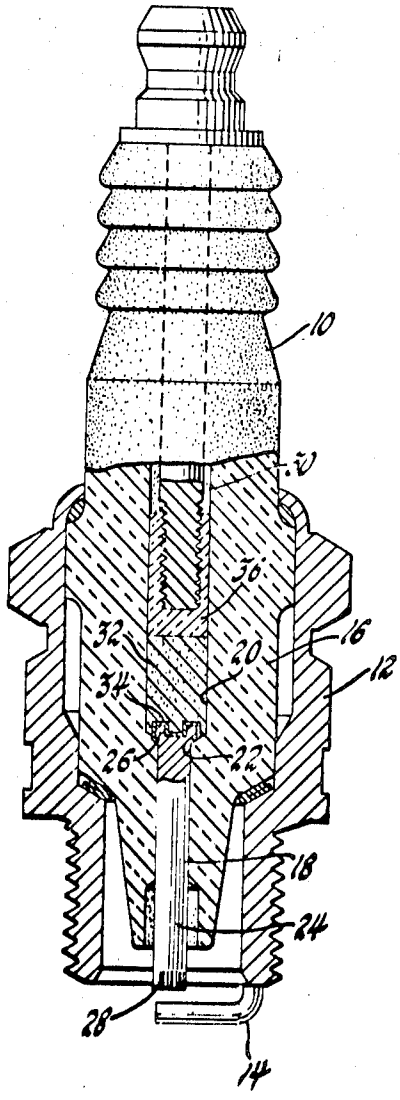
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RESISTOR COMPOSITION

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RESISTOR COMPOSITION

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ABSTRACT OF THE DISCLOSURE

An improved resistor composition for use in resistor spark plugs and other electrical devices is disclosed. The resistor composition contains a compound taken from the group consisting of lithium carbonate, zinc carbonate, magnesium carbonate and sodium carbonate and a metal powder taken from the group consisting of zinc, antimony and tellurium in addition to the conventional resistor composition ingredients. The carbonate, for example, lithium carbonate, and the metal, for example, zinc, combine to yield a strong resistor mass having a low porosity which bonds tightly to the center electrode head in the spark plug insulator centerbore.

This invention relates to resistors, and more particularly to glass phase semiconductor resistor compositions suitable for use in resistors and resistor spark plugs of the automotive or aviation type.

Resistors are commonly used to suppress the high frequency oscillations present with spark discharge in an ignition system, the oscillations resulting in rapid erosion of the spark plug electrodes and in interference with electronic equipment. Resistor spark plugs are effective in reducing automobile radio frequency interference and are particularly useful for FM broadcast and shortwave broadcast automobile receivers.

There are two basic types of resistor compositions. One type of monolithic resistor spark plug described in the McDougal et al. Pat. No. 2,459,282 granted Jan. 18, 1949, comprises a heterogeneous mixture of conductor material, that is, carbon either alone or in combination with various conducting metals, metal oxides and metal carbides with glass. In this type of resistor the conducting material, such as carbon, exists as a continuous phase and the resistance of the resistor is dependent solely upon the amount of conductor material that is present. The glass serves only to suspend the conductor material in a rigid structure. An example of this type resistor composition consists essentially of 64 weight percent borosilicate glass, 9 weight percent magnesium borate glass, 11 weight percent Fluorspar, 15% beryl and 1% Thermax carbon.

