

[54] **PROCESS OF COATING A GAS TURBINE ENGINE ALLOY SUBSTRATE**[75] Inventor: **Ray R. Dils**, Madison, Conn.[73] Assignee: **United Aircraft Corporation**, East Hartford, Conn.[22] Filed: **Aug. 6, 1973**[21] Appl. No.: **386,266**[52] **U.S. Cl.** **428/216**; 29/196.6; 29/197; 29/573; 136/201; 204/192; 148/6.3; 427/77; 427/123; 428/469[51] **Int. Cl.** **B44d 1/14**[58] **Field of Search** 117/45, 50, 52, 62, 71 R, 117/71 M, 105, 130 R, 131; 29/573, 196.6, 197; 204/192; 75/126 G, 134, 138, 144, 171

[56]

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[57]

ABSTRACT

A method of coating a gas turbine engine alloy substrate comprising depositing a rare earth and aluminum-containing alloy initial layer to a thickness sufficient to produce and maintain an adherent irregular aluminum oxide, mechanically working the surface of the initial layer to induce irregularity and angular topography in the aluminum oxide to be produced, oxidizing the initial layer to produce a sufficiently thick and irregular aluminum oxide layer to establish mechanical adherence of a noble metal layer and prevent alloying between the initial layer and the noble metal layer, depositing a noble metal layer on the oxidized layer to a thickness of approximately 0.1–0.2 mils and oxidatively treating the coated substrate to cause additional growth of the oxide layer to metallurgically insulate the noble metal layer from the substrate and the initial metal layer.

