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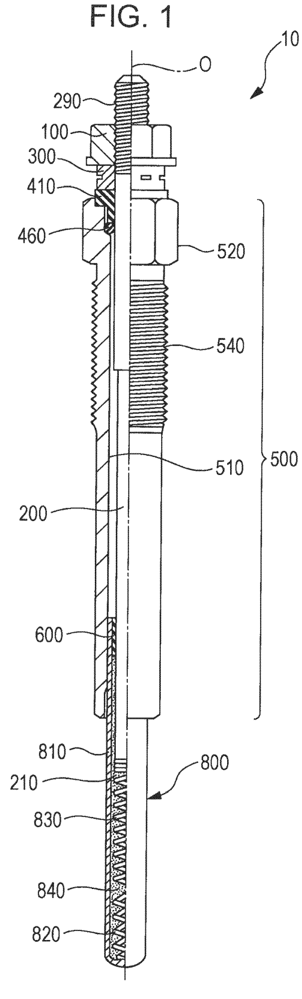
(54) ## GLOW PLUG

(57) [Object] An object of the present invention is to provide a glow plug that undergoes a smaller decrease in life despite the high-temperature employed during afterglow, with which short-circuiting of the glow plug and deterioration of the resistive heating element are suppressed during afterglow that involves using the glow plug in such a manner that the internal temperature around the resistive heating element reaches 1200°C or higher.

[Solving Means] A glow plug includes a heater that includes a tubular sheath having a distal end that is closed, a resistive heating element disposed in the tubular sheath, and insulating powder disposed around the resistive heating element in the tubular sheath. The resistive heating element is formed of an alloy containing aluminum. The insulating powder contains MgO and at least one oxide selected from the group consisting of CrO_3, Cr_2O_3, SnO, FeO, Fe_3O_4, MnO, MnO_2, Mn_2O_3, Mn_3O_4, SiO_2, WO_2, TiO_2, Ti_2O_3, and ZrO_2. "A" representing an amount of aluminum contained in the resistive heating element in terms of mass percent and "S" representing an amount of the oxide contained in the insulating powder in terms of mass percent on an oxide basis satisfy $0.04 \leq S/A \leq 0.31$.

FIG. 1



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Description

BACKGROUND OF THE INVENTION

1. Field of the Invention

[0001] The present invention relates to a glow plug for use as an auxiliary heat source for an internal combustion engine such as a diesel engine.

2. Description of the Related Art

[0002] Glow plugs are used as auxiliary heat sources for internal combustion engines such as compression-ignition diesel engines. A typical glow plug is constituted by a bottomed tubular sheath having a closed distal end and an open proximal end and a resistive heating element disposed inside the sheath to extend in the axial line direction of the tubular sheath. The resistive heating element is configured to generate heat when electrified. The distal end of the resistive heating element is connected to the distal end of the tubular sheath. The proximal end of the resistive heating element is connected to a center rod extending from the proximal end of the tubular sheath. The resistive heating element generates heat when electrified through the center rod. The interior of the tubular sheath is filled with insulating powder such as magnesia powder so that the outer peripheral surface of the resistive heating element and the inner peripheral surface of the tubular sheath are insulated from each other.

[0003] Recent years have seen growing demand for glow plugs with increased life. For example, Japanese Patent No. 4076162 (hereinafter referred to as the ’162 document) discloses providing oxygen donors inside a glow tube to form an aluminum oxide layer on a heating coil surface before or during heating of the heating coil. This is to avoid local elevation of electrical resistance and premature malfunction of the heating coil caused by formation of nitrides on the peripheral layer of the heating coil resulting from entry of air into the glow tube (in particular, refer to paragraphs 0005, 0021, etc., of the ’162 document, for example). The ’162 document discloses TiO_2 and ZrO_2 as examples of the oxygen donors (refer to paragraphs 0023 and 0024 of the ’162 document). The ’162 document also discloses that in the case where a control coil does not contain an aluminum component and a silicone component, oxygen released from the oxygen donors causes corrosion instead of forming an oxide layer, and thus the substances that act as oxygen donors are allowed to exist only in the heating coil 10 region and not in the control coil region (refer to paragraph 0022 etc., of the ’162 document).

[0004] Japanese Patent Application Laid-open No. 2011-12898 (hereinafter referred to as the ’898 document) discloses the ’162 document as a related art (refer to paragraph 0005 in the ’898 document). The ’898 document discloses that in the case where a metal oxide considered to enable re-formation of Al_2O_3 films over a long term is contained in the tube based on the invention described in the ’162 document, the metal oxide becomes reduced and turns into a metal having electrical conductivity, which may result in short-circuiting between the heating coil and the tube due to the existence of the conductive metal in the insulating powder (refer to paragraph 0007 of the ’898 document). In view of such a technical issue, the ’898 document provides a "sheath heater and a glow plug with which durability of the heating coil can be notably improved" (refer to paragraph 0009 of the ’898 document) and discloses a "sheath heater (Claim 1 of the ’898 document) in which an oxide film formed of a metal oxide having an equilibrium dissociation pressure of 10^{-10} Pa or higher at 1000°C is formed on the inner peripheral surface of the tube".

[0005] Glow plugs not only enhance starting abilities of engines by warming the air-fuel mixture upon being electrified at the time of starting the engines, but also enhance stability of the engines and decrease emission gas if electrification is continued for a certain time after starting the engines. Rapidly increasing the temperature of the glow plug by electrification at the time of starting the engine is called preglow. Continuing electrification of the glow plug after the engine has been started is called afterglow. Because the amount of emission gas can be reduced by increasing the temperature of the glow plug during afterglow, the recent trend has been toward increasing the temperature of the glow plug during afterglow. However, if a glow plug having a resistive heating element formed of an aluminum-containing alloy is heated to a temperature higher than in typical practice, i.e., in such a manner that the internal temperature around the resistive heating element reaches 1200°C or higher during afterglow, the glow plug undergoes short-circuiting and has a shorter life.

SUMMARY OF THE INVENTION

[0006] An object of the present invention is to provide a glow plug that undergoes a smaller decrease in life despite the high-temperature employed during afterglow, with which short-circuiting of the glow plug and deterioration of the resistive heating element are suppressed during afterglow that involves using the glow plug in such a manner that the internal temperature around the resistive heating element reaches 1200°C or higher.

