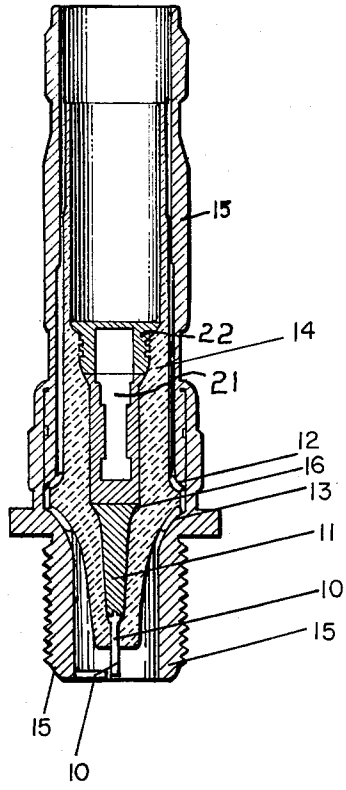
Oct. 30, 1962 C. M. HENDERSON 3,061,756

SPARK PLUG

Filed July 5, 1960 2 Sheets-Sheet 1

FIGURE 1.



Courtland M Henderson

INVENTOR.

BY Herman A. Bauermeister

ATTORNEY

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FIG. 3

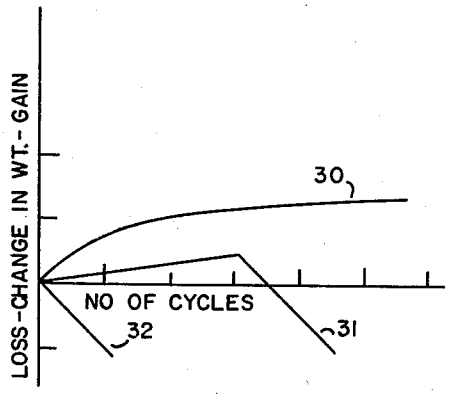


FIG. 4

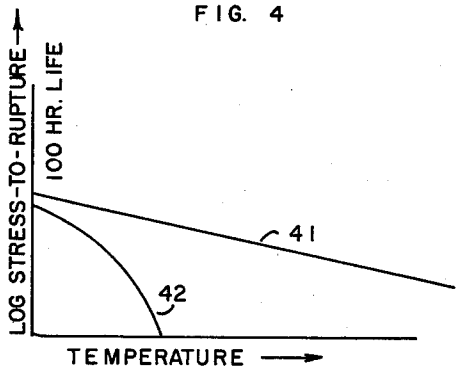


FIG. 5.

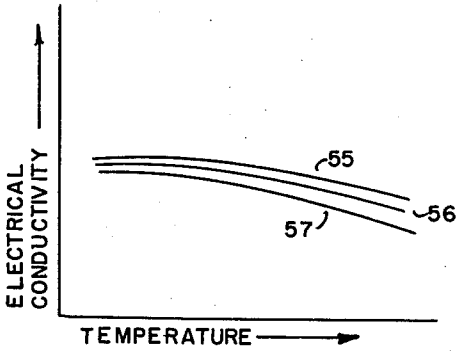


FIG. 6

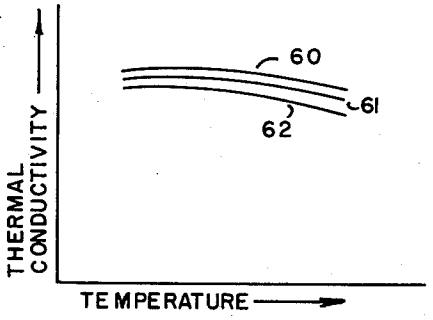
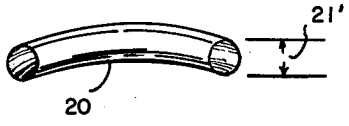


FIG. 2



Courtland M. Henderson

INVENTOR.