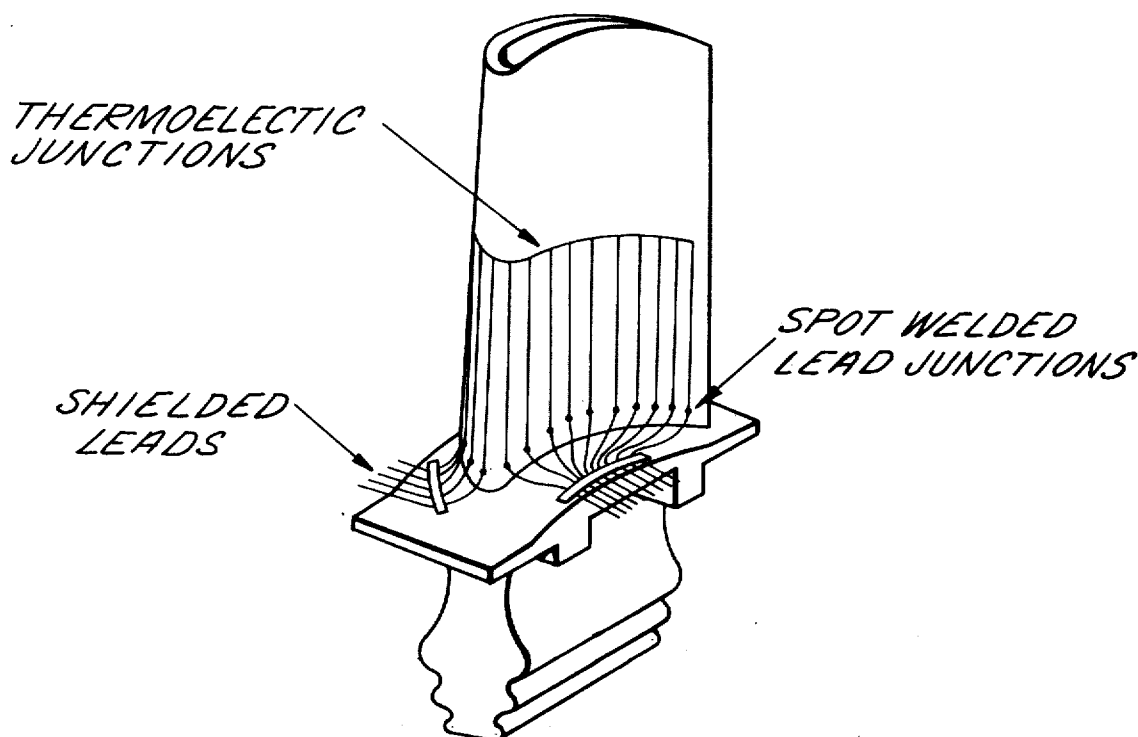
14 Claims, 11 Drawing Figures

FIG. 1a

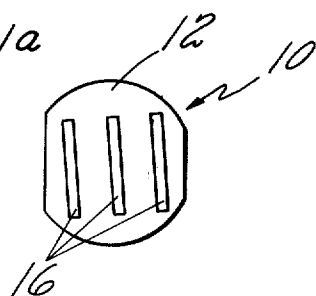


FIG. 1b

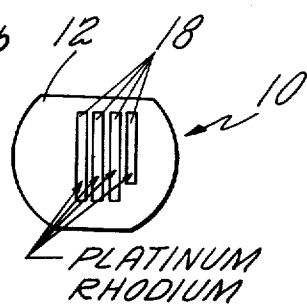


FIG. 1c

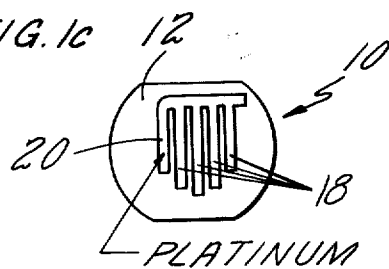


FIG. 1d

