

July 13, 1954

M. L. MARTIN

2,683,652

HEAT TREATMENT OF MICROPHONIC CARBON

Filed Dec. 28, 1951

6 Sheets-Sheet 1

FIG. 1A

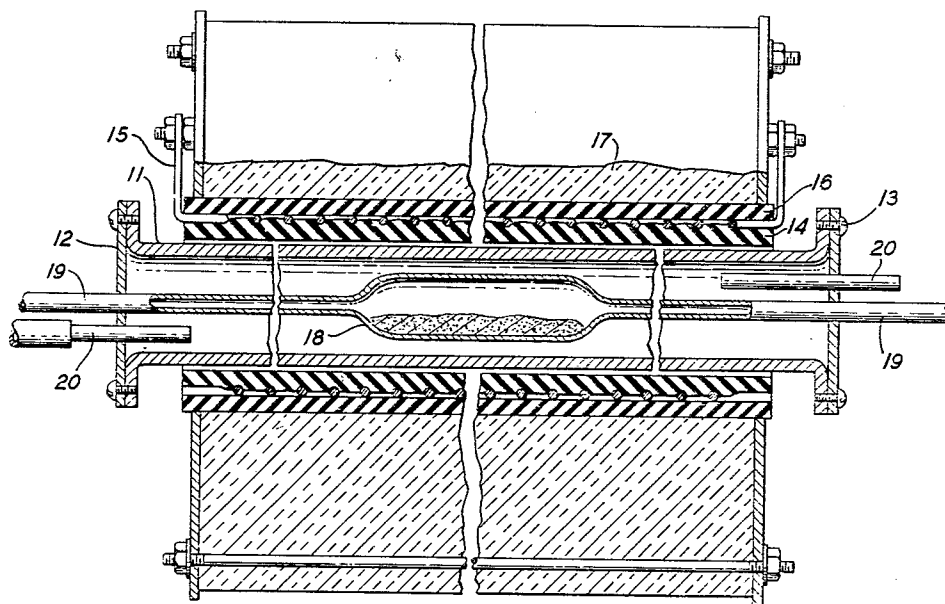
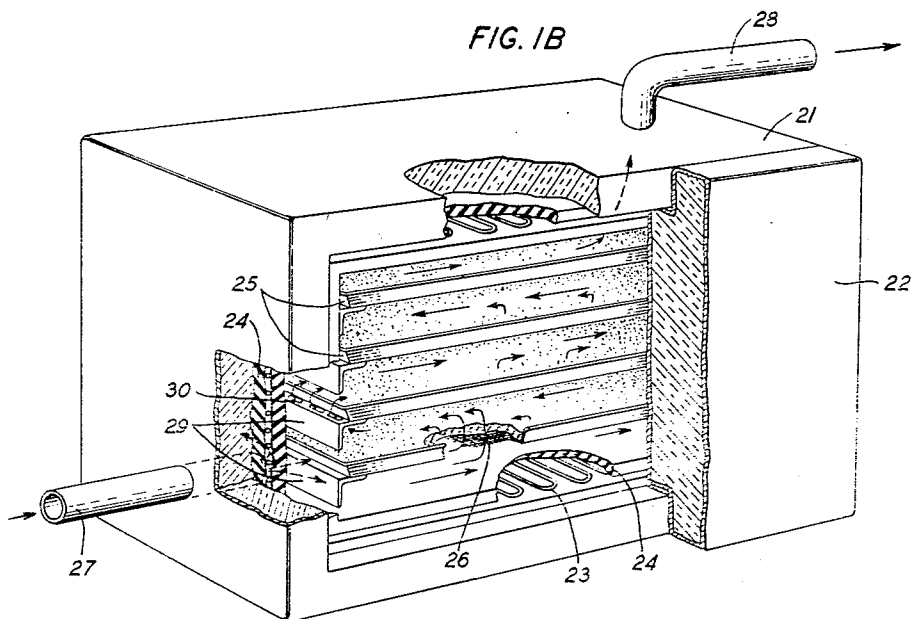


