 7. A raw mix for the manufacture of silicon carbide heating elements comprising silicon carbide grains and aluminum powder.

8. The method of making a silicon carbide resistor which comprises forming the resistor from a mix of silicon carbide grains and an easily oxidizable metal, and bonding the silicon carbide grains into a resistor of higher specific electrical resistance than obtains when no oxidizable metal is added to the mix by embedding the resistor in a mixture containing sand and carbon and curing the resistor by passing an electric current through the resistor.

- 25 In testimony whereof I affix my signature.
ERNST HEDIGER.

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