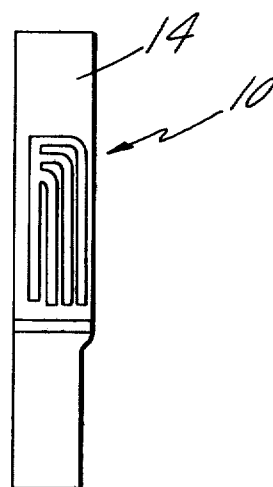
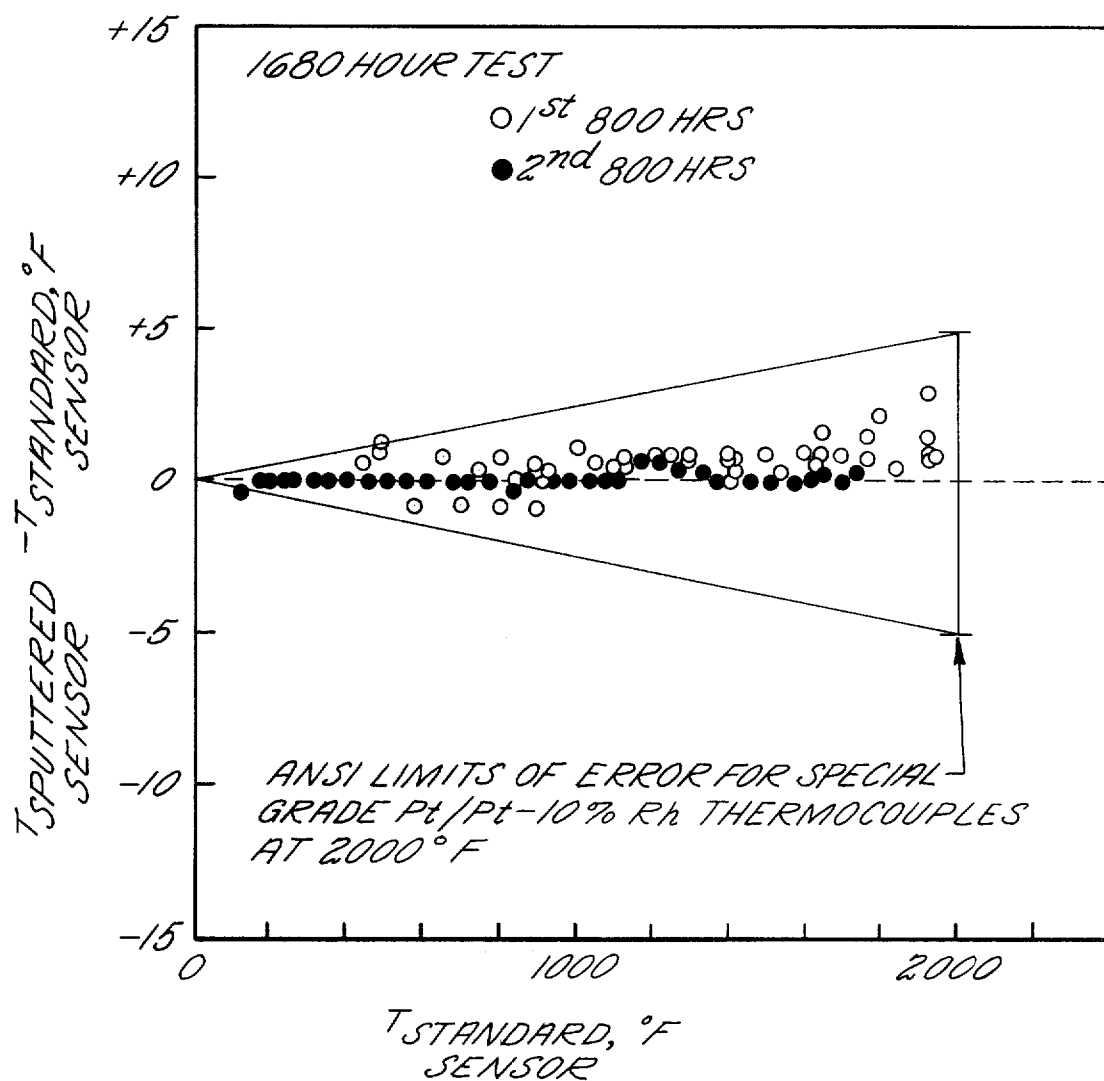
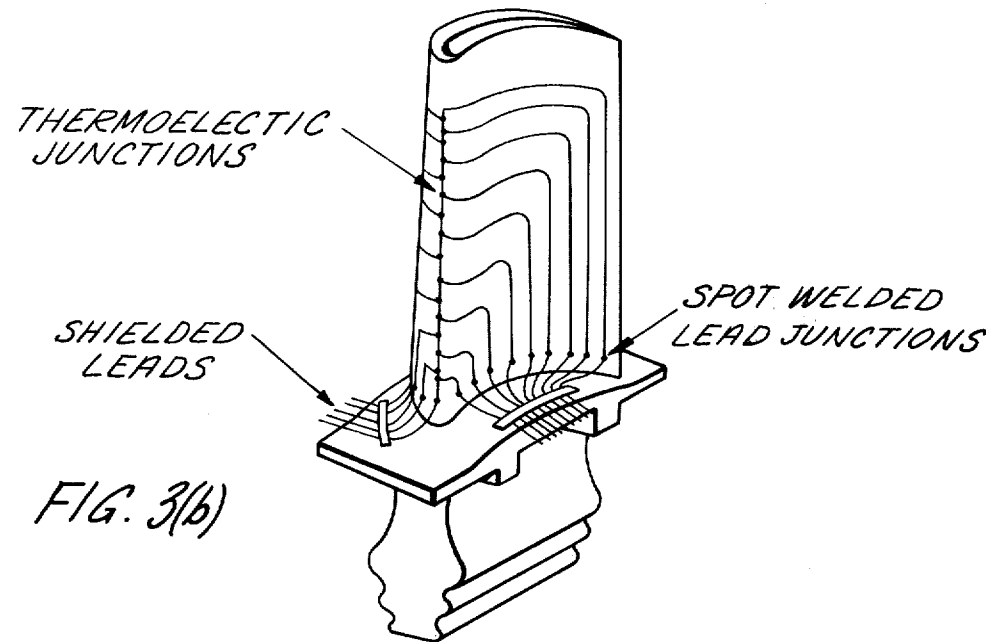
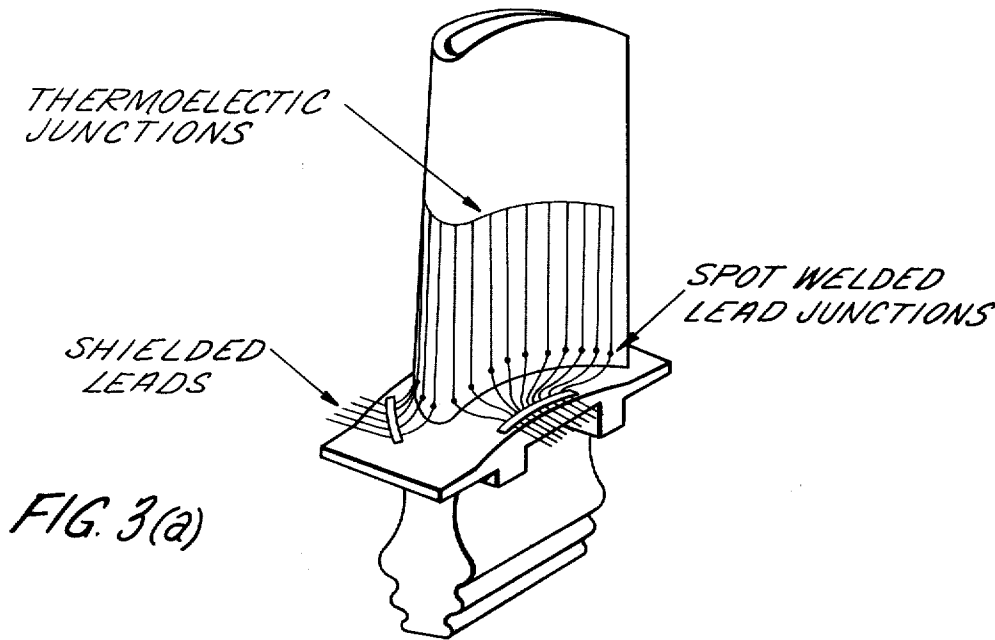
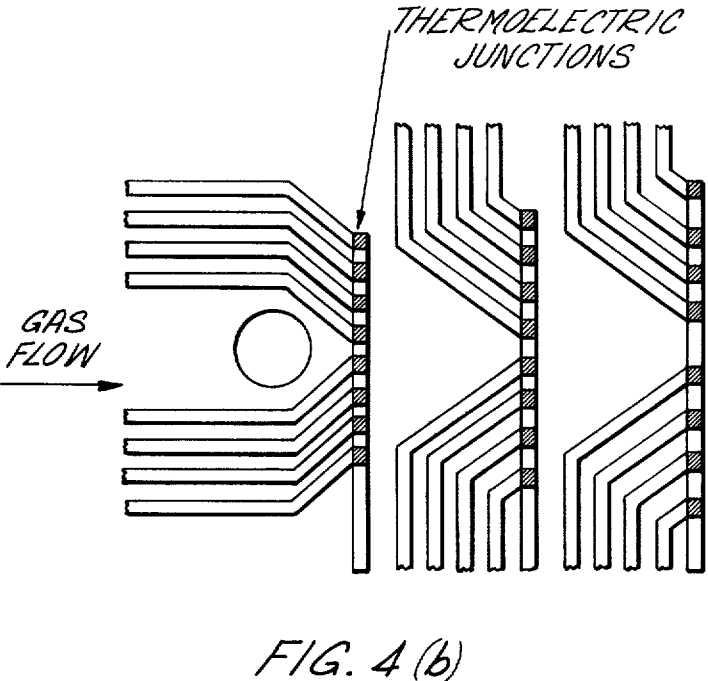
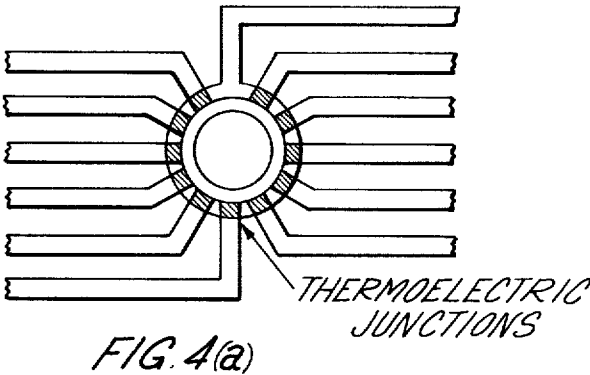