FIG. 1B



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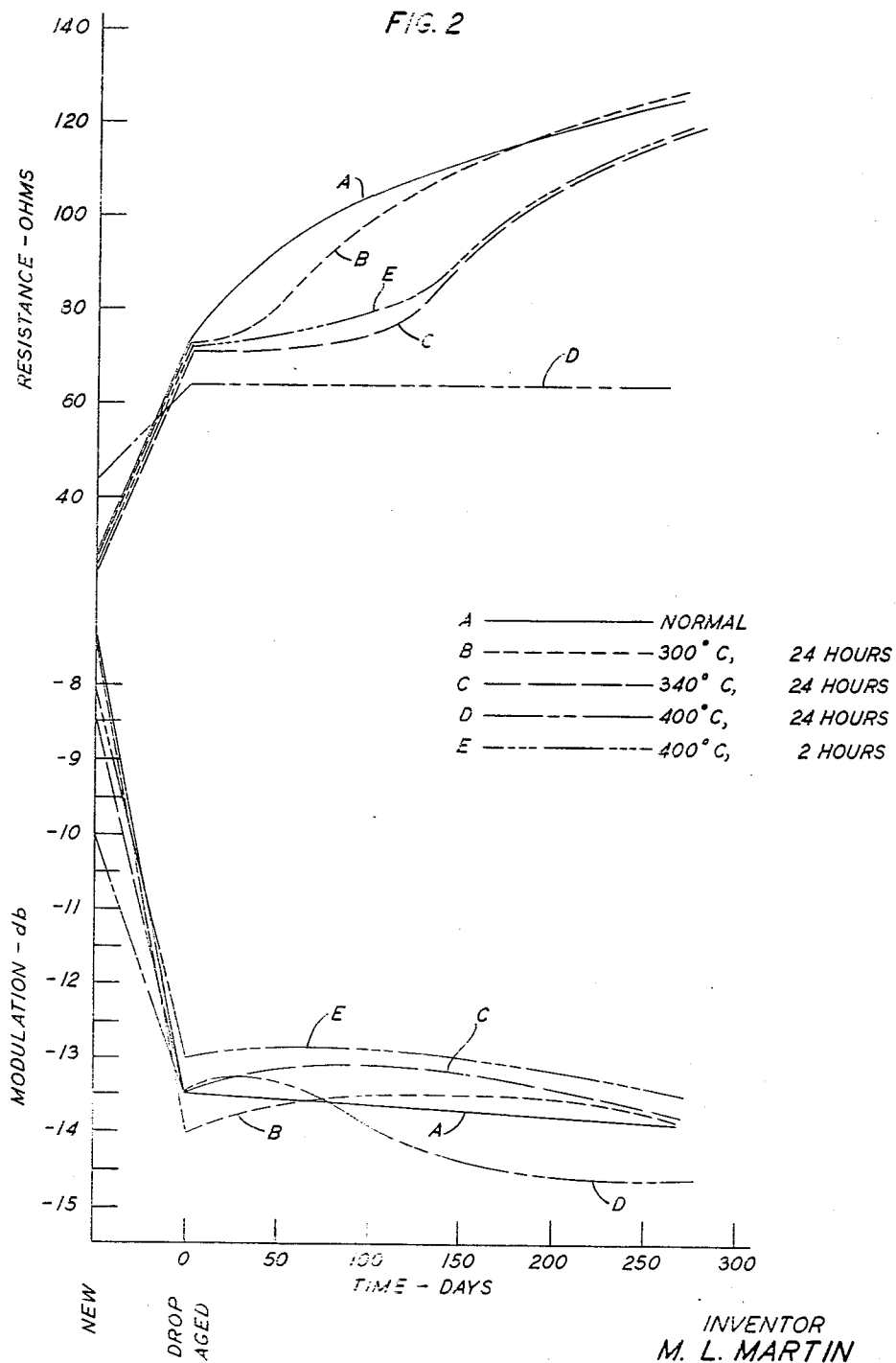
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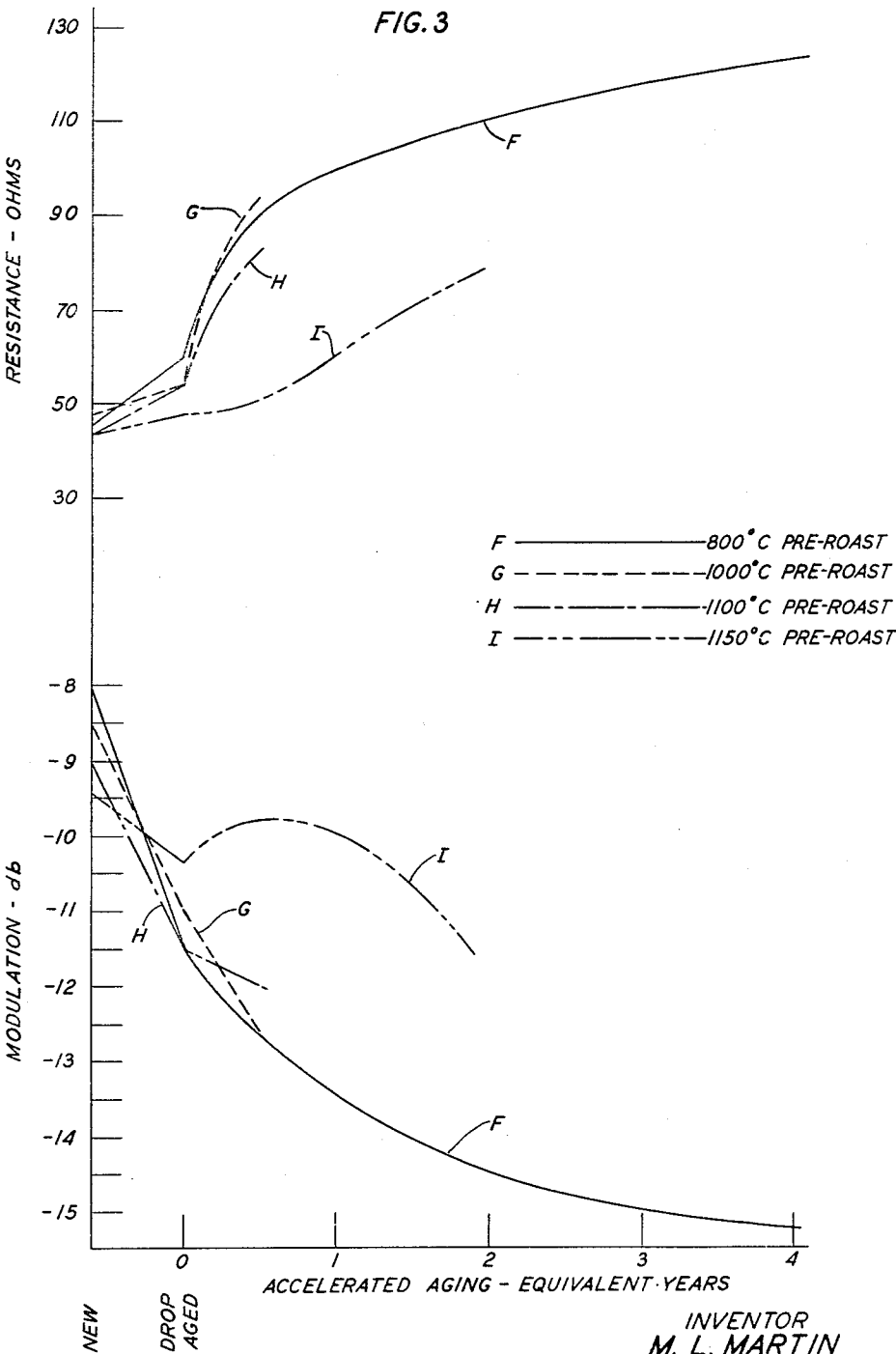
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HEAT TREATMENT OF MICROPHONIC CARBON