[1] An aspect of the present invention provides a glow plug that includes a heater. The heater includes a tubular sheath having a distal end that is closed, a resistive heating element disposed in the tubular sheath, and insulating powder disposed around the resistive heating element in the tubular sheath. The resistive heating element is formed of an alloy containing aluminum. The insulating powder contains MgO and at least one oxide selected from the group consisting of CrO_3, Cr_2O_3, SnO, FeO, Fe_3O_4, MnO, MnO_2, Mn_2O_3, Mn_3O_4, SiO_2, WO_2, TiO_2, Ti_2O_3, and ZrO_2. "A" representing an amount of aluminum contained in the resistive heating element in terms of mass percent and "S" representing an amount of the oxide contained in the insulating powder in terms of mass percent on an oxide basis satisfy $0.04 \leq S/A \leq 0.31$.

[0007] Preferable embodiments of [1] are as follows.

[2] In the glow plug recited in [1], the oxide may be SiO_2.

[3] The glow plug recited in [1] or [2] may further include a center rod electrically coupled to the resistive heating element, the center rod having a part that protrudes from an opening of a proximal end of the tubular sheath, and a packing disposed between the center rod and the tubular sheath to inhibit gas circulation between interior and exterior of the tubular sheath.

[0008] Since insulating powder disposed around the resistive heating element of this glow plug contains MgO and at least one oxide selected from the group consisting of CrO_3, Cr_2O_3, SnO, FeO, Fe_3O_4, MnO, MnO_2, Mn_2O_3, Mn_3O_4, SiO_2, WO_2, TiO_2, Ti_2O_3, and ZrO_2, and the amount of the oxide is adjusted to a particular ratio relative to the amount of aluminum in the resistive heating element, short-circuiting of the glow plug and deterioration of the resistive heating element can be suppressed even when the glow plug is operated at an inner temperature as high as 1200°C or higher around the resistive heating element during afterglow. A glow plug that undergoes a less decrease in life at high temperature of afterglow can be provided.

BRIEF DESCRIPTION OF THE DRAWINGS

[0009]

Fig. 1 is a partially sectional view of a glow plug according to one embodiment.

Fig. 2 is a partially sectional view of a related part showing a heater of the glow plug shown in Fig. 1 in close-up.

Fig. 3A is a diagram showing a part of a section of a heater of a typical glow plug observed with a scanning electron microscope (SEM), and Fig. 3B is a diagram showing a part of a section of a heater of the glow plug according to one embodiment observed with a SEM.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

[0010] Fig. 1 is a partially sectional view that shows a glow plug according to an embodiment of the present invention. In Fig. 1, the lower side of the plane of the paper of Fig. 1 along the axial line O, in other words, the side on which a heater described below is disposed, is referred to as a distal end side. The upper side of the plane of the paper of Fig. 1 along the axial line O is referred to as a proximal end side.

[0011] Referring to Fig. 1, a glow plug 10 includes a center rod 200, a metal shell 500, and a heater 800 that generates heat when electrified. These parts are assembled along the axial line O of the glow plug 10.

[0012] The metal shell 500 is formed of carbon steel or the like and has a substantially cylindrical shape. The metal shell 500 has an axis hole 510 that extends in an axial line O direction. The metal shell 500 holds the heater 800 at the distal-end-side end of the axis hole 510. The metal shell 500 holds the center rod 200 at the proximal-end-side end of the axis hole 510 via an insulating member 410 and an O ring 460. The position of the insulating member 410 with respect to the axial line O direction is fixed when a ring 300 in contact with the proximal end of the insulating member 410 is crimped onto the center rod 200. The proximal end side of the metal shell 500 is insulated by the insulating member 410. The metal shell 500 accommodates a part of the center rod 200 and a space is formed between the axis hole 510 and the center rod 200 so as to electrically insulate the metal shell 500 and the center rod 200 from each other. The metal shell 500 includes a tool engaging portion 520 and an external thread portion 540. The tool engaging portion 520 is formed on the outer peripheral surface of the metal shell 500. A tool such as a torque wrench engages with the tool engaging portion 520. The external thread portion 540 is formed on the outer peripheral surface of the metal shell 500, at a position on the distal end side with respect to the tool engaging portion 520. The external thread portion 540 is screwed into the installation hole of an engine head when the glow plug 10 is mounted onto the engine head of an internal combustion engine such as a diesel engine.

[0013] The center rod 200 is formed of a conductive material and has a columnar shape. The center rod 200 is inserted into the metal shell 500 and arranged to extend along the axial line O. The center rod 200 has a center rod distal end portion 210 at the distal end and a connecting portion 290 at the proximal end. The center rod distal end portion 210 is inside the heater 800. The connecting portion 290 protrudes from the metal shell 500 and has an external thread formed on its outer peripheral surface. The connecting portion 290 is fitted into an engaging portion 100. A connector-equipped cable to which power is supplied from a power source such as a battery is connected to the engaging portion 100.

[0014] Fig. 2 is a partially sectional view of a related part showing the heater 800 in close-up. The heater 800 includes a tubular sheath 810, a heating coil 820, a control coil 830, and insulating powder 840. The heating coil 820 is an example of the resistive heating element of the present invention.

[0015] The tubular sheath 810 extends in the axial line O direction and has a bottomed tubular shape with a closed distal end. The tubular sheath 810 is formed of a Ni alloy containing Ni as a main component. An example of the Ni alloy is a Ni-Cr-Fe alloy. Examples of the Ni-Cr-Fe alloy include Inconel 601 (registered trademark) and Alloy 602CA (registered trademark). The tubular sheath 810 accommodates the heating coil 820, the control coil 830, and the insulating powder 840. The tubular sheath 810 has a sheath distal end portion 811 and a sheath proximal end opening 819. The sheath distal end portion 811 is at the distal end of the tubular sheath 810 and is rounded toward the outer side. The sheath proximal end opening 819 is an open end of the tubular sheath 810 at the proximal end. A part of the center rod 200 that protrudes from the sheath proximal end opening 819 is disposed inside the tubular sheath 810. The tubular sheath 810 is electrically insulated from the center rod 200 by a packing 600 and the insulating powder 840. The packing 600 is an insulating member interposed between the center rod 200 and the tubular sheath 810. The packing 600 inhibits gas circulation between the interior and the exterior of the tubular sheath 810 and keeps the interior of the tubular sheath 810 airtight. The tubular sheath 810 is electrically coupled to the metal shell 500.