FIG. 2







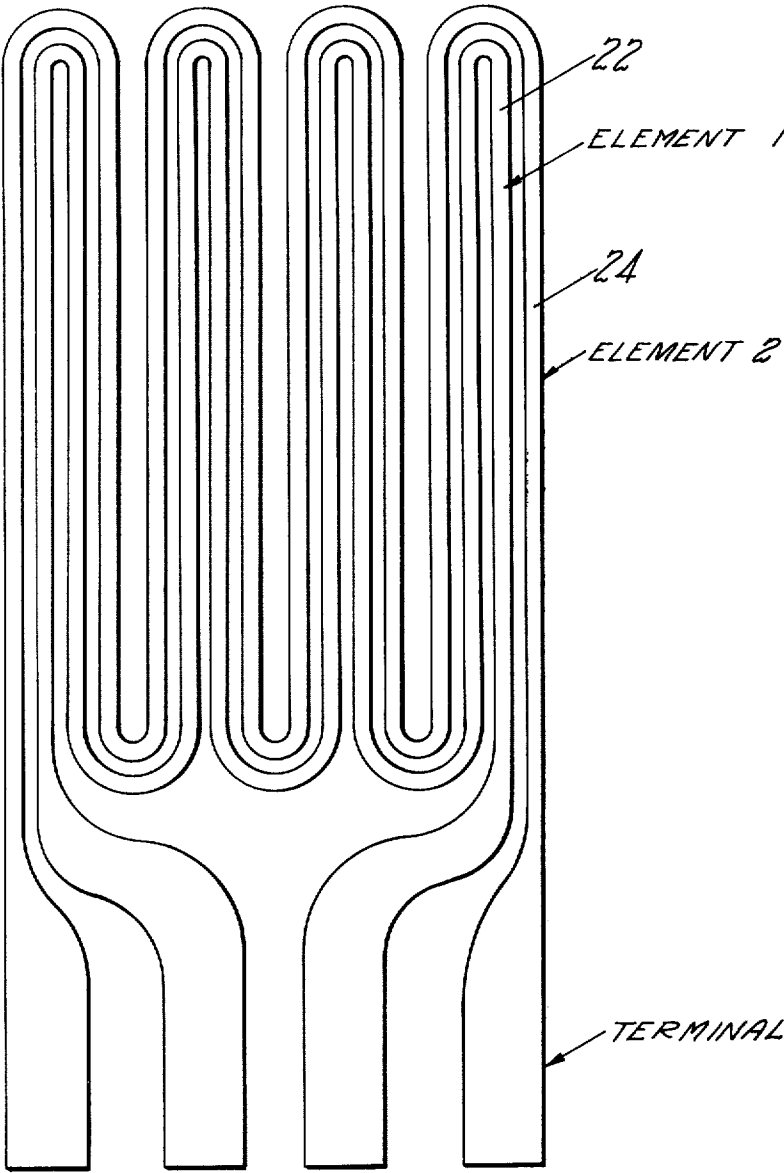


FIG. 5

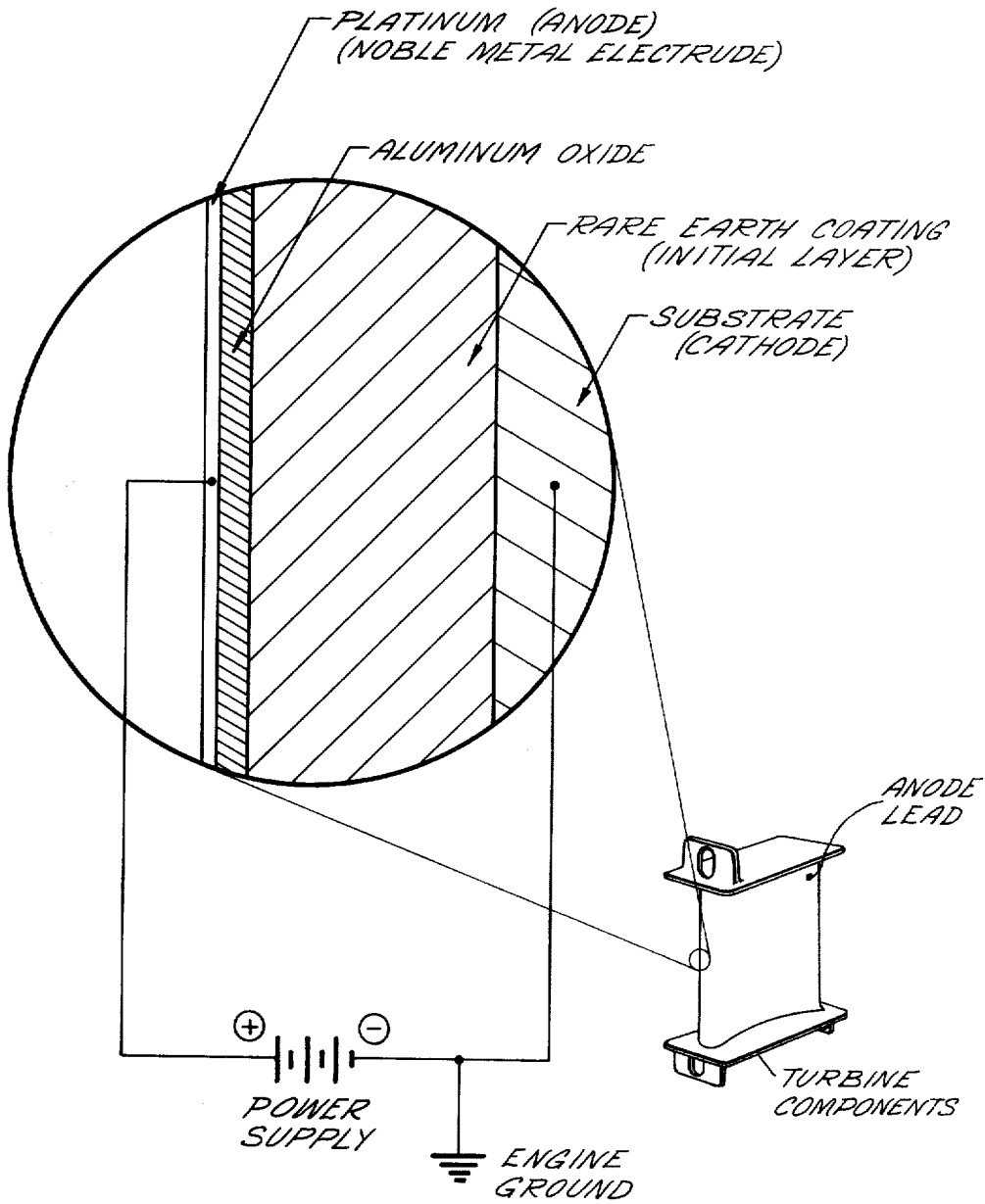


FIG. 6

PROCESS OF COATING A GAS TURBINE ENGINE ALLOY SUBSTRATE

BACKGROUND OF THE INVENTION

The present invention relates to the treatment of metals and alloys and more particularly relates to a method for coating gas turbine engine components either partially, as in the form of a thin strip array to provide surface temperature or surface strain sensors therefor, or completely to provide improved resistance of the component to high temperature sulfidation or oxidation.

One of the problems facing the gas turbine industry has been the need for sensors to provide accurate data such as the steady state temperature of static or rotating components either in or out of the gas path. The severe operating environment of gas turbine components presents particularly difficult problems in view not only of the requirement that the temperature cycling of the engine be withstood but also that there be compatibility with the substrate component and no perturbation of the airflow near or heat flow to the component. As will be appreciated, a sensor on a turbine airfoil capable of obtaining accurate broadband turbine temperatures including those in excess of 2000°F without perturbing airflow is an important step forward in the art.

SUMMARY OF THE INVENTION

The present invention relates to a method of coating a nickel-base, cobalt-base or iron-base gas turbine engine alloy. The invention contemplates a method comprising (1) depositing a rare earth and aluminum containing alloy initial layer to a thickness sufficient to produce and maintain an adherent irregular aluminum oxide, preferably NiCrAlY, CoCrAlY or FeCrAlY to a thickness of 0.5-5.0 mils, (2) mechanically working the surface of the initial layer to induce irregularity and angular topography in the aluminum oxide to be produced, preferably by grit blasting or peening, (3) oxidizing the mechanically worked initial layer to produce a sufficiently thick and irregular aluminum oxide layer to promote mechanical adherence of a noble metal layer and to prevent alloying between the initial layer and the noble metal layer, preferably by an oxidation treatment to form an oxide layer 0.05-0.1 mil thick such as heating, in air, for 70-170 hours at 1900°F, (4) depositing a noble metal layer on the oxidized initial layer to a thickness to form a noble metal thermocouple, preferably approximately 0.1-0.2 mil and (5) oxidatively treating the coated substrate to cause additional growth of the oxidized initial layer to metallurgically insulate the noble metal layer from the substrate and the initial layer. In the production of surface temperature sensors on the substrate, the noble metal coating is in the form of a suitable thin strip array of thermoelectric junctions having thickened end portions suitable for use as terminal connections.