BY Herman O. Bauermeister

ATTORNEY

United States Patent Office

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SPARK PLUG

Courtland M. Henderson, Xenia, Ohio, assignor to Monsanto Chemical Company, St. Louis, Mo., a corporation of Delaware

Filed July 5, 1960, Ser. No. 40,662
7 Claims. (Cl. 313—145)

The present invention relates to elements for use as components of spark plugs used in reciprocating and turbine type combustion engines including passenger car, truck, marine, and aircraft engines. It is an object of the invention to provide certain critical elements of spark plugs such as shells, mechanical seals, and conductor cores of unique composition, as well as a process for manufacturing the same. It is a further objective of the invention to provide components that exhibit superior resistance to attack by combustion products of gasolines and other motor fuels, particularly fuels in which additives such as lead and manganese compounds are used to promote better engine operation.

Prior art spark plugs with components composed of conventional metals and alloys have often performed unsatisfactorily because the usual metallic materials were too quickly corroded under severe operating conditions such as are encountered, for example, in high speed marine, truck, and aircraft engines. For example, considerable difficulties are encountered with the mechanical seals between the external metal shell components and the ceramic insulators of plugs operated under severe temperature conditions. The effect of such severe or high temperature operating conditions is to cause prior art metals such as copper or steel alloys to fail unpredictably by creep, thereby causing a loss in cylinder pressure, and resultant drop in engine performance. Such occurrences, especially in the case of aircraft reciprocating engines are, of course, highly dangerous.

It is, accordingly, an object of the present invention to provide spark plug components such as an internal conductive core, an external shell and mechanical seals or gaskets that resist creep at high temperatures.

In order to minimize undesirable preignition without causing fouling of spark plugs, it is common practice to use a relatively large, metal core, located within the ceramic insulator and in contact at the sparking end of the plug with fine wire electrodes. It is an industry practice to make this core of expensive conducting metals such as silver. It is difficult to obtain high quality, thermal junctions between the conductive silver and the poorly conducting ceramic insulators. In manufacturing such spark plugs, the silver must be melted and poured into the cavity within the ceramic insulator with attendant production problems in preventing the molten silver from leaking out past the fine wire electrodes, and of obtaining good thermal contact between the silver and the ceramic insulation. Consequently, it is also an object of the invention to provide spark plug cores that are characterized by improved electrical and heat conductivities as well as greater ease of fabrication. The resultant spark plug is thus operative under extreme engine temperature conditions which would cause the performance of spark plugs made of conventional components to deteriorate.

In the drawings of the present application, FIGURE 1 shows the several components of this invention as incorporated in a typical spark plug. Element **10** represents a fine wire type electrode tip as mounted in a thermally conducting core element **11** which is comprised of the present compositions described below. Element **22** is a conducting piece which is made in two parts for separability and which contains a central opening **21**.

The mechanical and thermal junction between the thermal core element **11** and ceramic element **14**, as well as between gaskets, elements **12** and **13**, and the external shell, **15**, is obtained by the manufacturing process described below. FIGURE 2 shows the type of cross section of the mechanical seal or gasket of the present invention and which are required to produce the desired amount of internal strain for highest creep strength in the materials of the present invention.

The spark plug metallic component comprised of a metal matrix consisting of at least one member selected from the class consisting of nickel, iron, cobalt, tungsten, molybdenum, columbium, tantalum, chromium, vanadium, copper, silver, gold, platinum, and iridium having internally dispersed therein a refractory additive as a reinforcing agent such as a metallic oxide, carbide, boride, silicide or nitride, particularly of the rare earth metals of the lanthanum group, thorium, titanium, zirconium, columbium, tantalum, hafnium, vanadium, molybdenum, and tungsten.

In a preferred embodiment of the invention, the internally dispersed modifying agent is a member of the class consisting of cerium oxide, neodymium oxide, praseodymium oxide, lanthanum oxide, thorium oxide, and mixtures thereof.

In a more preferred embodiment of the invention, the intimately dispersed additives in the said metal matrix are based upon very fine particles or nuclei of the additives, e.g., oxides, such as from 10 to 500,000 angstrom particle size. More preferred particle size ranges are 10 to 10,000 angstroms, or if narrower fractions are desired, 50 to 10,000 angstroms, with the most preferred range being 50 to 225 angstrom particle size.