[0016] The heating coil 820 is formed of an aluminum-containing alloy and is electrically conductive. Examples of the alloy forming the heating coil 820 include alloys that contain an element such as Fe or Cr as a main component and Al. Examples of such alloys include Fe-Cr-Al alloys such as Kanthal (registered trademark) and Pyromax (registered trademark). The heating coil 820 generates heat when electrified. The heating coil 820 is disposed in the tubular sheath 810 to extend in the axial line O direction. The heating coil 820 has a heating coil distal end portion 821 at the distal end and a heating coil proximal end portion 829 at the proximal end. The heating coil distal end portion 821 is welded to a portion of the tubular sheath 810 near the distal end so as to be electrically coupled to the tubular sheath 810.

[0017] The control coil 830 is formed of a metal that has a temperature coefficient of electrical resistivity higher than that of the alloy that forms the heating coil 820. The control coil 830 is electrically conductive. Examples of the metal that forms the control coil 830 include metals that contain Ni, Co, Fe, and the like as main components. Examples of such metals include pure Ni, Co-Ni alloys, and Co-Fe alloys. The control coil 830 controls the power fed to the heating coil 820. The control coil 830 is disposed inside the tubular sheath 810 so as to extend in the axial line O direction. The control coil 830 has a control coil distal end portion 831 at the distal end, and a control coil proximal end portion 839 at the proximal end. The control coil distal end portion 831 is welded onto the heating coil proximal end portion 829 of the heating coil 820 so as to be electrically coupled to the heating coil 820. The control coil proximal end portion 839 is joined to the center rod distal end portion 210 of the center rod 200 so as to be electrically coupled to the center rod 200.

[0018] The composition of the heating coil 820 and the composition of the control coil 830 can be determined by using a wave dispersive spectrometer (WDS) of an electron probe micro analyzer (EPMA) as follows. In the description below, the heating coil 820 is described as an example. In a first step, qualitative analysis is performed on the heating coil 820 by using the WDS of the EPMA. The analysis identifies the elements contained in the heating coil 820. The element that has the largest content in terms of mass percent is assumed to the main component.

[0019] In the first step, the heater 800 is embedded in a resin and cut to expose a section that includes the axial line O, and the section is polished to mirror finish. As shown in Figs. 3A and 3B, plural circular spots 820 are observed in one section. These circular spots are sections of the heating coil 820. From these circular spots 820, ten circular spots are arbitrarily selected. For each circular spot 820, five points near the center of the spot 820 are arbitrarily selected and used as the measurement points for elemental analysis. The arithmetic means of the measured values taken from a total of 50 points is assumed to be the composition of the heating coil 820. Measurement points are not to be selected from portions near where the heating coil 820 is welded to the tubular sheath 810, portions near where the heating coil 820 is welded to the control coil 830, or portions near the boundary to another component having a different composition.

[0020] In a second step, measurement conditions for EPMA are determined. This is to enhance accuracy of the analysis. The measurement conditions are determined so that 10,000 or more counts are obtained at a beam current value that does not cause count losses resulting from a large dose of incoming X-rays during detection of the main component identified in the first step.

[0021] In a third step, the elements identified in the first step are subjected to quantitative analysis under conditions determined in the second step. The measurement conditions are set as follows, for example. At an acceleration voltage of 20 kV, a probe current of 2.5×10^{-8} A, and a beam spot size of 100 μm, the main peak is acquired for 10 seconds and the background is acquired for 5 seconds for the high-angle side and the low-angle side each. The count per second (CPS) of each element is obtained from the raw intensity, and quantitative calculation is performed by a ZAF method using CPSs of comparative samples (standard samples produced by ASTIMEX) analyzed under the same conditions. The element contents of the comparative samples are determined in advance. ZAF is an acronym of Z effect (atomic number effect), absorption effect, and fluorescence excitation effect. During the quantitative calculation, normalization is performed so that the total of the element contents is 100%.

[0022] Next, the insulating powder 840 is described. The insulating powder 840 is powder having an electrically insulating property. Since electrically insulating powder fills the interior of the tubular sheath 810, the inner peripheral surface of the tubular sheath 810 is electrically insulated from the outer peripheral surfaces of the heating coil 820, the control coil 830, and the center rod 200. The insulating powder 840 contains MgO and at least one oxide selected from CrO_3, Cr_2O_3, SnO, FeO, Fe_3O_4, MnO, MnO_2, Mn_2O_3, Mn_3O_4, SiO_2, WO_2, TiO_2, Ti_2O_3, and ZrO_2. This oxide is more easily reducible than MgO. The insulating powder 840 is arranged so as to at least surround the heating coil 820 in the tubular sheath 810 and is preferably arranged so as to surround both of the heating coil 820 and the control coil 830 in the tubular sheath 810. More preferably, the insulating powder 840 fills all parts of the interior of the tubular sheath 810. In other words, whereas MgO in the insulating powder 840 fills all parts of the interior of the tubular sheath 810, the oxide in the insulating powder 840 is to be present at least around the heating coil 820, is preferably present around the heating coil 820 and the control coil 830, and is more preferably present in all parts of the interior of the tubular sheath 810. According to this glow plug 10, the insulating powder 840 containing the oxide at a particular ratio surrounds at least the heating coil 820. Thus, when the glow plug 10 during afterglow is operated to reach a temperature higher than in typical practice in such a manner that the inner temperature around the heating coil 820 reaches 1200°C or higher, short-circuiting of the glow plug 10 is prevented and the decrease in life can be suppressed. The meaning of the phrase, the "inner temperature around the heating coil 820" refers to a temperature inside the tubular sheath 810 around a part of the heating coil 820 predicted to reach the highest temperature. For example, this temperature is a temperature of a portion surrounded by the coil of the heating coil 820, the portion being near the center of the distal end side of the heater 800.