In the production of surface strain sensors on the substrate, the noble metal coating is in the form of an array of first and second thin strip elements, the noble metal of the first thin strip element, preferably platinum, having a large temperature coefficient of resistivity with respect to the noble metal of the second thin strip element and the second thin strip element, preferably an alloy consisting essentially of 8-12 weight percent W, balance Pt, having a large strain coefficient of resistivity with respect to the first or in the form of an array

consisting of the strain sensitive element, 8-12 weight percent W, balance Pt, and a sputtered Pt/Pt-Rh thermocouple located near the center of the strain sensitive element. To reduce the rate of oxidation of the Pt-W alloy element above approximately 1500°F, a protective layer of aluminum oxide or calcium stabilized zirconia may be provided, preferably by RF sputtering thereover.

The basic method disclosed herein is particularly useful for overcoating gas turbine components to provide increased resistance to sulfidation as well as to high temperature oxidation. In order to reduce or prevent further growth of the aluminum oxide layer on the component, an electric field is superimposed across the aluminum oxide layer with the noble metal layer as the anode and the substrate as the cathode.

BRIEF DESCRIPTION OF THE DRAWINGS

An understanding of the invention will become more apparent to those skilled in the art by reference to the following detailed description when viewed in light of the accompanying drawings, wherein:

FIG. 1(a) is a plan view showing noble metal test elements on a flat disk;

FIG. 1(b) is a plan view showing an incomplete four-junction sensor on a flat disk;

FIG. 1(c) is a plan view showing a completed four-junction sensor on a flat disk;

FIG. 1(d) is a side elevational view of a three-junction sensor array on an erosion bar;

FIG. 2 is a chart showing sensor accuracy;

FIGS. 3(a) and 3(b) are perspective views of a turbine blade having large scale sensor arrays on their surface;

FIGS. 4(a) and 4(b) are diagrammatic plan views of small scale sensor arrays near cooling holes;

FIG. 5 is a diagrammatic plan view of a two-element strain sensor; and

FIG. 6 is a perspective view, partly cross-sectionally enlarged, of a turbine component showing the imposition of an electric field across the oxide coating.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

The nickel-base, cobalt-base and iron-base gas turbine engine alloys are those strong, high temperature materials suitable for use in gas turbine engine applications. Typical of the alloys which may be coated according to the present invention are the so-called nickel-base and cobalt-base superalloys, viz., those which generally contain 5-25 weight percent Cr, 5-15 weight percent Mo, Ta or W and 2-8 weight percent Al and Ti. Also useful as substrates are the high temperature iron-base alloys such as the austenitic stainless steels or Kanthal A (5.5 Al, 22 Cr, balance Fe).