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6 Sheets-Sheet 3



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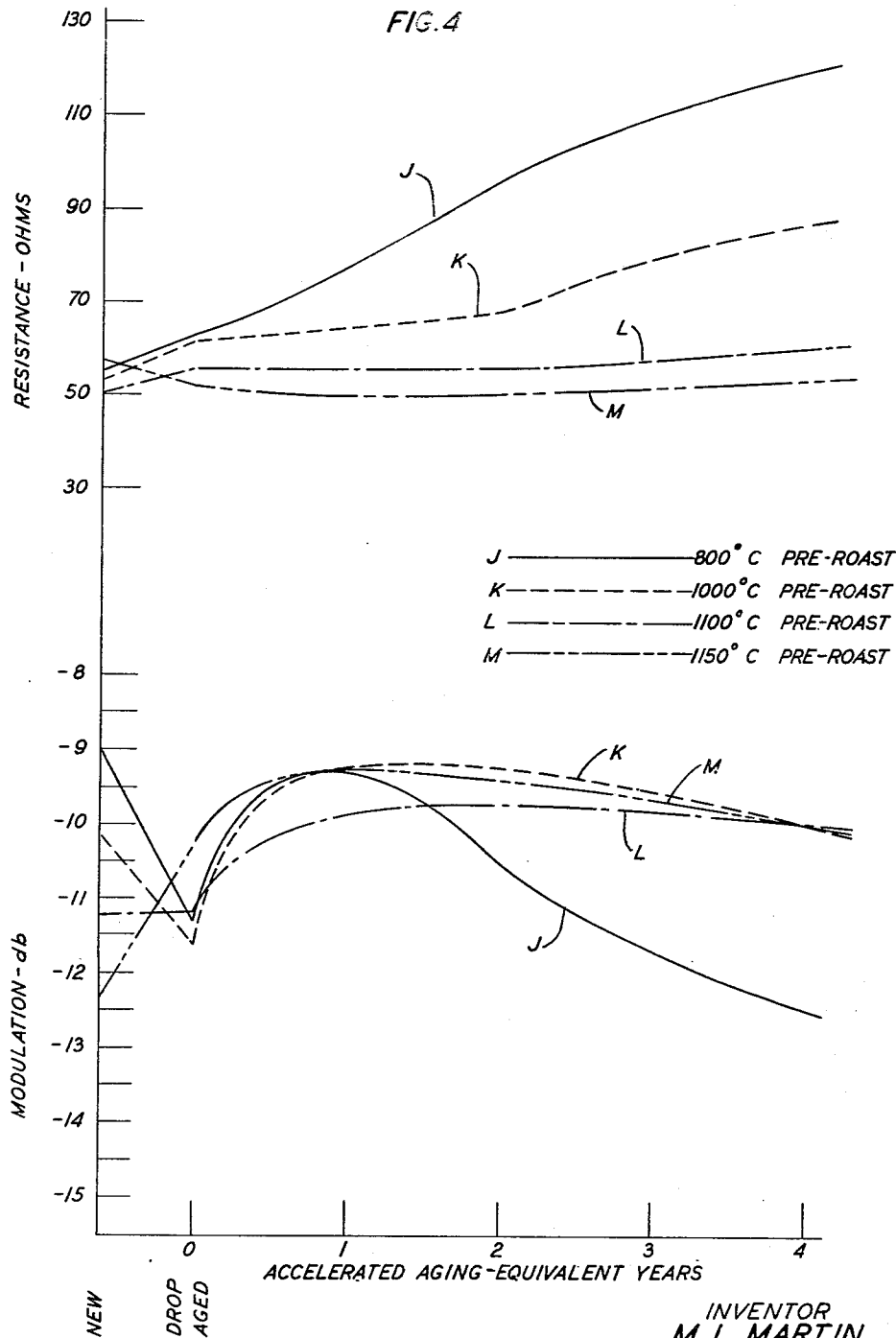
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HEAT TREATMENT OF MICROPHONIC CARBON

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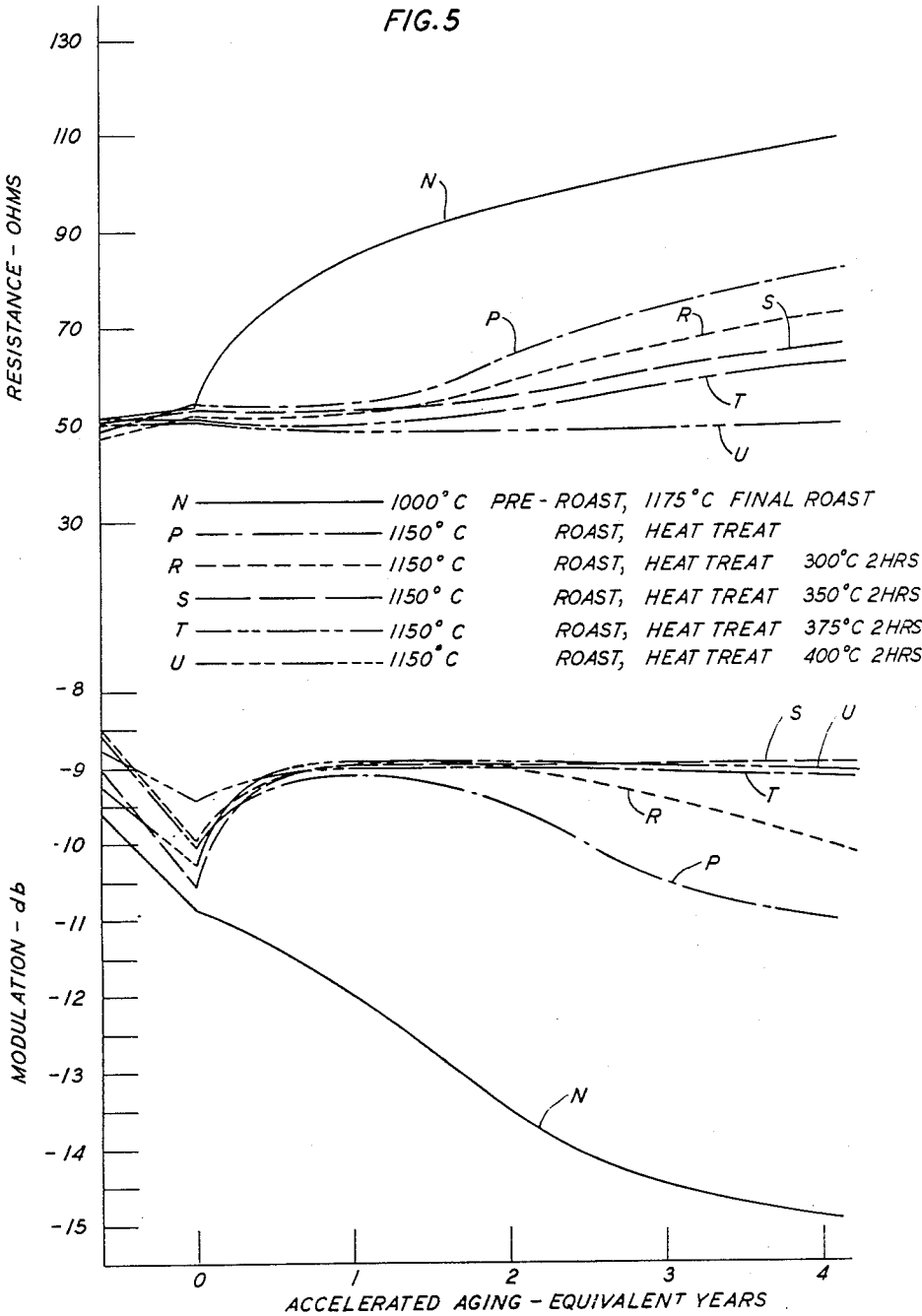
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HEAT TREATMENT OF MICROPHONIC CARBON