The second basic type of a monolithic resistor spark plug is described in the patent to Counts et al. No. 2,864,884 granted Dec. 16, 1958, and No. 3,235,655 granted Feb. 15, 1966. This type of resistor consists of a semiconductor material with glass and a small amount of reducing agent such as powdered aluminum or carbon. In this type of resistor, the semiconductor material exists as a continuous phase and the resistance of the resistor is dependent mainly upon the resistance characteristics of the semiconductor material that is present. In this type of resistor, the amount of reducing agent, that is carbon or powdered aluminum and the like, are added to obtain

precision-like control of the resistance of the final product, but the amount of reducing agent added should be small so that it is present in the product in a discontinuous phase and, as a result, does not function as a conductor material but solely as a reducing agent. An example of this type of resistor composition consists essentially of 25 parts barium borate glass, 30 parts filler (mullite), 0.8 part aluminum, 0.8 part thermax carbon black, 3 parts bentonite and 45 parts of a semiconductor composition containing 60 parts TiO_2, 20 parts SnO_2, 10 parts Ta_2O_5, 4 parts MoO_3 and 40 parts Al_2O_3.

These two basic types of spark plug resistors described above have been improved by the addition of a water soluble, charable carbon containing material so that they are substantially resistant to change when subjected to electrical aging or field use. This is described fully in the copending patent application Ser. No. 670,178, assigned to the assignee of the present patent application.

All of the resistor compositions referred to above are positioned in the spark plug centerbore of a resistor spark plug between two electrical conductive glass seals. One conductive glass seal bonds tightly to the center electrode head and the other conductive glass seal bonds tightly to the lower end of the terminal screw. It is necessary to use the conductive glass seals on either side of the resistor seal because resistor seals of the type described above do not bond tightly to and maintain good contact with the center electrode head and/or the terminal wire. Resistor spark plugs employing a resistor seal positioned between the two electrical conductive glass seals are typically referred to as three-load resistor plugs.

It is the primary object of this invention to provide an improved resistor composition. It is another object of this invention to provide a resistor composition which will bond tightly to the center electrode head and maintain good contact therewith to provide a stable spark plug resistance over a long period of time. It is another object of this invention to provide a two-load resistor spark plug. It is yet another object of this invention to provide a resistor composition which will form a dense, relatively non-porous resistor mass having high strength which will bond tightly to the center electrode head.

These and other objects are accomplished by a resistor composition containing a compound taken from the group consisting of lithium carbonate, zinc carbonate, magnesium carbonate and sodium carbonate and a metal taken from the group consisting of zinc, antimony and tellurium in addition to the conventional resistor composition ingredients. The resistor composition in accordance with this invention consists essentially of 20 to 75 weight percent glass, 15 to 60 weight percent inert filler, 0 to 60 weight percent semiconductor, 0 to 3.0 weight percent inorganic binder, 0.1 to 4.0 weight percent carbon black, 0.1 to 4.0 water soluble, charable carbon containing material, 0.1 to 10 weight percent of a compound taken from the group consisting of lithium carbonate, zinc carbonate, magnesium carbonate and sodium carbonate and a metal taken from the group consisting of 2 to 15 weight percent zinc, antimony and 0.5 to 10.0 weight percent tellurium. When a spark plug having a resistor composition of this type positioned above and around the nickel center electrode head is heated and pressed with a terminal screw, as in the practice in the art, the resulting fused mass forms a dense, relatively non-porous resistor seal having high strength which bonds tightly to

the center electrode head and which has a stabilized resistance for an extended period of time. The metal taken from the group consisting of zinc, antimony and tellurium alloys with the nickel center electrode head to form a metal coating thereon which enables the resistor composition to bond tightly to the center electrode head thereby eliminating the need for the lower conductive glass seal normally found in the prior art three-load resistor spark plugs. The carbonate, for example, lithium carbonate, improves the formation of the metal, for example, zinc, coating on the center electrode head by modifying the glass which inhibits the flow of the molten zinc down the insulator centerbore around the center electrode shaft. The presence of lithium carbonate in the resistor composition results in a resistor mass having a higher density and a lower porosity thereby increasing the strength of the resistor mass and contributing to a more effective bond between the resistor mass and the center electrode head.

Other objects and advantages of this invention will be apparent from the following detailed description, reference being made to the accompanying drawing wherein a preferred embodiment of this invention is shown.