In the production of surface temperature sensors on gas turbine engine components, the first step is the deposition of an initial layer of an alloy onto a gas turbine engine alloy substrate. The initial layer is any rare earth or rare earth particle-containing alloy which can form an adherent irregular aluminum oxide. In general, the initial layer contains no more than approximately 2 percent, by weight, of the rare earth metal and approximately 5-25 percent, by weight, aluminum and preferably consists of a coating such as NiCrAlY (20-35 weight percent Cr, 15-20 weight percent Al, 0.05-0.3 Y, balance Ni), CoCrAlY (19-24 weight percent Cr,

13-17 weight percent Al, 0.6-0.9 Y, balance Co), FeCrAlY (25-29 weight percent Cr, 12-14 weight percent Al, 0.6-0.9 Y, balance Fe) or an alloy such as 25 Cr, 15 Ni, 5 Ta, 5 Al, 0.1 Y, balance Co. In some cases it is desirable to use alloys with the same base metal in the substrate and initial layer, e.g., FeCrAlY with iron-base alloys, CoCrAlY with cobalt-base alloys, etc. However, in general, various combinations may be utilized. The initial layer thickness must only be thick enough to produce and maintain an adherent metal oxide, preferably approximately 0.5-5 mils, and may be deposited by conventional techniques as by sputtering or evaporation.

The second step in the construction of the coating is the mechanical working of the initial layer to induce the growth of an adherent metal oxide with an extremely irregular and angular topography. The subsequently deposited noble metal layer is primarily mechanically bonded to the initial layer and any method of surface preparation which will induce the growth of an irregular oxide will promote its mechanical adherence thereto. Grit blasting of the surface with various sizes of grit is considered a satisfactory technique, as is peening.

In the third step of the construction of the sensors, the initial layer is oxidized to produce a sufficiently thick and irregular oxide to promote the mechanical adherence of the noble metal layer, and to provide a sufficiently small number of paths through the oxide to the substrate to eliminate alloying between the initial layer and the noble metal layer. The oxide layer must be thin enough to permit rapid reoxidation of the specimen, provide oxide dimensions representative of the oxides grown in the turbine environment in order not to perturb the heat flow in the system and minimize the reduction of the turbine component life due to the oxidation treatment. It has been found that oxides from 0.05-0.1 mil thick which are grown in air for generally 70-300 hours at 1900°F fulfill the above requirements. However, it will be appreciated that any oxidation treatment which produces an oxide dimension approximating the above range is considered suitable.

The next step comprises the deposition of a noble metal layer to form a noble metal thermocouple. By noble metal is meant such elements or alloys as those of platinum, rhodium or palladium. Each layer is deposited, preferably by sputtering, to a thickness sufficient to be stable and durable in harsh environments yet thin enough to permit oxygen diffusion through the layer in order to insulate it during the subsequent oxidation treatment described below. It has been established that noble metal thicknesses between 0.1 and 0.2 satisfy these requirements. The adherence of the noble metal layer increases with increasing sputtering substrate temperature. However, some uses of sensors require high resolution spacial distributions of thermocouple functions on the surface of a component and these sensor arrays are best obtained by low temperature masking procedures. Thus the entire range of sputtering substrate temperatures, from room temperature to the melting point of the substrate may be utilized.

The fifth step in the process is the electrical insulation of the noble metal layer from the substrate and the initial layer. It has been found that oxidation in air for approximately 30 hours at 1900°F is sufficient to achieve this result.

The last step in the construction of a sensor is the formation of relatively thick terminals at the ends of the sputtered noble metal leads to permit lead wires to be directly connected, as by spot welding, thereto. Terminal thicknesses between 0.2-0.5 mil are sufficient to obtain a durable connection between the sensors and 0.003 mil diameter lead wires. The width of the terminals is smaller than the original width of the sputtered leads to prevent loss of the electrical insulation of the sensor.

As discussed in the following specific example, surface temperature sensors made according to the present invention have provided metal surface temperatures from 0°F to the melting point of several nickel-, cobalt- or iron-base alloys with the accuracy of special grade platinum/platinum-rhodium thermocouples. The presence of the sensor on the surface of a component did not significantly perturb the heat flow from the gas stream to the component or the heat flow within the component. The bandwidth of information was limited only by the relative amplitudes of the signal and the equivalent input noise of the associated electronics. In general, useful information may be received over a several kilohertz bandwidth. Tests indicated that the sensors are usually durable.

Example

Simple sensor elements **10** on flat disks **12** and an erosion bar **14** are shown in FIG. 1. The flat disks comprised a substrate of the nickel-base alloy B1900 (nominal composition, by weight percent, 8 Cr, 10 Co, 1 Ti, 6 Al, 6 Mo, 0.11 C, 4.3 Ta, 0.15 B, 0.07 Zr, balance Ni) and a sputtered initial layer three mils thick of FeCrAlY which had been mechanically worked by a No. 320 grit blast and subsequently oxidized in air for 170 hours at 1900°F to grow an aluminum oxide 0.1 mil thick. Platinum test elements **16**, 40 mils wide, 750 mils long and 0.1 mil thick were sputtered on the flat disk **12** shown in FIG. 1(a). The platinum-10 weight percent rhodium elements **18** of a four-junction sensor array were sputtered on the flat disk as shown in FIG. 1(b) followed by the sputtering of a platinum element **20** across the Pt-10 percent Rh elements **18** to complete the sensor as shown in FIG. 1(c). FIG. 1(d) shows a three-junction sensor array on an erosion bar. As will be appreciated, any desirable array of thermoelectric junctions can be sputtered on a turbine component. Since the initial layer coating and oxide are common to high temperature turbine components, the only real change in the component configuration is due to the sputtered noble metal layer having a thickness of 0.0001-0.0002 inch. The platinum and platinum-rhodium elements of FIG. 1 do not significantly affect the heat flow from the gas stream to the component or the heat flow within the component. For example, the narrowband (steady state) thermal impedance of a 0.0001 inch platinum element is 2.14×10^{-3} of the boundary layer impedance when $h = 1000 \text{ BTU/ft}^2 \text{ hr}^\circ\text{R}$. In addition, the thickness of the noble metal layer is small with respect to the thickness of the boundary layer and therefore does not alter the structure of the boundary layer. The platinum element narrowband impedance is 7.5×10^{-4} of the impedance of a 0.050 inch section of a nickel-base alloy. The broadband response near the turbine component surface is limited by the oxide layer. At 15 khz, a harmonic temperature wave travelling across a 0.0001 inch oxide layer is attenuated