The concentration of the reinforcing components existing as a distinct phase as nuclei internally dispersed in the matrix metal is from 0.25% to 50% by volume, a preferred range being from 0.25% to 35% by volume. A still more preferred range is from 0.5% to 10% by volume of the refractory components dispersed in the metal matrix.

The use of the present additives, e.g., the above oxides, has been found to result in the production of especially useful shells, gaskets and conductive cores when the reinforcing component such as the oxides existing as nuclei in the metal matrix have an inter-nuclei spacing of 10 angstroms to 200,000 angstroms, or preferably 10 angstroms to 5,000 angstroms.

Still more preferred ranges in the region of the close nuclei spacing is the use of an inter-nuclei spacing of 10 to 225 angstroms.

The metallic components of the present invention are high strength articles of manufacture consisting of a matrix of at least one metal of the matrices described above and having intimately dispersed therein a refractory additive. A preferred group of the said additives is an oxide selected from the group consisting of cerium oxide, neodymium oxide, praseodymium oxide, lanthanum oxide, thorium oxide, and mixtures thereof.

Examples of oxide mixtures commercially available and of utility in the invention have the following approximate compositions:

60% (by weight) of Nd_2O_3, 17% Pr_6O_{11}, 10% Sm_2O_3, and 13% of other rare earth oxides consisting primarily of Gd_2O_3 and CeO_2.

41% (by weight) Pr_6O_{11}, 24% Sm_2O_3, 14% Gd_2O_3, 5% Nd_2O_3, 5% CeO_2, and 11% of other rare earth oxides consisting primarily of Y_2O_3 and La_2O_3.

50% (by weight) Ce oxide, 24% La oxide, 17% Nd oxide, and 9% of other rare earth oxides consisting primarily of Pr oxide, Sm oxide, and Gd oxide.

46% (by weight) La_2O_3, 33% Nd_2O_3, 10% Pr_6O_{11}, 6%

Sm_2O_3 and 7% of other rare earth oxides consisting primarily of Gd_2O_3, CeO_2 and Y_2O_3.

95% (by weight) of ThO_2 and 5% of oxides consisting primarily of rare earth elements.

In general, the various commercially available mixtures of rare earth compounds and the refractory additives derived therefrom may be used with the above critical group of matrices to produce an improved electrode.

In the practice of the present invention, the specific dispersing refractory material, such as the oxide, is the essential additive, although minor proportions of metals other than the matrix metals as described above may also be present. The dispersed refractory material is employed either as a pure material or in various commercial mixtures wherein the said refractory material is the major component.

The metallic spark plug components of the present invention are prepared by consolidating an intimate dispersion of the aforesaid matrix metal and the refractory material. This may be based upon a mechanically blended mixture of the base metal and the dispersed refractory material, or a mixture resulting from chemical precipitation, or coating techniques whereby either the metal or the refractory material is the core and the outer covering of the indicated particles. However, a preferred embodiment of the invention is based upon the preliminary product of a mixture of oxides of the matrix metal group and the oxide group by oxidizing a solution of compounds of the respective components by volatilization and oxidation in a flame.

Such crude mixture is then subjected to reducing conditions such as by contacting with hydrogen gas to reduce the matrix metal while leaving the oxide component dispersed at a molecular level in the metal. The powder is consolidated by hot or cold pressing, extruding, rolling, impact or explosive forming, etc., to obtain the ultimate spark plug parts.

The following examples illustrate specific embodiments of the present invention and show various comparisons against prior art compositions, materials, and processes.