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6 Sheets-Sheet 5



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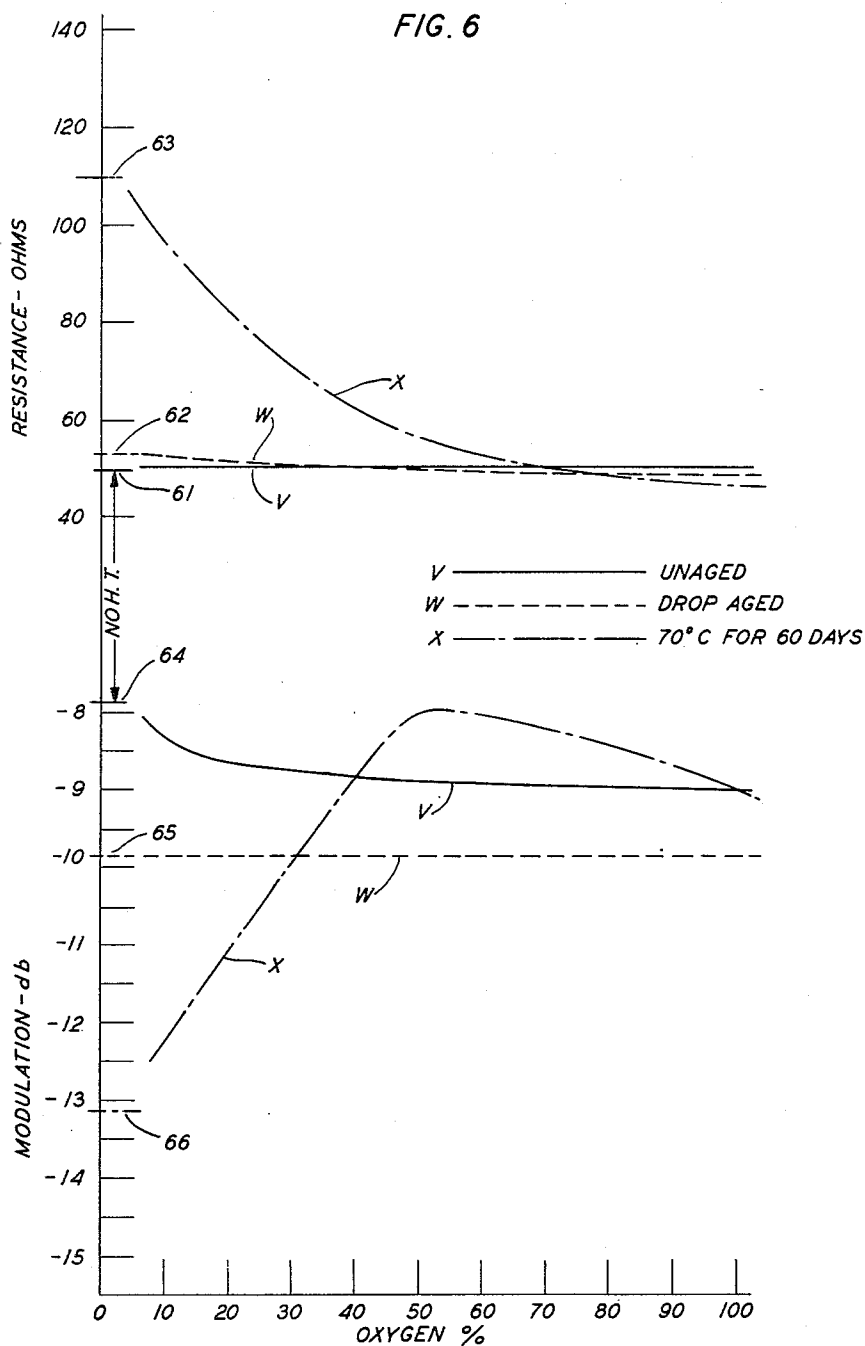
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HEAT TREATMENT OF MICROPHONIC CARBON

Filed Dec. 28, 1951

6 Sheets-Sheet 6



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UNITED STATES PATENT OFFICE

2,683,652

HEAT TREATMENT OF MICROPHONIC CARBON

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Application December 28, 1951, Serial No. 263,772

8 Claims. (Cl. 23—209.1)

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This invention relates to telephone transducers and more particularly to methods of producing microphonic carbon for use in transmitters of the general type disclosed in Patent 2,042,822 granted June 2, 1938, to A. F. Bennett and W. L. Tuffnell.