[0023] The insulating powder 840 containing the oxide at a particular ratio and being placed around the heating coil 820 prevents short-circuiting of the glow plug 10 and suppresses a decrease in life of the glow plug 10 when the glow plug 10 during afterglow is operated to reach a temperature higher than in typical practice in such a manner that the inner temperature around the heating coil 820 reaches 1200°C or higher. This is presumably due to the following reason. As discussed earlier, if the glow plug 10 having a heating coil 820 formed of an Al-containing alloy is operated to reach an inner temperature of 1200°C or higher around the heating coil 820 during afterglow, which is a temperature higher than in typical practice, the glow plug 10 undergoes short-circuiting and the life of the glow plug 10 is shortened. A section of the heater 800 of a short-circuited glow plug 10 is taken and observed with a scanning electron microscope (SEM). The observation reveals that there is a region inside the tubular sheath 810 where Mg aggregation occurred (hereinafter this region is referred to as a "Mg aggregation region 850"), as shown in Fig. 3A. Fig. 3A shows an example in which the Mg aggregation region 850 extends from the distal end of the center rod 200 to near a region where the control coil 830 is joined to the center rod 200 and to a region that comes in contact with the tubular sheath 810; however, the location where the Mg aggregation region 850 occurs is not particularly limited. The Mg aggregation region 850 may occur between the center rod 200 and the tubular sheath 810, between the heating coil 820 and the tubular sheath 810, or between the control coil 830 and the tubular sheath 810 in some cases, for example. Short-circuiting between the tubular sheath 810 and the control coil 830 and between the center rod 200 and the tubular sheath 810 is presumably caused by the Mg aggregation region 850 shown in Fig. 3A that extends from the distal end of the center rod 200 to near a region where the control coil 830 is joined to the center rod 200 and to a region that comes in contact with the tubular sheath 810. The Mg aggregation region 850 is presumably resulted from use of the glow plug 10 at high temperature during afterglow since Al contained in the heating coil 820 reduces MgO, which is electrically insulating powder disposed around the heating coil 820, and Mg thereby turns into vapor, travels inside the tubular sheath 810, and precipitates and aggregates at a particular location. The inventor has conceived that reduction of MgO can be suppressed by using electrically insulating powder that contains not only MgO but also a particular ratio of an oxide that is more easily reducible than MgO, and formation of the Mg aggregation region 850 can be suppressed thereby. Thus, the present invention has made.

[0024] The insulating powder 840 contains the oxide at a particular ratio relative to the Al content of the heating coil 820. In other words, A representing the Al content of the heating coil 820 (the amount of Al contained in the heating coil 820) in terms of mass percent and S representing the oxide content of the insulating powder 840 (the amount of the oxide contained in the insulating powder 840) in terms of mass percent on an oxide basis satisfy the following formula (1):

$$0.04 \leq S/A \leq 0.31 \cdots (1)$$

[0025] At S/A smaller than 0.04, the oxide content is too small to achieve the effects of suppressing reduction of MgO and occurrence of the Mg aggregation region when the glow plug 10 is operated at an inner temperature as high as 1200°C or higher around the heating coil 820 during afterglow. At S/A greater than 0.31, an excessively large oxide content accelerates reaction between Al in the heating coil 820 and oxygen from the oxide and deteriorates the heating coil 820 when the glow plug 10 is operated at an inner temperature as high as 1200°C or higher around the heating coil 820 during afterglow. The insulating powder 840 containing the oxide and satisfying the formula (1) above can suppress short-circuiting of the glow plug 10 that occurs when the glow plug is operated at an inner temperature as high as 1200°C or higher around the heating coil 820 during afterglow, and the decrease in life of the glow plug 10 can be suppressed since deterioration of the heating coil 820 is suppressed. Preferably, S/A is 0.29 or lower.

[0026] The oxide is a substance more easily reducible than MgO. Examples of the substance that is more easily reducible than MgO include oxides having a larger standard free energy of formation $\Delta G^0 f$ at 1200°C than MgO. The standard free energy of formation $\Delta G^0 f$ of the oxide at 1200°C can be determined by calculation; however, the data can also be acquired from "Diagram of standard free energy of formation of oxides" found on page 106 of "Metal Data Book, 4th edition [Kinzoku Deta Bukku, Kaitei 4 han]" (third print published April 30, 2007 by the Japan Institute of Metals and Materials). Among the oxides having a standard free energy of formation $\Delta G^0 f$ larger than that of MgO shown in this diagram, those oxides in which an element other than oxygen has a temperature of 1200°C or higher at a vapor pressure of 1 mmHg are used as the oxide. Specific examples of such oxides are CrO_3, Cr_2O_3, SnO, FeO, Fe_3O_4, MnO, MnO_2, Mn_2O_3, Mn_3O_4, SiO_2, WO_2, TiO_2, Ti_2O_3, and ZrO_2. The oxide contained in the insulating powder 840 may be one selected from these oxides or a combination of two or more oxides selected from these oxides. The insulating powder 840 preferably contains at least one oxide selected from SiO_2, TiO_2, and ZrO_2 among these. The larger the standard free energy of formation $AG^0 f$, the less easily oxidizable and more easily reducible the oxide. Presumably, when the insulating powder 840 contains an oxide that is more easily reducible than MgO, the oxide is reduced first and reduction of MgO is suppressed. The interior of the heater 800 is substantially at vacuum, i.e., about 1 mmHg. Accordingly, if the element in the oxide other than oxygen has a temperature of 1200°C or higher at a vapor pressure of 1 mmHg and the oxide is reduced by increasing the inner temperature to 1200°C or higher around the heating coil 820, the element in the oxide other than oxygen does not easily vaporize and travel within the tubular sheath 810. As a result, aggregation of the element in the tubular sheath 810 is suppressed, and a dispersed state is easily obtained. Accordingly, the insulating powder 840 containing the oxide can suppress formation of the Mg aggregation region and short-circuiting of the glow plug 10 when the glow plug 10 is operated at an inner temperature as high as 1200°C or higher around the heating coil 820 during afterglow.

[0027] The insulating powder 840 may contain an impurity such as CaO in addition to MgO and the oxide more easily reducible than MgO. The impurity content of the insulating powder 840 is preferably 0.7% by mass relative to the total mass of the insulating powder 840.

[0028] The components contained in the insulating powder 840 and their contents can be determined as follows. First, the insulating powder 840 is subjected to qualitative analysis by powder X-ray diffraction or the like so as to identify the components contained in the insulating powder 840. Then the elements contained in the insulating powder 840 are subjected to quantitative analysis by ICP emission spectroscopy described below. In the case where the components contained in the insulating powder 840 are known to be oxides by qualitative analysis, the element contents obtained by the quantitative analysis can be converted into oxide contents to determine the oxide contents. In the case where the main component of the insulating powder 840 is known to be MgO by the qualitative analysis, the ICP emission spectroscopy may be performed on the components other than MgO and the MgO content can be determined as the balance thereof.

[0029] In a first step, 0.5 g or slightly more than 0.5 g of insulating powder 840 is prepared. If 0.5 g of the insulating powder 840 cannot be sampled from one glow plug 10, the insulating powder 840 may be sampled from more than one glow plugs 10 (same type number) prepared in the same manner. The accurate mass of the insulating powder 840 thus prepared is recorded. For example, in the case where the unit is gram, the mass is measured down to five places of decimals, such as 0.50484 g. The insulating powder 840 and 10 mL of a diluted hydrochloric acid containing an undiluted hydrochloric acid and pure water at a 1:1 ratio are placed into a beaker. The beaker is covered and heated on a hot plate to dissolve the insulating powder 840.

