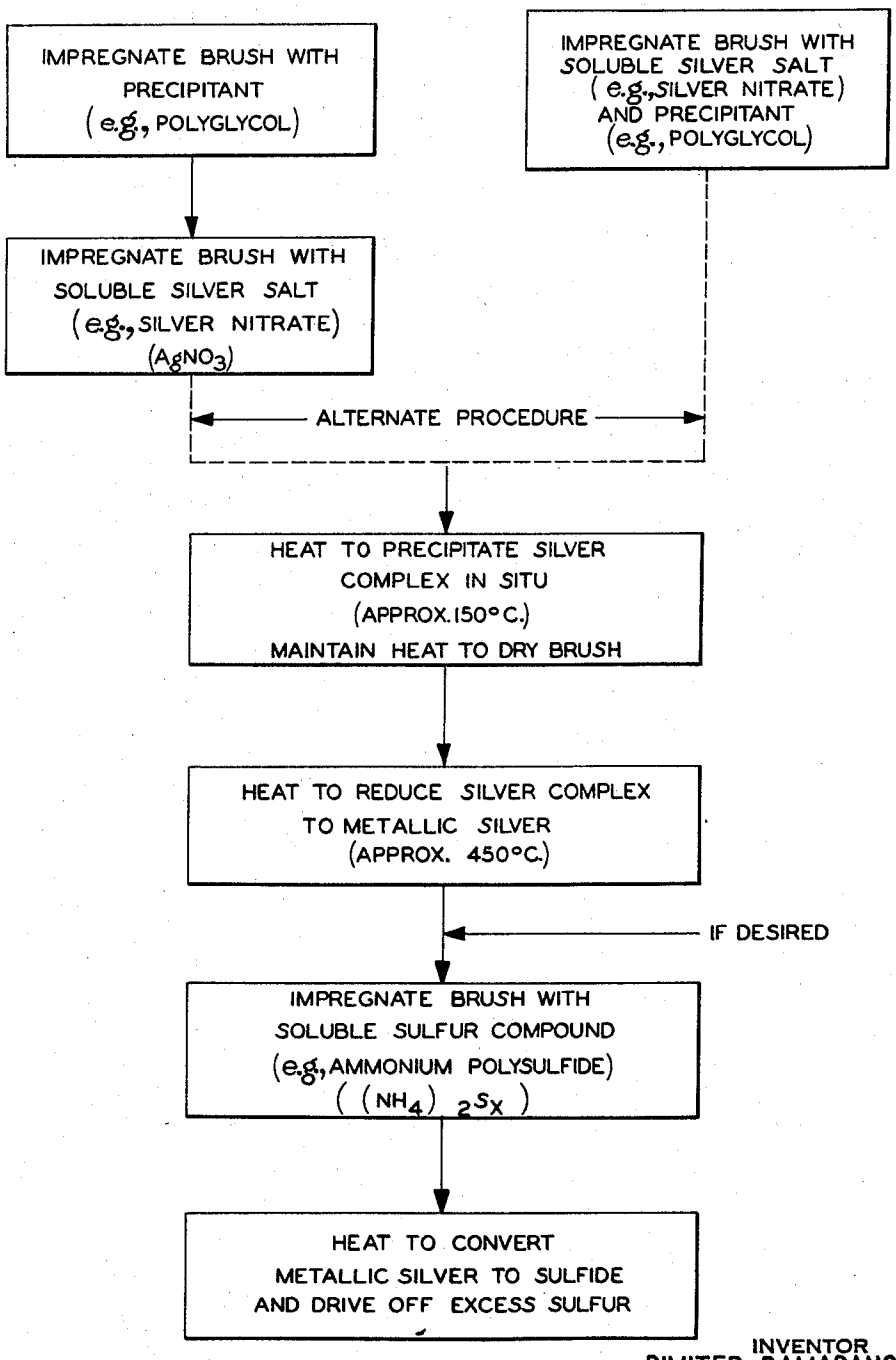
April 26, 1960 D. RAMADANOFF 2,934,460

METHOD FOR IMPREGNATING A CARBONACEOUS BRUSH WITH SILVER

Filed March 31, 1958



INVENTOR
DIMITER RAMADANOFF
BY John F Holzmann
ATTORNEY

United States Patent Office 2,934,460 Patented Apr. 26, 1960

2,934,460

METHOD FOR IMPREGNATING A CARBONACEOUS BRUSH WITH SILVER

Dimiter Ramadanoff, Berea, Ohio, assignor to Union Carbide Corporation, a corporation of New York

Application March 31, 1958, Serial No. 725,116

8 Claims. (Cl. 117—228)

This invention relates to a method of impregnating carbonaceous brushes, and, more particularly, concerns a method of impregnating carbonaceous brushes with silver and silver sulfide.