Microphonic carbon as employed in telephone transmitter units is subject to large relative changes in its resistance and modulating ability in the first few years of its life. These changes cause difficulty in the systems in which the transmitters are employed. Less than optimum transmission characteristics due to an impedance mismatch between the instrument employing microphonic carbon and the line to which it is connected must be accepted at some period in the operating life of the combination, usually during the initial operating period, because of the increase in carbon resistance. The resistance of these units increases with time thereby introducing signalling difficulties by attenuating the signal currents to such an extent that they may be insufficient to operate lamps, relays, or other apparatus of the system. The modulation decreases, reducing the quality of the signal. The noise generated in the carbon increases with time to further impair signal quality. Sensitivity to the position of the instrument has also been found to increase with age so that units which have been in use for some time and are mounted in certain positions are found to have relatively poor transmission characteristics.

The changes in microphonic carbon have been found to be principally of two forms; one which occurs with use is mechanical in nature and is referred to in the art as mechanical or drop aging, and the other which occurs with time is chemical or physical-chemical in nature and is referred to as shelf or time aging. Both of these forms of aging heretofore tended to increase the resistance of microphonic carbon while decreasing its modulating ability. It has been suggested that mechanical aging was due to changes in the surface characteristics of microphonic carbon granules, both the creation of an insulating layer and the general roughening of the carbon surface to increase the contact resistance between granules have been considered possible causes. Shelf aging, on the other hand, occurs with time and appears to be due to the evolution of a gas and other deleterious substances from the carbon granules. These substances, which at present are believed to include sulphur or compounds thereof, are believed not only to affect the carbon by chemical reactions therewith to create insulating films on the granules and to form deposited coat-

ings thereon but also to react with the materials of which the carbon container is composed.

It has been suggested that both of the above forms of change in the characteristics of microphonic carbon could be inhibited by employing roasting processes. These roasting processes are exemplified by Patent 1,646,389 issued October 25, 1927, to E. N. Bunting, Patent 1,722,055 issued July 23, 1929, to H. H. Lowry, and Patent 1,907,843 issued May 9, 1933, to H. H. Lowry. In these patents, processes are disclosed wherein the carbon is subjected to one or more roasts at temperatures ranging from a minimum of 500° C. to not over 1400° C. in atmospheres which are substantially non-oxidizing or reducing, either to convert the non-magnetic iron compounds present in raw coal from which microphonic carbon is obtained to magnetic compounds which can be separated from the mass, or to crack the volatile hydrocarbons present in the raw material and remove those products, other than carbon, of the cracking operation. Following the roast the carbon was cooled to less than 200° C. in the non-oxidizing atmosphere to prevent its burning when exposed to the air. While these processes produced a carbon which changed less during its useful life than had been previously obtainable, there were still substantial changes which occurred in the material. Further, it was found that while higher roasting temperatures improved the stability of the carbon it also materially reduced its modulation characteristic; hence, a balance between stability and modulation had to be accepted at some temperature below the optimum for stability.

More recently the problem of mechanical aging has been overcome to a substantial extent by a process involving the mechanical working of the microphonic carbon after it has been roasted. This process of mechanical working is disclosed in C. E. Mitchell application Serial No. 123,065 filed October 22, 1949, now Patent 2,606,817 which issued August 12, 1952. There it is shown that when microphonic carbon granules are vibrated in an enclosed space, usually containing a non-oxidizing atmosphere, until the resistance of the material increases to a predetermined value only slight further changes in resistance of the carbon, due to mechanical working occur over its useful life. Although this process greatly inhibits variations in the carbon resistance and a decay in modulation characteristics attributable to mechanical aging it appears to have little effect upon the shelf or time aging of the material and therefore further treatment is desirable.

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One object of this invention is to improve microphonic carbon. More particularly an object of this invention is to reduce the change in carbon characteristics which occurs over its useful life and is attributable to shelf or time aging. A further object is to inhibit shelf or time aging in carbon while maintaining a good modulation characteristic. Another object is to inhibit the effects of moisture on microphonic carbon. Another object is to reduce the noise in aged transmitter units containing microphonic carbon.

A feature of this invention resides in reducing the changes in the characteristics of microphonic carbon which heretofore have occurred with the passage of time by heating the carbon in an oxidizing atmosphere to a temperature below that at which it oxidizes to any substantial extent.

Another feature resides in roasting properly prepared coal granules, then subjecting them to a heat treatment in an oxidizing atmosphere and finally mechanically agitating the resultant microphonic carbon thereby substantially inhibiting changes in its electrical characteristics with both time and use.

In one embodiment of this invention, high grade anthracite coal is crushed, ground, sized, washed, and dried. Then it is roasted at temperatures of the order of 1000 to 1400° C. in a hydrogen or other non-oxidizing atmosphere, cooled in a non-oxidizing atmosphere to below that temperature at which the carbon oxidizes to a prohibitive degree and heat treated at from about 250 to 500° C. in an oxidizing atmosphere. It has been theorized that the heat treatment of the carbon at high temperatures effects a thermal decomposition of some deleterious substance, perhaps a sulphur compound. This high temperature treatment or roast greatly reduces the shelf aging of carbon by removing a substantial portion of the substances which cause it to change; however, it is believed that a substantially impermeable shell, probably of pyrolytic carbon, is formed before the deleterious substance mentioned above diffuses out of the granules. Thus, the final product if not further treated contains a substance that gradually diffuses to the surface of the granules to react with that surface and those of the surroundings to the detriment of the granule's electrical characteristics.