Example 1

One preferred method for preliminarily forming the starting materials of the present invention is to oxidize an atomized solution of at least one soluble salt of the matrix metal selected from the aforesaid group with a salt selected from the said oxide components, e.g., of cerium, neodymium, praseodymium, lanthanum, thorium, and mixtures thereof. In the present specific example, a salt of praseodymium is dissolved in a solvent such as water or alcohol, the said oxidation being conducted by means of an oxidizing flame to produce particles composed of members selected from the group consisting of the free metals and oxides of the first group and the Pr oxide in molecular combination and thereafter subjecting the said particles to reducing conditions, e.g., with hydrogen, to produce the said elemental metal of the aforesaid group, having dispersed therein unreduced Pr oxide. For example, when nickel nitrate and Pr nitrate are dissolved in water in the desired proportions, e.g., to yield 92% (by volume) nickel metal and 8% Pr oxide in the final product, and the said solutions are atomized and oxidized in an oxidizing flame, a powder is produced which is comprised of nickel oxide and Pr oxide. The Pr oxide is dispersed within the individual mixed oxide particles at a molecular level. The foregoing combination of Pr oxide and nickel oxide is reduced at a temperature of from about 500° C. to 700° C. in a hydrogen-containing atmosphere, preferably more than 8 volume percent hydrogen. Other reducing atmospheres such as carbon monoxide, water gas, forming gas, etc. are also useful for this purpose. The nickel oxide is substantially entirely reduced to metallic nickel with the Pr oxide remaining unaffected, and being dispersed at the substantially molecular level within the microstructure of the nickel as a matrix.

Example 2

As another example, when chloroplatinic acid and Ce nitrate in the proportions desired in the ultimate product, e.g., 95% Pt and 5% Ce oxide are dissolved in water and the resulting solution atomized and oxidized in an oxidizing flame, a powder is produced which is comprised of platinum and Ce oxide. The Ce oxide is dispersed within the platinum matrix of the individual particles at a molecular level. This material is readily fabricated to a shaped body under the pressure and temperature conditions set forth herein, e.g., at about 1500 p.s.i. and 1500° C. by hot pressing. After forming the powder comprised of the free metal and having the additive oxide dispersed therein, a preliminary fabrication or compacting step may be employed. This, for example, can consist of hydrostatic compaction, cold pressing, or slip casting, as well as other consolidation procedures to form a densified green billet. Such billets are then consolidated further by sintering in the aforesaid reducing atmosphere at temperatures of about three-quarters of the melting point (absolute) of the metal matrix material. It has been found preferable to use a reducing atmosphere (as by pure hydrogen or hydrogen diluted with nitrogen as obtained from cracked ammonia) in this sintering operation.

The reduced free metal matrix with the molecularly dispersed oxide is consolidated into a shaped body. Preferred conditions for such consolidation are pressing at pressures ranging from 1,000 p.s.i. to 500,000 p.s.i., the most preferred range of consolidation pressures being 40,000 p.s.i. to 140,000 p.s.i. Temperatures for such consolidation may range from room temperatures to 95% of the absolute melting point of the matrix metals. The application of pressure and heat may be carried out simultaneously, as in hot pressing or they may be completed in individual consecutive steps. Other fabrication methods such as slip casting or die compacting at room temperatures may be employed to produce green or preliminary billets of the present compositions that are further densified by sintering at temperatures up to 95% of the absolute melting point and in a reducing atmosphere. Further consolidation and shaping of such preliminary bodies into ultimate cores, gaskets and shells then make use of impact or explosive forming, hot or cold rolling and swaging or other metal fabrication processes due to the ready workability of the materials of this invention.

Example 3

The individual nuclei of the oxide in the broadest aspect of the invention are present with a nucleus-to-nucleus spacing of from 10 to 200,000 angstroms. In a preferred embodiment of the invention of a gasket ring of Ni metal containing 4% Pr oxide, the reinforcing oxide is present in the consolidated metal in a molecular degree of dispersion as shown by X-ray diffraction data, with more than 80% of such oxide nuclei separated at distances of from 10 to 5,000 angstroms. More preferred nucleus-to-nucleus spacing is of the order of from 10 to 225 angstroms and such spacings are quite common in the present components. These figures have also been found to be applicable to the other metal matrices and reinforcing oxides described above.

It has been found that the gaskets of the oval configuration, **20**, shown in FIGURE 2 form gas tight seals in a ready and reproducible manner. It has also been found that the deformation pressure required to provide the necessary gas tight seal must deform the gasket by at least 10% of its vertical height, element **21** of FIGURE 2 so as to introduce the necessary amount of internal strain into the gasket metal of the present invention to give it superior creep resistance. For example, a force of only 45 pounds was sufficient to hot deform the shell and to properly stress the gasket to obtain perfect gas tight seals in one type of spark plug.