Impregnation of carbon and graphite brushes with silver or silver sulfide has been found to benefit the operation of the brushes when in current-carrying contact with a steel ring or commutator of a dynamo-electric apparatus. Specifically, impregnation with silver or silver sulfide generally increases the life of the brushes and beneficially alters the voltage drop across the contact between the brushes and the ring or commutator. Of equal importance, silver or silver sulfide impregnated within a brush has been found to reduce the operating temperature of the brush and to enhance its frictional properties.

Heretofore, it has been the practice to incorporate silver in a carbonaceous brush by impregnating the brush with a soluble compound of silver, such as silver nitrate, and decomposing the silver nitrate to silver in situ, usually, by baking the brushes. This practice has the disadvantage that water contained within the brush must be removed and that this is normally accomplished upon baking, in which process the impregnant migrates to the brush surface. Silver sulfide has been produced in situ by prior processes by first impregnating brush stock with silver nitrate and then converting the nitrate to the sulfide. Here, too, migration of impregnant may be encountered.

Migration of the impregnant silver or silver sulfide is undesirable because it renders the structure of a brush non-homogeneous, the silver or silver sulfide additive forming a shell so to speak at the surface of the brush. In operation, this is disadvantageous for a number of reasons. For one, migration may alter the current-carrying characteristics of a brush, and for another, the frictional properties of a brush may not be as good as otherwise possible if silver or silver sulfide were uniformly distributed throughout the brush.

A solution to the above problem of migration is set forth in my copending application, Serial No. 533,021, filed September 7, 1955, now U.S. Patent No. 2,847,332. As described in that application, the problem is solved by impregnating a carbonaceous brush with an aqueous solution of silver nitrate, precipitating the silver ions out of the silver nitrate as an insoluble silver compound while the brush is still wet to prevent migration of the silver nitrate, and then heating the carbonaceous brush to reduce the insoluble silver compound to metallic silver. The insoluble compound formed in the carbonaceous brush, in accordance with the principles of the invention, would have a low volatility, and should be capable on thermal decomposition of yielding reaction products which are not deleterious to the composition of the carbonaceous brush. To this end the carbonaceous brush may be impregnated, by the alternate application of vacuum and pressure, with an aqueous solution of silver nitrate.

While the carbonaceous brush is still wet, and before migration commences, it may be exposed to ammonia fumes to precipitate the silver ions in situ as silver oxide, or, preferably, the silver ions may be precipitated in stiu as silver carbonate by placing the carbonaceous brush, while still in a wet state, in an aqueous saturated solution of ammonium carbonate. The silver oxide or silver carbonate is then reduced to silver by heat treatment.

The disadvantage of using this method is that the precipitation stage is time consuming and is limited by the rate of diffusion of the precipitant. The precipitation time is approximately 15 hours.

One object of the invention is to provide a new and novel method for treating carbonaceous brushes with silver.

Another object is to provide a method whereby a soluble compound of silver is impregnated within a brush and rapidly precipitated in situ by the application of heat, thereby prohibiting migration and more uniformly distributing the silver within the brush.

A more specific object is to provide a method for impregnating a carbonaceous brush with silver or silver sulfide by impregnating the brush with a soluble silver compound and effecting rapid precipitation of the soluble silver compound in situ, removing contained water by evaporation without migration, and reducing the precipitated silver compound to metallic silver which then may be converted to silver sulfide if desired.

Still another object is to provide a method which further insures a more uniform distribution of silver or silver sulfide by first introducing the precipitant within the brush before impregnation with an aqueous solution of a soluble silver compound.

The single figure in the drawing is a flow diagram illustrating the method of the present invention, using the preferred impregnants.