to $1/e$ of the initial amplitude of the wave at the surface. At the same frequency, the reduction in wave amplitude across the sensor is less than six percent. Therefore, the sensor elements do not affect narrowband or broadband measurements; the useful bandwidth of the information is determined by the relative amplitudes of the signal and the equivalent input noise of the associated electronics.

It will be appreciated that the width of the sputtered sensors is extremely small, in this case over 300 times smaller than the width of conventional thermocouples used by placement in slots in airfoils to measure temperatures near the airfoil surfaces. The sputtered sensor width is over 100 times smaller than the conventional strain and temperature sensors presently applied externally to airfoil surfaces.

The measurement errors of sputtered sensors of the present invention were maintained within the limits of error for special grade Pt/Pt-Rh thermocouples. A comparison of the thermoelectric voltage generated by a sputtered Pt/Pt-10 percent Rh sensor like the one shown in FIG. 1(c) and a special grade Pt/Pt-10 percent Rh thermocouple is presented in FIG. 2. The specimen was cycled from room temperature to 2000°F in random temperature intervals for two months. At each temperature, the specimen and standard thermocouple were equilibrated for at least four hours before the temperature was measured. The indicated errors are within those expected between different special grade Pt/Pt-10 percent Rh thermocouples. There appear to be no extraordinary errors associated with the sputtered sensors.

Durability of the sensors of the instant invention was proven. During several months of testing, no indication of signal deterioration due to extended exposure at high temperatures was observed. In one experiment a sensor was gradually cycled from 2000°F to room temperature for two months. The specimen holder failed but the sensor itself remained intact.

Platinum test elements such as those shown in FIG. 1(a) were cycled several hundred times from 2000°F to room temperature in a stationary gas. The thermal cycling had no effect on the test elements which remained electrically insulated from the substrate and strongly bonded to the substrate oxide. In another experiment a Pt/Pt-10 percent Rh sensor sputtered on a rod was cycled over 5000 times from 1800°F to room temperature in a moderate velocity gas stream ($Ma = 0.5$). Although the substrate was extensively cracked and plastically deformed causing a loss of electrical insulation between the sensor and the substrate, the sensor remained strongly bonded to the substrate oxide.

The sensors of the present invention are able to withstand extensive gradual or rapid plastic deformation. In one series of tests, platinum test elements sputtered on flat disks such as those of FIG. 1(a) were deformed approximately 10 percent to concave and convex shapes, yet remained attached to the substrate and electrically insulated therefrom. The sensors can be quite heavily scratched or abraded. Even if the units are inordinately handled so that a loss of insulation between the sensor elements and the substrate results, they may be repaired by reoxidizing the components. In one example, a platinum test element was struck repeatedly with a ballpeen hammer so that the sensor element was grounded to the substrate. The element was nevertheless subsequently electrically insulated from the sub-

strate by oxidizing the component for 20 hours at 1900°F.

Overall, the surface temperature sensors of the present invention provide data which cannot be obtained by state-of-the-art techniques of the gas turbine industry. The sensor units provide steady state temperatures of the external surfaces of both static or rotating components either in or out of the gas path. Sensor arrays to measure large-scale span and radial temperature distributions are shown in FIGS. 3(a) and 3(b). Small-scale sensor arrays to obtain local surface temperatures near an individual cooling hole are shown in FIGS. 4(a) and 4(b). In either case, the sensors provide the actual surface temperatures in the engine and, correspondingly, detailed experimental evaluations of the present analytical models of heat transfer in the engine.

Due to the rugged nature of the sensors, the units may be applied to surfaces of details or subassemblies prior to final fabrication steps. For example, internal surface temperatures of a split blade may be obtained by application to the internal surfaces of each half before the halves are bonded together. Large-scale heat flows in the blade can be obtained from combinations of internal and external surface sensor arrays.

The sensor units provide broadband surface temperatures and the surface temperature fluctuations important to turbine component oxidation may be obtained. Arrays of the sensors provide broadband correlations between temperature fluctuations at different locations on an airfoil. Direct, broadband evidence of the location, stability and efficiency of transpiration cooling jets may also be obtained.

The present invention also contemplates the production of two element strain sensors for use in gas turbines. A typical array is shown in FIG. 5. The process steps for making the two-element strain sensor include the six steps described above for the surface temperature sensors except that the sputtering of the noble metal layer is done with two different metals to form separately the first thin strip element 22 and the second thin strip element 24. The first element 22 must have a large temperature coefficient of resistivity relative to the second element and is preferably platinum while the second element must have a large strain coefficient of resistivity relative to the first element and is preferably a platinum alloy containing 8-12 weight percent tungsten. Alternatively, the strain sensor may be constructed with a strain sensitive element as described and a sputtered Pt/Pt-Rh thermocouple located near the center of the strain sensitive element. To reduce the rate of oxidation of the Pt-W alloy element above approximately 1500°F, a protective layer of aluminum oxide or calcium stabilized zirconia is deposited, preferably by sputtering, to a minimum thickness sufficient to protect the sensor element from the environment, e.g., to 0.1-0.5 mil.