Referring now to the drawing, the spark plug **10** comprises a conventional outer metal shell **12** having a ground electrode **14** welded to the lower end thereof. Positioned within the metal shell **12** and secured in the conventional manner is the insulator **16**. The ceramic insulator **16** should preferably be of a high aluminum base material containing upwards of 85% aluminum oxide such, for example, as covered by U.S. Pat. No. 2,760,875, issued to Karl Schwartzwalder and Helen Blair Barlett. The insulator **16** is formed with a centerbore having a lower portion **18** of relatively small diameter and an upper portion **20** of larger diameter which are connected by the insulator centerbore ledge **22**. Positioned in the lower portion **18** of the insulator centerbore is the conventional nickel center electrode **24**. The center electrode **24** is preferably nickel although other metals which can be coated with zinc, antimony or tellurium may be used. The center electrode **24** has an enlarged head **26** at the upper end thereof which rests on the inner insulator centerbore ledge **22** and a serrated lower end **28** thereof projecting beyond the lower tip of the insulator **16**. Positioned in the upper portion **20** of the insulator centerbore is a terminal screw **30**. The monolithic resistor element or seal **32** of this invention which will be hereinafter fully described is positioned in the insulator centerbore **20** and is bonded to the center electrode head **26** and to the inner walls of the ceramic insulator. The center electrode **26** has a metal coating **34** thereon which will be hereinafter also fully described. Positioned on top of the resistor seal **32** in the insulator centerbore **20** is a conductive metal-glass seal **36**. The conductive metal-glass seal **36** is bonded to the terminal screw **30** and the inner walls of the ceramic insulator.

The conducting metal-glass seal **36** may be made of any suitable material capable of being bonded to the insulator and to the resistance element and possessing good electrical conductivity. We prefer to use a mixture of glass and conducting materials as described and claimed in the Schwartzwalder et al. Pats. No. 2,106,578 granted Jan. 15, 1938 and No. 2,248,415 granted July 8, 1941, in the Blum et al. Pat. No. 3,349,275 granted Oct. 24, 1967 and in the copending patent application Ser. No. 563,775 assigned to the assignees of this invention. An example of a conducting metal-glass seal is a composition containing 50 parts copper powder, 14 parts zinc powder, 1 part of an organic binder such as hydrogenated cottonseed oil and 35 parts of a borosilicate glass containing 65 weight percent SiO_2, 23 weight percent B_2O_3 5 weight percent Al_2O_3, and 7 weight percent Na_2O. The conductive glass seal **36** together with the resistor seal **32** provides an electrical conductive path from the terminal screw **30** to the center electrode **24**.

In accordance with the present invention, the spark plug resistor seal **32** is a dense, fused mass having high strength and relatively low porosity containing glass, inert filler material, carbon, inorganic binder, a water soluble charable carbonaceous material, a compound taken from the group consisting of lithium carbonate, zinc carbonate, sodium carbonate and magnesium carbonate and a metal taken from the group consisting of zinc, antimony and tellurium. The composition of the resistor seal **32** of our invention may be formed of the following constituents in the percent by weight noted.

	Wt. percent
Glass	20–75
Inert filler—kyanite, borolon, zirconia, mullite, chromium oxide, and the like	15–60
Semiconductor material	0–60
Carbon black	0.1–4.0
Inorganic binder—bentonite (clay)	0–3
Water soluble charable carbon containing material-dextrin, sucrose, methyl cellulose, corn flour, polyvinyl alcohol, glycerin	0.1–4.0
Lithium carbonate, zinc carbonate, sodium carbonate, magnesium carbonate	0.1–10
Zinc, antimony (tellurium)	2–15 (0.5 to 10)

The glass used in the spark plug resistor seal **34** may be any conventional glass commonly used in spark plug seals. Barium borate glasses are preferred although this invention is not limited thereto. An example of a suitable barium borate glass is a composition containing 75 weight percent B_2O_3 and 25% BaO. Another barium borate glass is a composition containing 60 weight percent B_2O_3, 32 weight percent BaO, 6 weight percent Na_2O and 2 weight percent CaO. Another example is a composition containing 60 weight percent B_2O_3, 38 weight percent BaO and 2 weight percent Na_2O. It has been found that the amount of the particulate glass used has no appreciable effect on the resistance of the composition within the limits of 20 to 75 weight percent.