According to the invention, it has been found that a carbonaceous brush may be impregnated with silver or silver sulfide with a uniform distribution by impregnating the brush with an aqueous solution of a soluble compound of silver, such as silver nitrate, and a suitable precipitant; precipitating the silver compound rapidly in situ and heating to a temperature sufficient to remove contained water in the brush, and subsequently firing the brush at a sufficient temperature to reduce the silver compound to metallic silver, thereby also evolving any organic matter present within the brush. Thereafter the thus-impregnated brush may be treated suitably to convert the metallic silver in situ to silver sulfide, a suitable method being by subjecting the brush to a solution of a soluble compound of sulfur such as ammonium polysulfide, drying at an elevated temperature, and firing the brush at a temperature sufficient to convert the impregnated metallic silver to silver sulfide.

In the practice of the invention, a suitable precipitant may be a solution of a compound or mixture of compounds soluble in water and in which silver nitrate when heated to elevated temperatures is substantially insoluble. Acetone, an alcohol such as methyl or ethyl alcohol, or ethylene diamine are good precipitants. The more preferred precipitants are the polyglycols. For example, the polyoxyethylenes or polyoxypropylenes are very good precipitants in practicing the method of the invention. The most desirable precipitant to be used is an equal mixture of polyethylene oxide and polypropylene oxide.

The percentage of silver nitrate and the precipitant to be used are generally not too critical, those skilled in the art being well versed in the amounts of silver nitrate solution to be used for desired impregnation of carbon with silver or silver sulfide. For example, it is known

that to impregnate an electrographitic brush with 8 percent to 12 percent by weight of silver or silver sulfide within the brush, a solution of about 30 percent to 40 percent by weight of silver nitrate in water should be used. To this solution, it has been found that about 10 percent by volume of the precipitant; say 50 percent ethylene oxide and 50 percent propylene oxide, is a suitable mixture. More or less precipitant can be used effectively. The amount of the sulfur-containing solution used to convert the impregnated silver in the brush to silver sulfide should be at least sufficient to combine with the amount of silver deposited. More solution used than required will provide an excess of sulfur which is driven-off by baking. The removal of excess sulfur is important since it can otherwise increase brush friction and contact voltage drop.

Illustratively, the method of the invention may best be explained by the following example: A natural graphite brush was impregnated, by alternate application of vacuum and pressure, with a solution of 33.3 percent of silver nitrate in water containing about 10 percent by volume of the polyglycol precipitant, 50 percent ethylene oxide and 50 percent propylene oxide. The brush was then heated to an elevated temperature of about 150° C. and the impregnated silver nitrate was precipitated almost immediately in situ without migration, and this temperature was maintained for one hour to dry the brush. Subsequently, the brush was fired at a temperaure of about 400° C. for four minutes and the silver nitrate was thereby reduced to metallic silver. Any organic material remaining from the precipitant was driven-off upon firing. In a second step, the brush was similarly impregnated with an ammonium polysulfide solution, dried at 150° C. for one hour and fired at a temperature of 450° C. to convert the metallic silver impregnated in the brush to silver sulfide, while also removing any free sulfur present.

In another example, it was found that the properties of the brush of the above example could be improved by altering the initial silver nitrate impregnation. In this example, a natural graphite brush was first impregnated with the polyglycol precipitant in an amount equivalent to 10 percent by volume of silver nitrate to be later impregnated. Thereafter, the brush was dried and then impregnated with a solution of 33.3 percent by volume of silver nitrate in water and the brush further treated as explained in the above example. Referring to the tables below, this brush in operation exhibited a lower contact voltage drop and a slightly improved wear life.

The following tables illustrate the improved properties achieved by impregnating a brush with silver and silver sulfide according to the method of the invention. In one series of tests, an unimpregnated natural graphite brush and the same brush impregnated with silver sulfide by prior art methods were compared with two brushes impregnated according to the invention, one impregnated with silver and another with silver sulfide. Table I summarizes the results of this test from which the benefits of the invention are readily apparent.

TABLE I

High speed tests on steel rings

[Current density 120 a.p.s.i.; rubbing speed 14,150 f.p.m.; spring pressure 3.0 p.s.i.]