Example 4

FIGURE 3 shows the relative resistance to oxidation by Pr oxide-nickel, curve **30**, as compared with a conventional Ni-Cr-W alloy, curve **31**, under cyclic heating and quenching conditions in which the specimen were heated rapidly in an atmosphere of lead compounds, water vapor, carbon dioxide, methane, nitrogen and other gases as produced in an auto or aircraft engine, held for one hour at 950° F. in air, then air quenched to room temperature. The superiority of Pr oxide-strengthened nickel over the conventional Ni-Cr-W heat resistant alloy, curve **31**, under such severe conditions is quite significant and of value for applications where metals are to be used under combustion conditions at temperatures up to and exceeding those used in this test. For example, as shown in curve **30** of FIGURE 3, nickel strengthened with 6.5 volume of Pr oxide showed a leveling off tendency in percent gain-in-weight after 3–4 thermal cycles. Its surface was covered by a thin, impervious and tenaciously bonded coating. As shown in curve **31**, the Ni-Cr-W alloy failed catastrophically after only three cycles and pure nickel, curve **32**, was severely damaged after only one cycle.

An additional and valuable feature of the impervious and tenaciously bonded protective film formed on the surface of metals of the present invention is that the said films are usually self-healing. This is particularly the case for metal matrix materials strengthened with rare earth oxides from the lanthanide series.

Example 5

The strength of Nd oxide-nickel combinations of this invention are significantly higher over a broad range of temperatures than those for conventional alloys used in gaskets and shells of spark plugs. This is shown by comparing curves **41** and **42** in FIGURE 4 where typical stress-to-reduce-rupture in 100 hours versus temperature curves for a 7% by volume of Nd oxide in nickel shell, and a conventional shell steel are shown, respectively, for tests conducted in air.

In FIGURE 4, curve **41** shows that the Nd oxide-nickel metal of this invention had greater strength at room temperature and more than twice the strength of the conventional alloys at 550° C. Clearly, the present metals have superior strength at elevated temperatures to shells and gaskets made of conventional prior art metals and therefore perform more reliably for longer periods of time in spark plugs subjected to severe operating conditions.

The use of the present additives, e.g., the above oxides, has been found to result in the production of especially creep resistant bodies or shells when the reinforcing component such as the oxides existing as nuclei in the metal matrix have an internuclei spacing of 10 angstroms to 200,000 angstroms, or preferably 10 angstroms to 5,000 angstroms. Still more preferred ranges in the region of close nuclei spacing is the use of an internuclei spacing of 10 to 225 angstroms.

The thermal and electrical conductivities of Ce oxide-copper material of the present process as compared with pure silver and pure copper core materials are shown, respectively, in FIGURES 5 and 6 in which values of thermal conductivity and electrical conductivity vs. temperature are plotted.

Example 6

In FIGURE 5, curve **57**, it is shown that the specific use of a 3.5% by volume of Ce oxide in a metal matrix (e.g., copper) gives electrical conductivities which are very close to that for pure copper, curve **56** and in excess of 90% of the electrical conductivity for pure silver, curve **55**.

The thermal conductivity of the Ce oxide-copper metal of this invention is shown as curve **62**, FIGURE 6. Curve **60** shows the thermal conductivity of pure silver and curve **61** represents the thermal conductivity of pure copper. The thermal conductivity of the 3.5% by volume Ce oxide-copper material of this example averaged 90% of the thermal conductivity for silver over the same temperature range and performed in a superior manner to conventional silver cores in that it exhibited greater creep resistance, greater oxidation resistance and greatly reduced the cost of such cores.

In the production of spark plugs of the configuration shown in FIGURE 1 where a thermal core is used to modify fouling and preignition of spark plugs during operation, it has been found that the present process for producing cored type spark plugs greatly decreases the tendency of spark plugs to fail through cracking of the insulator around the thermal core. Such insulator failure has been traced in many cases to the tendency of conventional gravity or centrifugally cast molten silver to bridge across small irregularities in the interior surface of the insulator. Such bridging in effect leaves pockets of greatly reduced thermal conductivity. Such pockets cause hot spots in the ceramic insulator. Such hot spots cause sharp enough thermal gradients under severe engine operating conditions to unpredictably break the insulators.