[0030] In a second step, the heated and dissolved solution is filtered. The filter with adhering residue and the filtrate obtained by filtration are separated.

[0031] In a third step, the filter with the adhering residue is placed in a platinum crucible. The crucible is heated with a burner to burn the filter (ashing treatment).

[0032] In a fourth step, potassium sodium carbonate powder and boric acid powder are placed into the platinum crucible containing the residue obtained in the third step. The crucible is heated to 1000°C using a burner to melt the content (alkali fusion treatment).

[0033] In a fifth step, the fused product that has cooled and solidified after the alkali fusion treatment and 10 mL of a diluted hydrochloric acid containing undiluted hydrochloric acid and pure water at 1:1 ratio are placed in a beaker to dissolve the fused product.

[0034] In a sixth step, the solution obtained in the second step and the solution obtained in the fifth step are separately analyzed by induction-coupled plasma (ICP) emission spectroscopy. During analysis, the concentration is calculated on the basis of the mass measured in the first step. Qualitative analysis and provisional quantitative analysis are possible by this analysis. The analysis is conducted with a commercially available ICP emission spectroscope. In the case where the solution obtained in the second step and the solution obtained in the fifth step contain the same element identified by the analysis, the sum is assumed to be the concentration of that element.

[0035] In a seventh step, standard solutions for an element detected by the qualitative analysis in the sixth step are prepared (for example, a standard solution containing 1000 ppm of Si is prepared). The standard solution is diluted to prepare three types of diluted standard solutions (thin solution, intermediate solution, and thick solution). At least the thick standard solution is prepared so that the concentration thereof exceeds the concentration obtained by the provisional quantitative analysis. The solution with zero concentration and the three diluted standard solutions are analyzed by ICP emission spectroscopy and calibration curves showing the relationship between the known concentrations and the optical intensities obtained by the ICP emission spectroscopy are determined (for example, calibration curves plotted along the x axis indicating the concentration and the y axis indicating the optical intensities obtained by the ICP emission spectroscopy). The results (intensities) of the provisional quantitative analysis are compared with the calibration curves so as to obtain the quantitative analysis results. The results of the quantitative analysis of other elements obtained by the qualitative analysis in the sixth step are also obtained in the same manner.

[0036] In an eighth step, the same steps as the first to seventh steps are performed again and the average values of two sets of the analysis results are assumed to be the element contents in the insulating powder 840. In the case where the components contained in the insulating powder 840 are known to be oxides by the previously conducted qualitative analysis, the observed element contents are converted into oxide contents so that the oxide contents of the insulating powder 840 are calculated.

[0037] The glow plug 10 is prepared as follows, for example.

[0038] First, a resistive heating wire having a particular composition is processed into coils to prepare a heating coil 820 and a control coil 830. Then an end of the heating coil 820 is joined to an end of the control coil 830 by arc welding or the like to form a coil member. The control coil 830-side of the coil member is joined to the distal end of a center rod 200.

[0039] A steel pipe having a particular composition is processed to have a diameter larger than the final dimension of the tubular sheath 810. A distal end portion thereof is processed to have a smaller diameter than the rest of the pipe so as to form a tubular sheath precursor having a narrow end.

[0040] The coil member integrated with the center rod 200 is inserted into the tubular sheath precursor so that the distal end portion of the heating coil 820 comes at the narrow distal end opening of the tubular sheath precursor. The distal end opening of the tubular sheath precursor and the distal end portion of the heating coil 820 are fused by arc welding or the like so as to close the distal end of the tubular sheath precursor and form a heater precursor having the coil member inside.

[0041] Next, the interior of the tubular sheath 810 of the heater precursor is filled with insulating powder 840 containing MgO powder and powder of an oxide more easily reducible than MgO. The insulating powder 840 fills the interior of the tubular sheath 810 so that the insulating powder 840 surrounds at least the heating coil 820. The insulating powder 840 preferably surrounds both the heating coil 820 and the control coil 830 in the tubular sheath 810. Alternatively, the insulating powder 840 may be arranged so that only the MgO powder surrounds the control coil 830. As mentioned earlier, powder of at least one oxide selected from CrO_3 , Cr_2O_3, SnO, FeO, Fe_3O_4, MnO, MnO_2, Mn_2O_3, Mn_3O_4, SiO_2, WO_2, TiO_2, Ti_2O_3, and ZrO_2 is used as the oxide powder. The MgO powder and the oxide powder are thoroughly mixed with each other and placed in the tubular sheath 810. The oxide powder preferably has an average particle size smaller than that of the MgO powder since such oxide powder can be evenly dispersed in the MgO powder. A packing 600 is inserted into the proximal end opening of the tubular sheath 810 to seal the tubular sheath 810. The tubular sheath 810 is swaged to form a heater 800 having a particular outer diameter.

[0042] The heater 800 prepared as such is press-fitted and fixed into the axis hole 510 of the metal shell 500. An O ring 460 and an insulating member 410 are fitted into the proximal end portion of the metal shell 500, and the center rod 200 is crimped with a ring 300. As a result, a glow plug 10 is obtained.

[0043] A glow plug according to the present invention is used as an auxiliary heat source of an internal combustion engine such as a compression ignition diesel engine or the like. The glow plug is fixed at a particular position as the external thread portion 540 is threaded into a threaded hole in a head that defines and forms a combustion chamber of an internal combustion engine. The glow plug undergoes less decrease in life even when operated at an inner temperature as high as 1200°C or higher around the resistive heating element during afterglow, which is a temperature higher than in typical operation; therefore, the glow plug is suitable for use in internal combustion engines required to achieve less gas emission.

[0044] A glow plug according to the present invention is not limited to the embodiments described above and is subject to various modifications without departing from the scope of the invention.

[0045] For example, the heating coil 820 serving as a resistive heating element in the glow plug 10 of the embodiment described above is spirally wound; however, the shape of the resistive heating element is not particularly limited and may have a rod shape extending in the axial line O direction.

[0046] The shape of the tubular sheath may be any tubular shape. For example, the cross-sectional shape of the sheath taken in a direction orthogonal to the axial line O may be circular, elliptical, polygonal, or the like.

[0047] The glow plug 10 of the embodiment described above includes the control coil 830 placed between the heating coil 820 and the center rod 200 to prevent overheating of the heating coil 820. Alternatively, the heating coil 820 may be directly jointed to the center rod 200 and the control coil 830 may be omitted.