The fluidity of the final resistor composition, as exhibited during the hot pressing operation, is controlled by the presence of a filler which does not react chemically with the other constituents of the resistor composition. These fillers may be selected from the broad group of filler materials commonly used in resistor glass seals. Examples of suitable fillers are kyanite, alumina, zirconia, mullite, borolon and the like. A preferred filler is a mixture of 25 parts by weight kyanite and 35 parts by weight zirconia. The concentration of the filler is from about 25 to 60 weight percent.

Since the resistor composition is best handled in a granulated form, a binder such as bentonite, a very plastic aluminum silicate, is added to bond the particles together during processing. Inorganic binders such as bentonite, as well as other clays, may be used. Organic binders are not recommended for the practice of this invention since it is well known in the art that this type of material causes the resistance of the resistor to be unstable. The concentration of the binder is from 0.0 to 3.0 weight percent.

The concentration of the carbon black in the resistor composition is from about 0.1 to 4.0 weight percent. The carbon black may function as the sole conductor material for resistors of the type described in the McDougal et al. Pat. No. 2,459,282 previously referred to, or solely as a reducing agent as in the resistors of the type described in the Counts et al. Pat. No. 2,864,884.

A water soluble, charable carbonaceous material such as dextrin, sucrose, methocel, corn flour, polyvinyl alcohol and glycerine is incorporated into the resistor mass in a concentration so that this material combines with the carbon black and/or semiconductor material to provide a resistor mass which is resistant to electrical aging. The use of water soluble, charable carbonaceous material in resistors is described in the copending patent application

SN 670,178 assigned to the assignees of the present invention and is incorporated herewith by reference. The concentration of the water soluble, charable carbonaceous material in the resistor composition is from about 0.1 to 4.0 weight percent.

It has been found that a resistor composition containing a compound taken from the group consisting of lithium carbonate, zinc carbonate, sodium carbonate and magnesium carbonate and a metal powder taken from the group consisting of zinc, antimony and tellurium forms a strong relatively non-porous resistor mass which bonds tightly to the nickel center electrode head. These carbonates, lithium carbonate, zinc carbonate, sodium carbonate and magnesium carbonate and these metals, zinc, antimony and tellurium have relatively low melting points which are 1144° F., 572° F., 1499° F., 662° F., 788° F., 1258° F. and 846° F., respectively. The melting points of these materials are lower than the temperature encountered during the assembly of the spark plugs thereby indicating that these materials are most likely in a liquid state during the pressing of the terminal screws in the assembly of the resistor plug. At these elevated temperatures, the carbonates give off carbon dioxide and the cation portion of the molecule that is the lithium, zinc, sodium and magnesium, goes into the glass phase of the resistor mass to modify it. The metal powder melts during the pressing of the terminal screw and while it is in a liquid state, it either forms a metal coating, for example, zinc, or a metal alloy coating, for example, zinc-nickel on the center electrode head. A substantial portion, for example, 70 to 80 percent, of the metal on the resistor composition migrates to the two ends of a resistor composition, thereby making a portion of metal readily available for coating the center electrode head. This coating lessens oxidation of the center electrode and is wetted by the fluid glass seal more effectively during the pressing operation. The presence of the carbonate cation in the glass phase affects the viscosity of the glass phase so that the liquid metal, for example, zinc is inhibited from leaking around the center electrode shaft below the center electrode head thereby enabling the metal to more effectively form a coating. The modified glass has a reduced porosity which results in a stronger resistor mass.