Example 7

For example, it was found that 2% by volume of cerium oxide in a copper matrix could be formed into a combined core-electrode unit when an electrode tip, element **10** of FIGURE 1, comprised of 6% by volume neodymium oxide strengthened iridium metal matrix was placed in the bottom of a core die, the die filled with the cerium oxide-copper core powdered material and then compacted under a pressure of 90 t.s.i. The resulting core, formed with a taper to approximately match that of the opening in the ceramic insulator, **14**, and sized so that it would protrude above the shoulder, element **16**, of the insulator by a height of about ¼ inch was then annealed for 10 minutes in a reducing or neutral atmosphere at 60% of the absolute melting point of copper. Following the annealing operation, the core was then dropped into the insulator and passed under an automatic press device which upset the oxide-copper core material by 10% to form a top cap and to completely fill the insulator cavity, thereby creating a good fit, devoid of bridged gaps, between the insulator and core. Such integrally processed core and electrode units performed in a superior manner to cores and electrodes made by conventional coating techniques.

Example 8

A still further example which gave superior life and resistance to corrosion attack as well as to thermal failure was produced by a process which utilized a 2.5% by volume of cerium oxide-nickel metal to form the core and electrode tip as a single unitized mass. This metal required 100 t.s.i. compacting pressures and an anneal of 10 minutes at 850–950° C. prior to using an upset volume change of 8% to obtain the desired high quality thermal-mechanical seal between the metal core and the insulator. Spark plugs made with the unitized core-electrode had excellent life and anti-fouling characteristics.

In both the copper and nickel matrix metals, the presence of oxides improved the quality of the junction between the ceramic insulator and narrowed the difference in thermal coefficients between the two materials, thus prolonging the useful life of these spark plugs as compared to plugs made by conventional means.

The use of the present additives, e.g., the above oxides, has been found to result in the production of especially long life cores and electrodes when the reinforcing component such as the oxides existing as nuclei in the metal matrix have an inter-nuclei spacing of 10 angstroms to 200,000 angstroms, or preferably 10 angstroms to 5,000 angstroms.

Still more preferred ranges in the region of close nuclei

spacing is the use of an inter-nuclei spacing of 10 to 225 angstroms.

The present patent application contains subject matter disclosed in copending patent applications, Serial Nos. 27,542; 27,543; 27,544; 27,545; and 27,546 filed May 9, 1960.

The range of particle sizes of the oxide nuclei used for strengthening purposes is from 0.001μ to 1μ, with a preferred range being 0.005μ to 0.5μ and most preferred range being 0.005μ to 0.022μ with spacings between the oxide nuclei ranging from 1 to 1,000 times the oxide dimensions for the said oxide-strengthened materials of this invention.

What is claimed is:

1. A spark plug comprising an insulating body in combination with a pair of spaced electrodes, and having a shell surrounding the said insulating body, the said shell comprising a metal matrix consisting of at least one member selected from the class consisting of nickel, iron, cobalt, tungsten, molybdenum, columbium, tantalum, chromium, vanadium, copper, silver, gold, platinum and iridium having internally dispersed therein as a refractory additive a reinforcing agent selected from the group consisting of a metallic oxide, carbide, boride, silicide and nitride of the rare earth metals of the lanthanide group, thorium, titanium, zirconium, columbium, tantalum, hafnium, vanadium, molybdenum and tungsten.

2. A spark plug comprising an insulating body in combination with a pair of spaced electrodes, a shell surrounding the said insulating body, and with at least one metallic gasket of high creep strength between the said shell and said insulating body, the said gasket comprising a metal matrix consisting of at least one member selected from the class consisting of nickel, iron, cobalt, tungsten, molybdenum, columbium, tantalum, chromium, vanadium, copper, silver, gold, platinum and iridium having internally dispersed therein as a refractory additive a reinforcing agent selected from the group consisting of a metallic oxide, carbide, boride, silicide, and nitride of the rare earth metals of the lanthanide group, thorium, titanium, zirconium, columbium, tantalum, hafnium, vanadium, molybdenum and tungsten.