Heat treatment of the roasted carbon in an oxidizing atmosphere at temperatures above 250° C. is believed to disrupt the surface shell of the carbon granules to permit the diffusion of the oxidizing constituents of the atmosphere into the granules and of the oxidation products out of them to eliminate the deleterious substance. It is to be noted that the heat treatment to be most effective is carried out after a high temperature roast and that its maximum temperature should be about 500° C. The latter limitation has been set since a satisfactory reaction can be accomplished below this temperature, and carbon begins to oxidize rapidly above about 500° C. The maximum temperature depends upon the amount of oxygen present. The carbon oxidizes too rapidly in air above 450° C. and temperatures higher than this are suitable only when the oxygen content of the atmosphere is below that of air. The first-mentioned limitation may be attributable to the following: since sulphur is known to be emitted from granular carbon after it has been roasted, some sulphur compound which decomposes at high temperatures may be involved in shelf aging. Compounds of sulphur existing in coal, except certain carbon-sulphur

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complexes, apparently decompose at temperatures considerably lower than 800° C. Pyrite and marcasite (FeS_2) decomposition is completed at 600° C., the products being ferrous sulphide and hydrogen sulphide. (When the coal is heated in an atmosphere containing hydrogen.) Ferrous sulphide is stable up to about 1100° C., at which temperature it begins to decompose into the elements. If the sulphur emitted from granular carbon has its source in the above decompositions, then heat treatment in an oxidizing atmosphere would be expected to be effective in removing this sulphur only after the roast had conditioned the sulphur for removal.

Specific treatments and ranges of parameters for the inhibiting of and for reducing the magnitude of changes in the electrical characteristics of microphonic carbon with time are disclosed in the following detailed description from which the invention and its various objects and features will be more readily understood when read with reference to the accompanying drawings in which:

Fig. 1A is a partially broken, sectioned elevation of a furnace suitable for batch roasting of microphonic carbon;

Fig. 1B is a perspective of an oven of the type which can be employed in heat treating the carbon;

Figs. 2, 3, 4, and 5 are plots of resistance and modulation against time for transmitter units of the type disclosed in Patent 2,042,822 granted June 2, 1936, to A. F. Bennett and W. L. Tuffnell employing carbons which have been subjected to various roasts, heat treatments, and combinations thereof; and

Fig. 6 is a curve showing the resistance and modulation of a transmitter unit of the construction shown in Patent 2,042,822 containing heat treated carbon, plotted against the percentage of oxygen in the carbon heat treating atmosphere.

In the following description the term "roast" refers to a high temperature treatment of carbon carried out under substantially non-oxidizing conditions. The purpose of roasting is to vary the resistivity of the carbon and partially stabilize its characteristics with both age and mechanical agitation. Two methods of roasting will be discussed, "batch roasting" and "continuous roasting." Batch roasting is effected by slowly raising the temperature of carbon from room temperature to roasting temperature in a furnace which has a controlled atmosphere, maintaining the carbon at the roasting temperature for a substantial period and then cooling it in this atmosphere. Continuous roasting involves feeding the carbon into a furnace through which it travels while controlling the atmosphere of the furnace. In the continuous roasting process discussed here, the carbon is maintained at temperature for about 40 minutes out of a total traveling time through the oven of approximately two hours. The term "double roast" includes a process in which the carbon is subjected to a preliminary roast, usually a batch roast, and a final roast, usually a continuous roast. "Heat treatment" is a process including the heating of carbon to moderate temperatures in an oxidizing atmosphere. "Mechanically stabilized carbon" is that which has been subjected to the mechanical agitation treatment disclosed in the above-noted application of C. E. Mitchell whereby variations in resistance with agitation in use are inhibited or substantially eliminated. "Drop aging" is a treatment simulating actual use of a telephone transmitter unit

and is effected by dropping a handset containing the unit a distance of about $1\frac{1}{2}$ inches to its cradle 22,000 times. This corresponds to four years of average telephone use and results reported following this treatment are substantially free of any further changes due to mechanical agitation.

Broadly, this invention comprises the reduction of shelf or time aging of roasted microphonic carbon by heat treating the carbon at temperatures between 250 and 500° C. in an oxidizing atmosphere. Since it appears that up to a point the amount of time stabilization of the carbon which occurs in this heat treatment depends on the amount of oxidation of the process, three interrelated factors must be considered and correlated in any particular sequence of heat treating steps, namely, the amount of oxidizing constituent present, the temperature at which the treatment is practiced, and the period over which the oxidation can occur. At present it appears particularly advantageous to heat treat the carbon in air for periods of from one to ten hours at temperatures of from 350 to 450° C.

In practicing the various roasts and heat treatments discussed below standard forms of furnaces and ovens arranged for controlled atmospheres have been employed. A furnace suitable for roasting is disclosed in Fig. 1A, and an oven in which the carbon can be heat treated is shown in Fig. 1B. The furnace comprises a central tube 11 of suitable inert material such as hard porcelain or nickel fitted with gas inlet and outlet caps 12 and 13 and gas ducts 20. A tube 14 of Alundum or other refractory insulating material is wound with a resistance wire 15 to provide a heating unit which surrounds the tube 11. The heating unit fits into a tube 16 of refractory material over which is applied a coating 17 of insulating material such as magnesia, asbestos, or magnesia-asbestos. The granular coal is contained in a closed crucible 18 of some material which is inert at high temperatures, for example the nickel, iron, chromium alloy known as Fire Armour. Gas inlet and exhaust ducts 19 are provided integral with the crucible so that a controlled atmosphere can be maintained around the roasting coal.

The heat treating oven shown in Fig. 1B comprises an outer housing 21 of suitable construction to provide heat insulation. Access is gained to the interior of housing 21 through door 22. The oven is heated by electrical resistances 23 embedded in its walls between layers of refractory material 24. The carbon is heat treated on trays 25 having screen bottoms 26 of some material such as Monel metal to permit oxygen to contact a greater free surface of the aggregate and diffuse through it. In the heat treatments described here, Monel metal screens were employed and the carbon was spread on the screens to a depth of about $\frac{3}{8}$ inch. These heat runs were made with only a few ounces of carbon; hence the thermal inertia of the aggregate is small and heat treatment could be effected by heating the oven to temperature, opening it and placing the screens filled with carbon in it. With small quantities of carbon and the oven employed the carbon reached temperature in about 15 minutes. Hence, in the two hour heat treatments discussed below the carbon was at temperature for about one hour and 45 minutes. During the heat treating process the atmosphere of the oven was changed at rates up to 5000 cubic centimeters per minute for each pound of carbon being treated. However, these rates are not believed to be critical it being necessary

only that the oxidizing constituent remain at about the desired concentration throughout the oven. Gas is admitted to the bottom of the oven through duct 27 and is exhausted through duct 28 flowing through the oven along the paths indicated by the arrows. In order to insure free passage of gas the tray supports 29 at the ends of the oven are provided with apertures 30 which permit the gas to flow to the next higher level at alternate ends of the oven.