Brush (Natural Graphite Grade)	Contact Drop (Volts)	Life (Hrs./In.)	Coefficient of Friction	Brush Temp. (° C.)
Unimpregnated	3.15	1,190	0.20	120
Impregnated (12.7% Ag_2S—prior methods)	2.35	3,990	0.21	134
Impregnated (7.7% Ag—method of the invention)	2.40	3,880	0.20	115
Impregnated (8.8% Ag_2S—method of the invention)	2.10	4,465	0.18	97

The figure of Table I show that a brush impregnated with silver sulfide by the method of the invention has a lower contact drop, an improved coefficient of friction, a much lower brush temperature, and a greatly improved life as compared to a brush impregnated with silver sulfide by prior methods. Moreover, a brush impregnated with silver by the method of the invention has a lower contact drop, a tremendously greater life, and a lower brush temperature than an unimpregnated brush and only a slightly higher contact drop and shorter life than a brush impregnated with silver sulfide by prior methods and does have a lower coefficient of friction and a much lower brush temperature than such brush.

In another test, a natural graphite brush was treated by incorporating silver powder within the brush and another brush treated by converting the powdered silver to silver sulfide. These brushes were compared in the test with two brushes impregnated with silver sulfide according to the invention, one of the brushes being prepared by first impregnating the brush with the polyglycol precipitant as in the above example. Table II illustrates the results of the test.

TABLE II

[Current density 120 a.p.s.i.; rubbing speed 14,150 f.p.m.; spring pressure 3.0 p.s.i.]

Brush (Natural Graphite Grade)	Contact Drop (Volts)	Life (Hrs./In.)	Coefficient of Friction	Brush Temp. (° C.)	Test Hours
Powders (10% Ag added in mix)	2.50	2,000	0.15	111	43.25
Powders (10% Ag changed partly to Ag_2S)	2.60	2,215	0.12	104	43.08
Impregnated (12.91% Ag_2S—method of the invention)	2.30	3,425	0.13	103	66.34
Impregnated (10.5% Ag_2S—prior impregnation of precipitant)	2.30	2,940	0.15	110	16.67
	1.98	3,535	0.25	120	65.75

Probably the most desirable advantage of the invention is the uniform distribution of silver or silver sulfide throughout the brush. Heretofore, by prior art methods, due to poor distribution of the silver additive, the brush was impregnated with a much greater quantity of silver nitrate than now required if it was desired to impregnate the whole brush with silver or silver sulfide. By the invention, it is now possible to achieve a more uniform distribution of silver and silver sulfide in a brush with the use of lesser quantities of the impregnant in a much shorter period of time.

A test was made to evaluate the effect of precipitating silver nitrate in situ within a brush before the impregnant was decomposed to silver as by prior art methods. In the test, two cylinders of an electro-graphitic brush grade were impregnated with a solution of 33.3 percent of silver nitrate in water, one of the cylinders being treated with the solution modified by the addition of 10 percent by volume of 50 percent ethylene oxide-50 percent propylene oxide. The cylinders measured 1½ inches long and 1 inch in diameter. Immediately after impregnation, the brushes were heated at about 150° C. for one hour to remove contained water. Thereafter, the cylinders were fired for five minutes at 450° C. to decompose silver nitrate to silver. The cylinders were weighed and the final pick-up of silver calculated. The ends of each cylinder were cut and the cylinders drilled with a hollow drill ½ inch in diameter, and the center cores of the cylinders removed. The outer shell and center core of each cylinder were separately ashed and the silver pick-up calculated. Table III summarizes the results of the test from which the beneficial effect of impregnation and precipitation of silver nitrate according to the invention is readily apparent.

TABLE III

Evaluation of uniform distribution

Designation	Total pick-up calculated (percent)	Pick-up from Outer shell	Ashing (percent) Center core	Uniformity (percent)
Impregnation of $AgNO_3$ Alone	3.72	4.00	1.65	41.2
Impregnation of $AgNO_3$ plus Precipitants (50% ethylene oxide-50% propylene oxide)	3.32	2.7	1.83	67.6

The above tables clearly show the success of the subject invention. When carbonaceous brushes are treated by prior art methods of impregnating the brush with a soluble compound of silver such as silver nitrate and decomposing the silver nitrate to silver by baking the brushes, the migration of the silver to the outer surface results in non-uniformity of the silver distribution. However, treatments according to the method of the invention show highly improved uniformity of silver distribution. Also, the present invention provides a method whereby the reduced silver in the brush may be readily converted to silver sulfide.