As a result of the metal coating on the center electrode head and of the relatively non-porous, strong glass seal phase, a strong bond between the resistor glass seal phase and the center electrode head is formed. Resistor compositions which are substantially idential except that they do not contain the metal powders referred to above do not adhere to the center electrode head whereas when one of these metals is present, the resistor composition does adhere to the electrode. Resistor compositions which are substantially identical except that they do not contain the carbonates referred to above, do not have as strong and as non-porous a glass phase thereby resulting in a bond which is not as strong. Although the theory mentioned above is believed to be applicable, this invention is not limited thereto.

One advantage of such a system is that one of the conductive glass seals can be eliminated thereby reducing the manufacturing costs. The particle size of the metal powder is not critical in the practice of this invention as long as a uniform resistor composition can be made. The concentration of the zinc or the antimony is 2 to 15 weight percent with the preferred concentration being about 7 to 10 weight percent. The concentration of the tellurium is 0.5 to 10.0 weight percent with the preferred concentration 4 to 5 weight percent. Zinc is the preferred metal.

The concentration of the lithium carbonate, sodium carbonate, zinc carbonate and magnesium carbonate is about 0.1 to 10 weight percent with the preferred concentration being about 0.4 to 1.0 weight percent. Lithium carbonate and zinc carbonate are the preferred carbonates.

The resistor compositions of this invention may be prepared in granular form by first dry mixing the materials and then adding water to make a plastic mass. The plastic mass is then forced through a 20 mesh screen and the resulting granules dried. The dried material is then regranulated through a 28 mesh screen and the material retained between 28 and 100 mesh is used. This sizing procedure has been found to produce granules which are most suitable for uniform volumetric feed in the mass production of resistor spark plugs. Alternatively, the materials may be dry mixed and formed into a free-flowing slip by the addition of water. The slip is then passed into a spray drying tower where the desired agglomerates are formed.

In assembling the spark plug **10**, the center electrode **24** is positioned within the centerbore **18** of the insulator **16**. The desired amount of the granulated resistor composition **32** is then placed in the centerbore on top of the center electrode head **26** and rammed, followed by a measured amount of the powdered copper-glass seal material **36** which is likewise rammed to form the upper seal **36**. A terminal screw **30** is then positioned within the bore and the whole assembly is then heated to a temperature which softens the glass and chars the charable, carbonaceous material, a temperature of 1600 to 1750° F. being generally satisfactory. A temperature of 1675° F. with an 18-minute heating cycle and a 6- to 8-minute hold at the aforementioned temperature is preferred with the barium borate glass used in the preferred embodiment. Other temperatures may be employed for glasses which soften at a lower temperature. When the glass is sufficiently softened, pressure is applied to the terminal screw **30** to force it down into the centerbore, thereby depressing the softened materials and causing the upper seal portion **36** to surround and grip the lower end of the terminal screw. By hot pressing in this manner, which is the customary manner in the art, a continuous electrical path is formed from the terminal screw **30** to the center electrode head **28** through the resistance element and the conductive seal, the portions intermediate the top of the electrode and the bottom of the screw being sealed in glass-tight relationship with the walls of the insulator of the metal parts. The thus formed insulator assembly is then assembled in the shell **12** to form plug **10**.

EXAMPLE NO. 1

A resistor spark plug having a resistor seal composition containing 0.5 part by weight lithium carbonate, 5 parts by weight percent zinc, 30 parts by weight glass, 44 parts by weight zirconia, 25 parts by weight Indian kyanite, 1.8 parts by weight thermax (carbon black), 0.34 part by weight sucrose and 1.83 parts by weight bentonite was made by positioning the resistor composition about the center electrode head in the spark plug centerbore, placing a conventional conductive glass seal on top of the resistor composition and by pressing the terminal screw in to the resistor composition at a temperature in the range of 1600 to 1700° F. The resultant resistor spark plug was cut in half to observe the adherence of the resistor seal to the center electrode. The resistor mass adhered tightly to a zinc or a zinc-nickel coating which covered the nickel center electrode head. The good adherence was attributed to the formation of the metal coating on the center electrode head and a strong, relatively non-porous resistor glass phase.