EXAMPLES

Production and analysis of glow plug

[0048] A glow plug having the same structure as the glow plug 10 shown in Fig. 1 was prepared as described above. The material used to form the tubular sheath precursor was INC601, and the material used to form the control coil 830 was pure Ni. The material used to form the heating coil 820 was an Fe-Cr-Al alloy. The Al content in the alloy was varied from one sample number to another. A mixture of MgO powder and SiO_2 powder was used as the insulating powder 840. The SiO_2 content thereof was varied from one sample number to another.

[0049] The Al content of the heating coil 820 was measured as described above. First, the heater 800 of each sample was cut along a plane that included the axial line O, and the section was polished to mirror finish. The heating coil 820 appeared as plural circular spots 820 in one section. Of these circular spots 820, ten spots 820 were arbitrarily selected. For each circular spot 820, five points near the center of the spot 820 were arbitrarily selected and used as the measurement points for elemental analysis using the WDS of an EPMA as described above. The arithmetic means of the measured values from a total of 50 measurement points was assumed to be the composition of the heating coil 820, and the Al content of the heating coil 820 was determined.

[0050] The MgO content and the SiO_2 content of the insulating powder 840 were measured as described above by using an ICP emission spectroscope. All samples contained a total of 99.4% by mass of MgO and SiO_2 as the insulating powder 840.

[0051] The particle size distribution was measured for MgO powder and SiO_2 powder in all samples using a laser diffraction particle size distribution analyzer. The average particle size of the SiO_2 powder was smaller than the average particle size of the MgO powder.

Test I

[0052] The glow plug 10 was subjected to Test I involving applying voltage to the glow plug 10 so that the surface temperature of the position 2 mm from the distal end of the heater 800 toward the proximal end in the axial line O direction was 1200°C ±10°C and electrifying the glow plug 10 for 150 hours while keeping this surface temperature. Four glow plugs were prepared for each sample number and Test I was performed on each of the four glow plugs. The inner temperature around the heating coil 820 was assumed to be maintained at 1200°C or higher since the inner temperature is usually higher than the surface temperature of the heater 800.

[0053] Results of Test I are shown in Table 1. In Table 1, YES is indicated if disconnection occurred during Test I and NO is indicated if disconnection did not occur during Test I. After Test I, the heater 800 was cut along the plane that included the axial line O, the section was polished, and the polished surface was observed with a scanning electron microscope (SEM). In Table 1, YES is indicated if a Mg aggregation region 850 was observed as shown in Fig. 3A, and NO is indicated if the Mg aggregation region 850 was not observed as shown in Fig. 3B.

[0054] Evaluation was conducted as follows. A sample whose evaluation results included one or more "YES" was rated "Poor" and a sample whose evaluation results included only "NO" was rated "Good".

Table 1

Sample No.	Heating coil	Insulating powder	S/A	Lot No.	Test results		Rating
	Al content (A) (mass%)	SiO_2 content (S) (mass%)			Whether disconnection occurred	Whether Mg aggregation occurred	
1	5	0	0	1	YES	YES	Poor
				2	NO	YES	
				3	NO	YES	
				4	NO	NO	
2	5	0.1	0.02	1	NO	YES	Poor
				2	NO	NO	
				3	NO	NO	
				4	NO	NO	
3	5	0.2	0.04	1	NO	NO	Good
				2	NO	NO	
				3	NO	NO	
				4	NO	NO	
4	7.5	0	0	1	YES	YES	Poor
				2	NO	YES	
				3	NO	YES	
				4	NO	YES	
5	7.5	0.2	0.027	1	NO	YES	Poor
				2	NO	NO	
				3	NO	NO	
				4	NO	NO	
6	7.5	0.3	0.04	1	NO	NO	Good
				2	NO	NO	
				3	NO	NO	
				4	NO	NO	

Test II

[0055] The glow plug 10 was subjected to Test II involving applying voltage to the glow plug 10 so that the surface temperature of the position 2 mm from the distal end of the heater 800 toward the proximal end in the axial line O direction was 1200°C ±10°C and electrifying the glow plug 10 for 50 hours or 150 hours while keeping this surface temperature.

[0056] The metallic Al content of the heating coil 820 of each glow plug 10 was measured before performing Test II, after 50 hours of electrification, and after 150 hours of electrification. The Al content in the heating coil 820 was determined as in Test I and the result was assumed to be the metallic Al content. Mapping analysis of the section of the heater 800 by EPMA detected oxygen in the outer peripheral portions of the heating coil 820 that appeared as the circular spots in the section of the heater 800, but no oxygen was detected near the center of each circular spot. Accordingly, no Al oxides exited near the center of the heating coil 820. The Al content obtained by measuring the portion near the center of the heating coil 820 is thus the metallic Al content. The metallic Al content of the heating coil before performing Test II (initial) was assumed to be 100, and the ratio of the metallic Al content after 50 hours of electrification and the ratio of the metallic Al content after 150 hours of electrification relative to the metallic Al content 100 before performing Test II (initial) were calculated. The results are shown in Table 2.

[0057] Evaluation was conducted as follows. A sample in which the metallic Al content ratio after 50 hours of electrification was 40% or less and/or the metallic Al content ratio after 150 hours of electrification was 30% or less was rated "Poor". A sample in which the metallic Al content ratio after 50 hours of electrification was more than 40% and the metallic Al content ratio after 150 hours of electrification was more than 30% was rated "Good". The ratings are indicated in Table 2.

Table 2

Sample No.	Heating coil	Insulating powder	S/A	Initial	Ratio (%) of metallic Al content to 100 (initial)		Rating
	Al content (A) (mass%)	SiO_2 content (S) (mass%)			50 Hr	150 Hr	
1	5	0	0	100	63.9	62.5	Good
7	5	1	0.2	100	54.0	52.1	Good
8	5	1.45	0.29	100	47.7	41.1	Good
9	5	1.55	0.31	100	45.7	39.8	Good
10	5	1.75	0.35	100	37.1	24.4	Poor
4	7.5	0	0	100	61.3	56.0	Good
11	7.5	1	0.13	100	52.7	38.7	Good
12	7.5	2.2	0.29	100	51.7	34.7	Good
13	7.5	2.3	0.31	100	48.3	33.5	Good
14	7.5	2.5	0.33	100	39.3	20.0	Poor

[0058] As shown in Table 1, the glow plugs of Sample Nos. 3 and 6 within the scope of the present invention did not undergo disconnection during Test I and no Mg aggregation region was observed therein. In contrast, the glow plugs of Samples Nos. 1, 2, 4, and 5 which had S/A smaller than 0.04 and are outside the scope of the present invention underwent disconnection during Test I and/or had a Mg aggregation region observed. Test I shows that glow plugs having S/A of at least 0.04 can suppress short-circuiting.