A suitable continuous roast oven is disclosed in Bunting Patent 1,646,389 and may comprise a cylinder rotatably mounted with its axis on an angle to the horizontal in an insulated heating element similar to that described for the batch roast oven so that carbon may be fed into and caused to pass continuously through the tube during the roast. This oven is also provided with means for controlling the atmosphere of the roast and has suitable seals, an inlet, and an outlet.

In the following examples, all batch roasts were carried out in commercially pure hydrogen flowing through the roasting chamber at rates up to one cubic foot per hour per pound of carbon. The carbon was brought up to temperature in about 14 hours, held there for three hours, and then cooled at the cooling rate of the oven or at an accelerated cooling rate produced by passing air into tube 11 through ducts 20.

The continuous roasts have also been carried out in hydrogen flowing at rates up to six cubic feet per hour, although those shown are at one cubic foot per hour.

In Fig. 2 the resistance and modulation characteristics of ordinary microphonic carbon over a period of 260 days after drop aging is shown. Modulation as used here and in the remaining figures is plotted as decibels down from 100 per cent modulation. This carbon was double roasted, the preliminary batch roast being at 800° C. with one-tenth of a cubic foot of hydrogen per hour per pound of carbon and a final continuous roast at 1150° C. with one cubic foot of hydrogen per hour flowing. Curve A illustrates the characteristics of this carbon when subjected to no further treatment, its initial and drop aged values of modulation being shown, while curves B, C, and D illustrate the characteristics of this carbon after it has been subjected to heat treatments in air for 24 hours at 300, 340 and 400° C., respectively. It will be noted particularly at the higher temperatures that the increase in resistance with time has been lessened by the heat treatment. The delay mechanism present in the resistance curves B and C indicate that the depth of penetration of the treatment increases as the temperature increases, thereby removing the deleterious substance to an increasing depth and delaying time aging until the remaining substance has had time to diffuse to the surface of the granules. Curve E shows the effects of a heat treatment for two hours at 400° C., it being noted that this treatment is approximately equivalent to 24 hours at 340° C., curve C. This equivalence illustrates that the effect of a long time treatment at relatively low temperature can be substantially duplicated by a shorter treatment at a higher temperature and tends to indicate that it is the amount of oxidation of the deleterious substance present which determined the decrease in shelf aging effected since it is to be expected that this oxidation at the lower temperatures is about equal to the amount of oxidation which occurs in the shorter period at the higher temperature. As

indicated in this and the remaining figures the modulation of microphonic carbon varies with the roasts and heat treatments and therefore is an important factor to be considered in choosing the optimum heat treatment for most applications. For example, while improvement in characteristics is noted for heat treatments above 300° C. at both 2 and 24 hours the best heat treatment disclosed here in preparing this carbon for telephone transmitters from the resistance against time standpoint is 400° C. for 24 hours and from the modulation against time standpoint is 400° C. for two hours.

Having established by Fig. 2 that the heat treatment of this invention improves the characteristics of microphonic carbon as prepared according to processes of the prior art let us now consider modifications of these processes to obtain further improvements. Fig. 3 is a plot of a series of modifications of what was heretofore generally accepted as a standard double roast of microphonic carbon.

All of the carbon represented in Fig. 3 was double roasted. The preliminary batch roasts were at various temperatures in hydrogen flowing at the rate of 0.1 cubic foot per hour per pound of carbon. The final continuous roast for all batches was at 1150° C. with one cubic foot of hydrogen flowing per hour and all were mechanically stabilized in nitrogen and drop aged. This carbon was placed in transmitter units and subjected to an accelerated shelf aging at 70° C. for 60 days which is equivalent to four years at room temperature. Curves F, G, H, and I represent the characteristics of carbon subjected to a preliminary roast of 800, 1000, 1100 and 1150° C., respectively. The general trend shown by these curves indicates that increasing the pre-roast temperature reduces time aging of the carbon, and in the case of the 1150° C. preroast markedly improves the modulation characteristic.

The effect of heat treatment in air at 425° C. for two hours on the characteristics of the double roasted carbons shown in Fig. 3 is disclosed in Fig. 4. The curves labeled J, K, L, and M are for carbons preroasted at 800, 1000, 1100, and 1150° C., respectively, and are for mechanically stabilized carbon from the same batches as reported on in Fig. 3 as curves F, G, H, and I, respectively. At the higher preroasting temperatures the heat treatment effected a material reduction in aging.

Although a flat resistance against time characteristic results from the higher temperature preroasts of Figs. 3 and 4 it will be noted that the modulation characteristics for these roasts are initially relatively low. Modulation both initially and with aging can be improved while maintaining the desired substantially constant resistance with time by employing a single batch roast at 1150° C. with 0.5 cubic foot per hour of hydrogen per pound of carbon flowing through the roasting chamber particularly when this roast is coupled with a heat treatment in air.

Fig. 5 shows plots of resistance and modulation against time for transmitter units of the type employed in obtaining the preceding results filled with mechanically stabilized carbon which has been heated in various ways. The carbon of curve N was double roasted at 1000 and 1175° C. and exhibited slightly improved shelf aging over that roasted at 800 and 1150° C. Curve P is for carbon single batch roasted at 1150° C. in hydrogen flowing at 0.5 cubic foot per hour per pound of carbon. A further improvement in both re-

sistance and modulation over that shown in curve P is disclosed in curves R, S, T, and U for the carbon of curve P which has been heat treated in air for two hours at 300, 350, 375, and 400° C. The unaged modulation of the single roasted heat treated carbons is better than that of the double roasted carbon shown, indicating that these single roasts, even when heat treated, are better in modulation than the double roast carbon having relatively low shelf aging characteristics.