EXAMPLE NO. 2

A resistor seal composition identical to that described in Example No. 1 except that the lithium carbonate was replaced with 0.5 part by weight sodium carbonate, was used to form a resistor spark plug by the same steps described in Example No. 1. The resultant resistor spark plug had a strong, relatively non porous resistor glass phase which adhered tightly to a zinc or a zinc-nickel coating which covered the nickel center electrode head.

EXAMPLE NO. 3

A resistor seal composition identical to that described in Example No. 1 except that the lithium carbonate was replaced with 0.5 part by weight zinc carbonate, was used to form a resistor spark plug. The resultant resistor spark plug had a strong, relatively non-porous resistor glass phase which adhered tightly to a zinc or zinc-nickel coating which covered the nickel center electrode head.

EXAMPLE NO. 4

A resistor seal composition identical to that described in Example No. 1 except that the lithium carbonate was replaced with 0.5 part by weight magnesium carbonate, was used to form a resistor spark plug. The resultant resistor spark plug had a strong, relatively non-porous glass phase which adhered tightly to a zinc or a zinc-nickel coating which covered the nickel center electrode head.

EXAMPLE NO. 5

A resistor seal composition identical to that described in Example No. 1, except that the zinc was replaced with 5 parts by weight antimony powder, was used to form a resistor spark plug. The resultant resistor spark plug had a strong, relatively nonporous resistor glass phase which adhered tightly to an antimony or an antimony-nickel coating which covered the nickel center electrode head.

EXAMPLE NO. 6

A resistor seal composition identical to that described in Example No. 1 except that the zinc was replaced with 5 parts by weight tellurium, was used to form a resistor spark plug. The resultant resistor spark plug had a strong, relatively non-porous resistor glass phase which adhered tightly to a tellurium or a tellurium-nickel coating which covered the nickel center electrode head.

The semiconductor materials that may be used in this spark plug resistor seal are of the type described in the patent to Counts et al. No. 3,235,655 granted Feb. 5, 1966, which describes a semiconductor material formed from the binary metal oxide systems consisting of TiO_2-ZrO_2, Ta_2O_5-ThO_2, Ta_2O_5-CeO_2 and Ta_2O_5-Di_2O_4 as well as the semiconductors described in the patent to Counts et al. No. 2,864,884 granted Dec. 16, 1958, where the semiconductor material comprises titanium oxide, tin oxide, tantalum oxide, vanadium oxide, molybdenum oxide, and tungsten oxide. The concentration of the semiconductor material is from 0 to 60 weight percent. An example showing the good adherence between this type of resistor composition when it contains zinc is given below.

EXAMPLE NO. 7

A resistor composition containing 0.5 part by weight lithium carbonate, 5 parts by weight zinc, 33 parts barium borate glass, 23 parts kyanite, 2.4 parts thermax (carbon), 2.4 parts aluminum powder, 2.8 parts bentonite and 37 parts of a stannous titanate semiconductor consisting of 44.4 weight percent titanium dioxide, 29.6 weight percent alumina, 14.8 weight percent tin oxide, 7.4 weight percent tantalum oxide and 3.7 weight percent molybdic acid was prepared and placed in the spark plug insulator centerbore around the nickel center electrode head. After the resistor spark plug was completely assembled by the same steps as previously described, the resistor spark plug was cut in half to observe the adherence of the resistor to the center electrode. This resistor spark plug had a zinc-nickel alloy coating on the center electrode head and there was good adherence between the strong, relatively non-porous resistor glass phase and the center electrode head.