[0059] As shown in Table 2, in the glow plugs of Sample Nos. 1, 7 to 9, 4, and 11 to 13 whose S/A was 0.31 or less, oxidation of Al in the heating coil 820 was suppressed when voltage was applied so that the surface temperature of the heater 800 was 1200°C ±10°C and long electrification was conducted. Thus, the decrease in metallic Al content of the heating coil 820 was small. In contrast, in the glow plugs of Sample Nos. 10 and 14 outside the scope of the present invention and whose S/A was greater than 0.31, Al in the heating coil 820 was oxidized and the decrease in the metallic Al content was large compared to the glow plugs of Sample Nos. 1, 7 to 9, 4, and 11 to 13. Test II shows that the glow plugs having S/A of 0.31 or less can suppress oxidation of Al in the heating coil 820 and deterioration of the heating coil 820.

[0060] Test I and Test II show that the glow plugs within the scope of the present invention undergo less short-circuiting and deterioration even when they are operated at an inner temperature as high as 1200°C or higher around the heating coil 820 during afterglow, and that thus the decrease in life can be suppressed.

Claims

1. A glow plug comprising:

a heater including

a tubular sheath having a distal end that is closed,
a resistive heating element disposed in the tubular sheath, and
insulating powder disposed around the resistive heating element in the tubular sheath,

wherein the resistive heating element is formed of an alloy containing aluminum,
the insulating powder contains MgO and at least one oxide selected from the group consisting of CrO_3, Cr_2O_3, SnO, FeO, Fe_3O_4, MnO, MnO_2, Mn_2O_3, Mn_3O_4, SiO_2, WO_2, TiO_2, Ti_2O_3, and ZrO_2, and
A representing an amount of aluminum contained in the resistive heating element in terms of mass percent and

S representing an amount of the oxide contained in the insulating powder in terms of mass percent on an oxide basis satisfy $0.04 \leq S/A \leq 0.31$.

2. The glow plug according to Claim 1, wherein the oxide is SiO_2.

3. The glow plug according to Claim 1 or 2, further comprising a center rod electrically coupled to the resistive heating element, the center rod having a part that protrudes from an opening of a proximal end of the tubular sheath; and a packing disposed between the center rod and the tubular sheath to inhibit gas circulation between interior and exterior of the tubular sheath.

FIG. 1

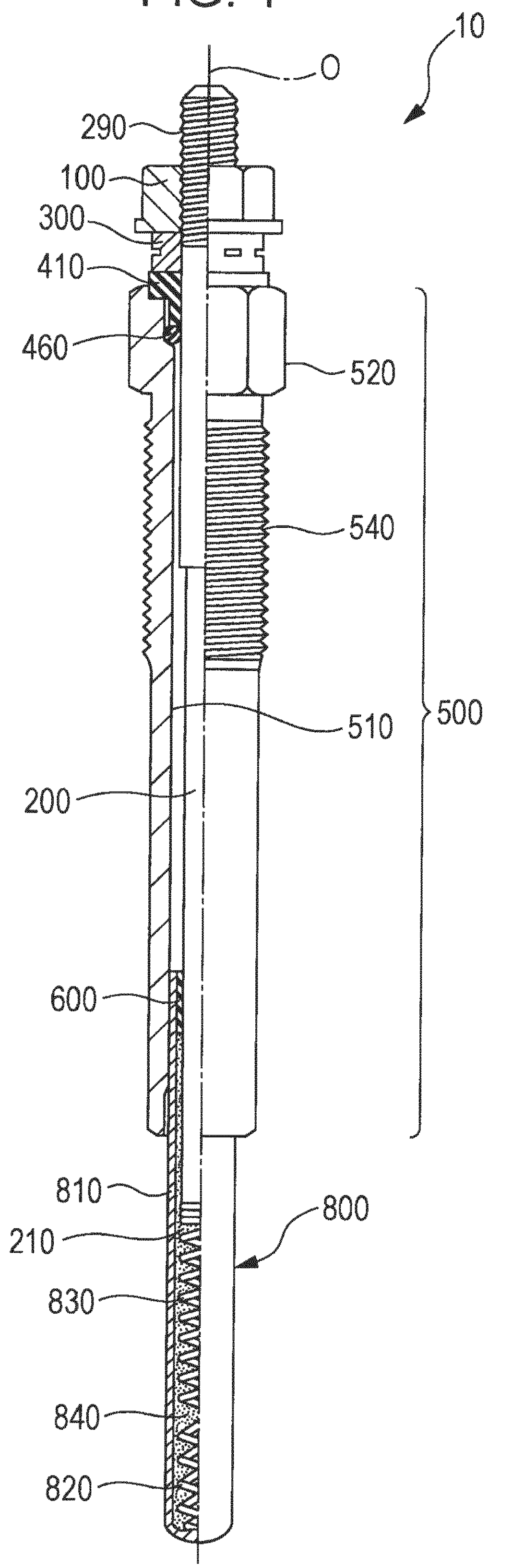
10
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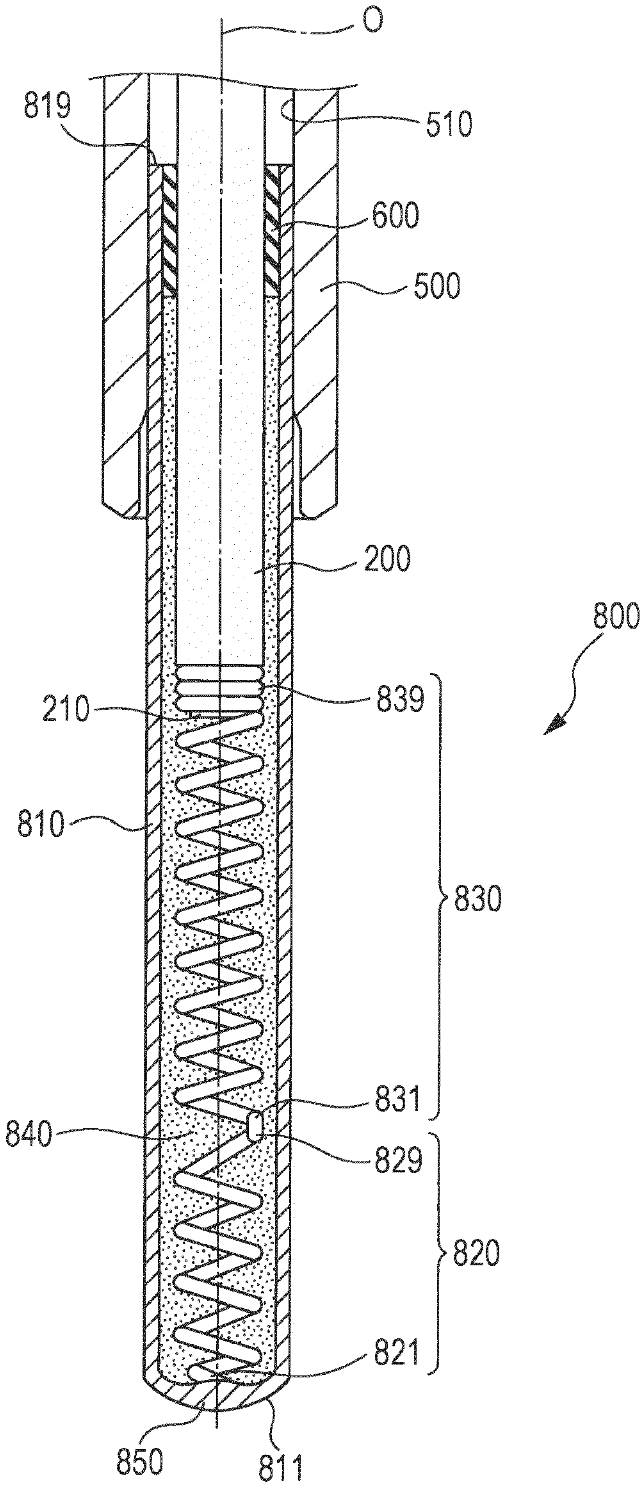
FIG. 2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811