Also, the present invention provides a method whereby the silver may be uniformly distributed in the carbonaceous brush in a much shorter period of time than by the method of my copending application, Serial No. 533,021, filed September 7, 1955, now U.S. Patent No. 2,847,332. Brushes made by the method of that invention require a day of processing time or more whereas brushes made according to the principles of the subject invention may be impregnated, dried, and sulfided within an hour.

What is claimed is:

1. A method of introducing and uniformly distributing metallic silver in a carbonaceous brush, which method comprises impregnating the brush with an aqueous solution of silver nitrate and a precipitant selected from the group consisting of acetone, methyl alcohol, ethyl alcohol, ethylene diamine, polyglycols, and an equal mixture of ethylene oxide and propylene oxide, heating the brush to a temperature such that the silver nitrate is precipitated rapidly in situ and the contained water is evaporated, and subsequently further heating the brush to a sufficient temperature to reduce the silver nitrate to silver, while evolving any organic matter present in the brush.

2. The method of claim 1 wherein the impregnated silver is further converted to silver sulfide by impregnating the brush with ammonium polysulfide, heating said brush to an elevated temperature to dry said brush, and further elevating temperature of said brush to convert said impregnated metallic silver to silver sulfide, while also removing any free sulfur present.

3. A method of introducing and uniformly distributing metallic silver in a carbonaceous brush which method comprises impregnating the brush with a precipitant selected from the group consisting of acetone, methyl alcohol, ethyl alcohol, ethylene diamine, polyglycols, and an equal mixture of ethylene oxide and propylene oxide,

drying the brush, subsequently impregnating the brush with an aqueous solution of silver nitrate, heating the brush to rapidly precipitate the silver nitrate in situ without migration while contained water is evaporated, and subsequently further heating the brush to a sufficient temperature to reduce the silver nitrate to silver, while evolving any organic matter present in the brush.

4. The method of claim 3 wherein the impregnated silver is further converted to silver sulfide by impregnating the brush with ammonium polysulfide, raising said brush to an elevated temperature to dry said brush, and further elevating the temperature of said brush to complete the conversion of said impregnated metallic silver to silver sulfide, while also removing any free sulfur present.

5. A method of introducing and uniformly distributing metallic silver in a carbonaceous brush, which method comprises impregnating said brush with an aqueous solution of silver nitrate and a precipitant which is an equal mixture of ethylene oxide and propylene oxide, heating said brush to approximately 150° C. to rapidly precipitate said silver nitrate in situ and to evaporate the contained water, and subsequently further heating said brush to approximately 400° C. to reduce said silver nitrate to silver, while evolving any organic matter present.

6. A method of introducing and uniformly distributing metallic silver in a carbonaceous brush which method comprises impregnating said brush with a precipitant which is an equal mixture of ethylene oxide and propylene oxide and drying said brush, subsequently impregnating said brush with an aqueous solution of silver nitrate, heating said brush to approximately 150° C. to rapidly precipitate said silver nitrate in situ without migration while contained water is evaporated, and subsequently further heating said brush to approximately 400° C. to reduce said silver nitrate to silver, while evolving any organic matter present in said brush.

7. The method of claim 5 wherein the impregnated silver is further converted to silver sulfide by impregnating said brush with ammonium polysulfide, heating said brush to approximately 150° C. to dry said brush, and further heating said brush to approximately 450° C. to convert said impregnated metallic silver to silver sulfide, while also removing any free sulfur present.

8. The method of claim 7 wherein the impregnated silver is converted to silver sulfide by impregnating said brush with ammonium polysulfide, heating said brush to approximately 150° C. to dry said brush and further heating said brush to approximately 450° C. to complete the conversion of said impregnated metallic silver to silver sulfide, while also removing any free sulfur present.

References Cited in the file of this patent

UNITED STATES PATENTS

2,428,036	Peters et al.	Sept. 30, 1947
2,602,757	Kantrowitz et al.	July 3, 1952
2,748,034	Bobal	May 29, 1956
2,847,332	Ramadanoff	Aug. 12, 1958

FOREIGN PATENTS

777,511	Great Britain	June 26, 1957