3. A spark plug comprising an insulating body having an inner passage connecting to an external electrode at the sparking end, the said passage containing a conductive core comprising a metal matrix consisting of at least one member selected from the class consisting of nickel, iron, cobalt, tungsten, molybdenum, columbium, tantalum, chromium, vanadium, copper, silver, gold, platinum and iridium having internally dispersed therein as a refractory additive a reinforcing agent selected from the group consisting of a metallic oxide, carbide, boride, silicide and nitride of the rare earth metals of the lanthanide group, thorium, titanium, zirconium, columbium, tantalum, hafnium, vanadium, molybdenum and tungsten.

4. A spark plug comprising an insulating body and at least two electrodes, and as metallic components in combination therewith a metallic shell surrounding the said insulating body, at least one metallic gasket between the said shell and the said insulating body, the said insulating body having an inner passage connecting to an external electrode at the sparking end, the said passage containing a metallic core, the said metallic components comprising a metal matrix consisting of at least one member selected from the class consisting of nickel, iron, cobalt, tungsten, molybdenum, columbium, tantalum, chromium, vanadium, copper, silver, gold, platinum, and iridium having internally dispersed therein as a refractory additive a reinforcing agent selected from the group consisting of a metallic oxide, carbide, boride, silicide and nitride of the rare earth metals of the lanthanide group, thorium, titanium, zirconium, columbium, tantalum, hafnium, vanadium, molybdenum and tungsten.

5. As an article of manufacture, a metallic spark plug shell comprising a metal matrix consisting of at least one member selected from the class consisting of nickel, iron, cobalt, tungsten, molybdenum, columbium, tantalum, chromium, vanadium, copper, silver, gold, platinum, and iridium having internally dispersed therein as a refractory additive a reinforcing agent selected from the group consisting of a metallic oxide, carbide, boride, silicide and nitride of the rare earth metals of the lanthanide group, thorium, titanium, zirconium, columbium, tantalum, hafnium, vanadium, molybdenum, and tungsten.

6. As an article of manufacture, a metallic gasket of high creep strength suitable for sealing an insulating body from the shell of a spark plug comprising a metal matrix consisting of at least one member selected from the class consisting of nickel, iron, cobalt, tungsten, molybdenum, columbium, tantalum, chromium, vanadium, copper, silver, gold, platinum and iridium having internally dispersed therein as a refractory additive a reinforcing agent selected from the group consisting of a metallic oxide, carbide, boride, silicide and nitride of the rare earth metals of the lanthanide group, thorium, titanium, zirconium, columbium, tantalum, hafnium, vanadium, molybdenum, and tungsten.

7. As an article of manufacture, a highly thermally and electrically conductive core for a spark plug comprising a metal matrix consisting of at least one member selected from the class consisting of nickel, iron, cobalt, tungsten, molybdenum, columbium, tantalum, chromium, vanadium, copper, silver, gold, platinum, and iridium having internally dispersed therein as a refractory additive a reinforcing agent selected from the group consisting of a metallic oxide, carbide, boride, silicide, and nitride of the rare earth metals of the lanthanide group, thorium, titanium, zirconium, columbium, tantalum, hafnium, vanadium, molybdenum and tungsten.

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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,061,756 October 30, 1962

Courtland M. Henderson

It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 8, line 52, after "tungsten." insert the following claims:

8. As an article of manufacture a metallic component of a spark plug comprising a metallic matrix of copper, having internally dispersed therein cerium oxide.

9. As an article of manufacture a metallic component of a spark plug comprising a metallic matrix of nickel having internally dispersed therein praseodymium oxide.

in the heading to the printed specification, line 7, for "7 Claims" read -- 9 Claims --.

Signed and sealed this 12th day of May 1964.

(SEAL)
Attest:

ERNEST W. SWIDER
Attesting Officer

EDWARD J. BRENNER
Commissioner of Patents

UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

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