FIG. 3A

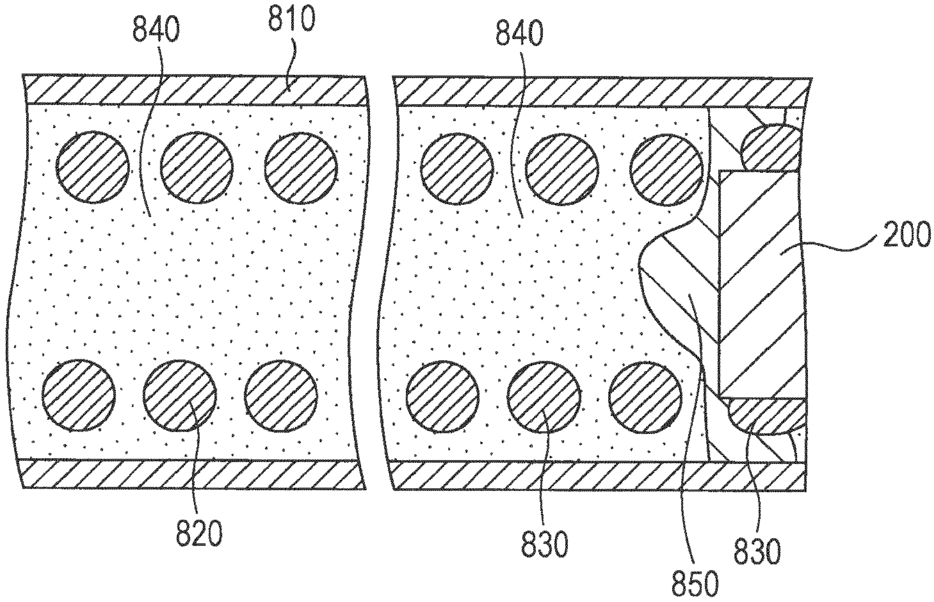
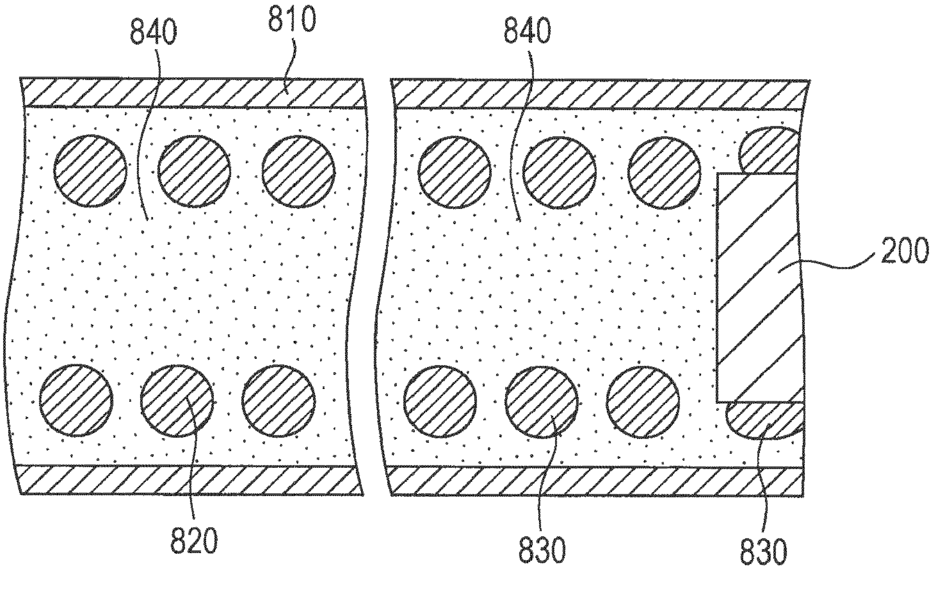


FIG. 3B



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EUROPEAN SEARCH REPORT

Application Number
EP 15 19 1142

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Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
X	JP 2008 016304 A (NGK SPARK PLUG CO) 24 January 2008 (2008-01-24) * figures 1, 2, and the relevant description * -----	1-3	INV. F23Q7/00 F23Q7/22
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X	WO 2011/162074 A1 (NGK SPARK PLUG CO [JP]; YATSUYA YOSUKE [JP]; SAKURAI TOSHIYUKI [JP]; N) 29 December 2011 (2011-12-29) * figures 2-8, and the relevant description * -----	1-3	
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The present search report has been drawn up for all claims

1

Place of search	Date of completion of the search	Examiner
Munich	22 March 2016	Rudolf, Andreas

CATEGORY OF CITED DOCUMENTS

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