These examples indicate that a carbonate taken from the group consisting of lithium carbonate, zinc carbonate, sodium carbonate and magnesium carbonate combine with a metal taken from the group consisting of zinc, antimony and tellurium to promote the formation of a metal coating on the center electrode head which will adhere tightly to the strong, relatively non-porous resistor glass phase and the center electrode head.

The resistor compositions of this invention contain a carbonate taken from the group consisting of lithium carbonate, zinc carbonate, sodium carbonate and magnesium carbonate and a metal taken from the group consisting of zinc, antimony and tellurium which enable a good bond to be formed between the resistor seal and the center electrode thereby reducing the need of a conductor glass phase about the center electrode head. The resistor spark plug of this invention has a high degree of stability when subjected to field use since good contact between the center electrode head and the glass phase of the resistor seal is maintained for substantially the life of the resistor spark plug.

While the invention has been described in terms of specific examples, it is to be understood that the scope of the invention is not limited thereto except as defined by the following claims.

What is claimed is:

1. A gas sealing resistance composition for use in a resistor spark plug comprising glass having a softening temperature of from 1600–1750° F., an inert filler material, a compound taken from the group consisting of lithium carbonate, zinc carbonate, sodium carbonate and magnesium carbonate, and a metal powder taken from the group consisting of zinc, antimony and tellurium, said carbonates and said metals having melting points lower than the softening temperature of said glass.

2. A resistance composition as described in claim 1 wherein the concentration of the compound taken from the group consisting of lithium carbonate, zinc carbonate, sodium carbonate and magnesium carbonate is about 0.1 to 10 weight percent and the concentration of said metal powder taken from the group consisting of zinc and antimony is about 2 to 15 weight percent.

3. A resistance composition as described in claim 1 wherein the concentration of said lithium carbonate is about 0.2 to 1.5 weight percent and the concentration of said zinc powder is about 5 to 10 weight percent.

4. A gas sealing resistance element having a tight bond with the center electrode head in a spark plug insulator centerbore formed from a composition consisting essentially of 20 to 75 weight percent glass having a softening temperature of from 1600–1750° F., 15 to 60 weight percent inert filler, 0 to 60 weight percent semiconductor material, 0.1 to 4.0 weight percent carbon black, 0 to 3 weight percent inorganic binder, 0.1 to 4.0 weight percent water soluble charable carbonaceous material, 0.1 to 10 weight percent of a compound taken from the group consisting of lithium carbonate, zinc carbonate, sodium carbonate and magnesium carbonate and 2 to 15 weight percent of a metal powder taken from the group consisting of zinc and antimony, said composition having been heated in said insulator centerbore to a temperature at which said glass softens and said metal and said carbonate melt, a portion of said metal forming a coating on said center electrode head.

5. A resistor spark plug comprising a ceramic insulator having a centerbore therethrough, a center electrode in said centerbore and a fused gas sealing resistor mass bonded to said center electrode, said resistor mass comprising glass having a softening temperature of from 1600–1750° F. modified by the addition of a compound taken from the group of compounds consisting of lithium carbonate, zinc carbonate, sodium carbonate and magnesium carbonate and a metal taken from the group consisting of zinc, antimony and tellurium, said carbonates and said metals having melting points lower than the softening temperature of said glass, a portion of said metal forming a coating on said center electrode.

6. A gas sealing resistance element having a tight bond with the center electrode head in a spark plug insulator centerbore formed from the composition con-

sisting essentially of 20 to 40 weight percent glass having a softening temperature of from 1600–1750° F., 50 to 70 weight percent inert filler, 1 to 3 weight percent carbon black, 1 to 3 weight percent inorganic binder, 0.2 to 3 weight percent water soluble charable carbonaceous material, 0.2 to 1.5 weight percent lithium carbonate and 5 to 10 weight percent zinc powder, said composition having been heated in said insulator centerbore to a temperature at which said glass softens and said carbonate and said zinc powder melt, a portion of said zinc forming a coating on said center electrode head.

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