The description of the heat treating process has thus far been restricted to treatments in air. While air appears desirable from the standpoint of simplicity, it is to be understood that oxidizing atmospheres other than air are effective for heat treating. It has been found, as disclosed in Fig. 6, that gaseous compositions including from about two per cent to 100 per cent oxygen mixed with nitrogen are effective in improving the stability of microphonic carbon with time. The characteristics of resistance and modulation against per cent oxygen in the heat treating atmosphere shown in Fig. 6 are for single batch roast carbon which has been roasted at 1165° C. with 0.5 cubic foot of hydrogen per hour per pound of carbon, then has been heat treated in oxygen-nitrogen mixtures at 420° C. for two hours, mechanically stabilized to a uniform resistance and filled in transmitter units. Curve V is for units prepared as above which are unaged. Curve W shows the effect of drop aging on the units of curve V and again indicates uniform values of resistance and modulation over the range. The effect of oxidation by heat treatment is evident in curve X showing the resistance and modulation of the carbon after an accelerated shelf aging of 60 days at 70° C., equivalent to about four years of shelf aging at room temperature. It will be noted that from about 50 per cent oxygen to 100 per cent oxygen at this temperature and period of heat treatment the effects of shelf aging have been substantially eliminated. Further, the heat treatment in any atmosphere from slightly less than five per cent to 100 per cent oxygen effects an improvement as compared to similar units containing the same type carbon which has not been heat treated. The unaged, drop aged, and accelerated aged values of resistance and modulation for non-heat treated carbon are shown as points 61, 62, 63, 64, 65, and 66, respectively, on the ordinate of Fig. 6.

Since it appears that the reduction of shelf aging achieved by heat treating depends upon the amount of deleterious substance oxidized, it should be possible to duplicate any result reported in Fig. 6 with different oxygen concentrations. At lower oxygen concentrations the results could be duplicated by increasing the time the carbon is held at the heat treating temperature, by increasing the heat treating temperature or by increasing both parameters. As an example of the correlation obtainable between temperature and oxygen concentration where time of heat treatment was maintained constant, carbon was single batch roasted and heat treated in 100 per cent oxygen at 400° C. for two hours and mechanically stabilized to give a product which had a drop aged resistance of 62 ohms and a shelf aged resistance of 83 ohms. The same values and change of resistance were observed in single batch roasted carbon which was heat treated in air (about 20 per cent oxygen) for two hours at 430° C. and mechanically stabilized.

While particular roast and heat treatment at-

mospheres, times, and temperatures have been discussed above, it is to be understood that the improvements in microphonic carbon disclosed occur with combinations other than those suggested. The temperatures, oxidizing atmospheres, and lengths of treatment are so inter-related that they may be correlated in many instances to produce substantially identical results with widely differing heat treatment parameters. Therefore, it is to be understood that the above-described arrangements are illustrative of the application of the principles of the invention. Numerous other arrangements may be devised by those skilled in the art without departing from the spirit and scope of the invention.

What is claimed is:

1. The method of treating granular anthracite to adapt it for microphonic transmission which comprises roasting anthracite in a non-oxidizing atmosphere at from about 800 to 1400° C., cooling the roasted anthracite in the non-oxidizing atmosphere to below about 500° C., and maintaining the roasted anthracite at from about 250 to 500° C. in an oxidizing atmosphere for at least an hour.

2. The method of treating granular anthracite to adapt it for microphonic transmission which comprises roasting the anthracite in a non-oxidizing atmosphere at about 1150° C., cooling the roasted anthracite in the non-oxidizing atmosphere to below 500° C., and maintaining said roasted anthracite at about 250 to 500° C. in an oxidizing atmosphere for at least an hour.

3. The method of treating granular anthracite to adapt it for microphonic transmission which comprises single batch roasting the granular anthracite at about 1150° C. in a hydrogen atmosphere which is replenished at a rate of about 0.5 cubic foot of hydrogen per hour per pound of anthracite, cooling the roasted anthracite in said atmosphere to below 500° C., and maintaining the roasted anthracite at from about 250 to 500° C. in an oxidizing atmosphere for at least an hour.

4. The method of treating granular anthracite to adapt it for microphonic transmission which comprises roasting the granular anthracite in a non-oxidizing atmosphere at from about 800 to 1400° C., cooling the roasted anthracite to below 450° C. in the non-oxidizing atmosphere, and maintaining the roasted anthracite at about 250 to 460° C. in air for at least an hour.

5. The method of treating granular anthracite to adapt it for microphonic transmission which

comprises roasting the granular anthracite in a non-oxidizing atmosphere at from about 800 to 1400° C., cooling the roasted anthracite to below 450° C. in the non-oxidizing atmosphere, and maintaining said roasted anthracite at from about 350 to 450° C. in air for from one to ten hours.

6. The method of treating granular anthracite to adapt it for microphonic transmission which comprises roasting the granular anthracite in a non-oxidizing atmosphere at from about 800 to 1400° C., cooling the roasted anthracite to below 500° C. in the non-oxidizing atmosphere, and maintaining said roasted anthracite at from about 250 to 500° C. in an atmosphere containing from 5 to 100 per cent oxygen and the remainder an inert gas for at least an hour.

7. The method of treating granular anthracite to adapt it for microphonic transmission which comprises placing said granular anthracite in a non-oxidizing atmosphere, slowly raising the temperature of said anthracite to from about 1100 to 1200° C., maintaining the anthracite at from about 1100 to 1200° C. in the non-oxidizing atmosphere for about three hours, cooling the roasted anthracite in the non-oxidizing atmosphere to below 450° C., and maintaining the roasted anthracite in air at from about 350 to 450° C. for from one to ten hours.

8. The method of treating granular anthracite to adapt it for microphonic transmission which comprises batch roasting the granular anthracite in a substantially non-oxidizing atmosphere at a temperature of from about 800 to 1150° C., continuously roasting the anthracite at from 1000 to 1400° C. in a substantially non-oxidizing atmosphere, cooling the roasted anthracite in a non-oxidizing atmosphere to below 500° C., and maintaining the roasted anthracite at from about 250 to 500° C. in an oxidizing atmosphere for at least an hour.

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