In addition to the utilization of the basic five-step procedure for producing surface temperature sensors, surface strain sensors and a simple gas turbine component coating for protection against sulfidation, it may be utilized, with the addition of a step wherein an electric field is superimposed across the oxide to prevent high temperature oxidation. The field acts to cancel the electromechanical gradient which occurs naturally within the oxide and which provides the driving force for cation and/or anion motion in the oxide. The noble metal layer, preferably platinum, is the anode and the

metallic coating is the cathode which is at the engine ground potential as shown in FIG. 6. Fields on the order of 10^4 volts/cm are sufficient to reduce the rate of oxidation. In one test, it was experimentally observed that a one volt potential across a 1μ (10^{-4} cm) oxide significantly reduces the rate of oxidation. A specimen having a FeCrAlY coating was prepared with No. 320 grit blast and preoxidized for 24 hours at 2000°F. Three 0.1 mil Pt electrodes were sputtered on the oxidized surface and the specimen was reoxidized for 24 hours at 2000°F. Positive and negative potentials were applied to two electrodes and the specimens were again oxidized. After an oxidation of 120 hours at 2000°F in the presence of the electric fields, the specimens were cross sectioned and measurements were made of the oxides beneath each electrode including the electrode to which no voltage had been applied. The results indicated that with the platinum electrode as the anode, a field of approximately 8×10^3 volts/cm reduced the rate of oxidation by a factor of two whereas with the platinum electrode as the cathode, a field of approximately 1.2×10^4 volts/cm increased the rate of oxidation by a factor of three.

It was determined that with the noble metal layer as the anode, the rate of oxidation decreases as the voltage increases until the electrochemical gradient and the opposing electrical field balance and oxidation ceases. The voltage at which oxidation ceases should be the voltage equivalent of the change in free energy of the oxidation reaction which, in the case of aluminum oxide, is approximately 2.1 volts.

What has been set forth above is intended primarily as exemplary to enable those skilled in the art in the practice of the invention and it should therefore be understood that, within the scope of the appended claims, the invention may be practiced in other ways than as specifically described.

What is claimed is:

1. In a method for coating nickel-base, cobalt-base or iron-base gas turbine engine alloy substrates having an initial rare earth and aluminum-containing nickel-, cobalt- or iron-base alloy coating approximately 0.5–5.0 mils thick thereon, said initial coating containing no more than 2%, by weight, rare earth metal and approximately 5–25%, by weight, aluminum, the improvement which comprises:

mechanically working the surface of said initial layer to induce irregularity and angular topography in the aluminum oxide to be produced;

oxidizing said initial layer to produce an irregular aluminum oxide layer approximately 0.05–0.1 mil thick to promote mechanical adherence of a noble metal layer and to prevent alloying between said initial layer and said noble metal layer;

depositing a noble metal layer selected from the group consisting of platinum, rhodium, palladium and alloys thereof on said oxidized initial layer to a thickness of approximately 0.1–0.2 mil; and oxidizing said coated substrate to cause additional growth of said oxidized initial layer to metallurgically insulate said noble metal layer from said substrate and said initial layer.

2. A method of coating an alloy substrate selected from the group consisting of the nickel-base, cobalt-base and iron-base gas turbine engine alloys comprising:

depositing an initial rare earth and aluminum-containing alloy layer on said substrate to a thickness of approximately 0.5–5.0 mils, said initial layer being an alloy selected from the group consisting of, by weight, 20–35% Cr, 15–20% Al, 0.05–0.3% Y, balance Ni; 19–24% Cr, 13–17% Al, 0.6–0.9% Y, balance Co; 25–29% Cr, 12–14% Al, 0.6–0.9% Y, balance Fe; and 25% Cr, 15% Ni, 5% Ta, 5% Al, 0.1% Y, balance Co;

mechanically working the surface of said initial layer to induce irregularity and angular topography in the aluminum oxide layer to be produced;

oxidizing said initial layer to produce an irregular aluminum oxide layer approximately 0.05–0.1 mil thick to promote mechanical adherence of a noble metal layer and to prevent alloying between said initial layer and said noble metal layer;

depositing a noble metal layer selected from the group consisting of platinum, rhodium, palladium and alloys thereof on said oxidized initial layer to a thickness of approximately 0.1–0.2 mil; and

oxidizing said coated substrate to cause additional growth of said oxidized initial layer to metallurgically insulate said noble metal layer from said substrate and said initial layer.

3. The method of claim 2 wherein said mechanical working comprises grit blasting.

4. The method of claim 2 wherein said mechanical working comprises peening.

5. The method of claim 2 wherein said initial layer is heated in air at approximately 1900°F for 70–300 hours.

6. The method of claim 5 wherein said noble metal layer is deposited by sputtering.

7. The method of claim 6 wherein said noble metal layer is deposited in the form of a thin strip array of thermoelectric junctions with thickened end portions suitable for use as terminal connections whereby said coating acts as a surface temperature sensor.

8. The method of claim 7 wherein said noble metal layer is deposited in an array of first and second thin strip elements, said first thin strip element having a large temperature coefficient of resistivity with respect to the second thin strip element and said second thin strip element having a large strain coefficient of resistivity with respect to the first whereby said coating acts as a surface strain sensor.

9. The method of claim 8 wherein platinum is deposited as the first thin strip element and an alloy consisting essentially of 8–12 weight percent tungsten, balance platinum is deposited as the second thin strip element.

10. The invention of claim 9 wherein said second thin strip element is coated with a protective oxide layer selected from the group consisting of aluminum oxide and calcium stabilized zirconia.

11. The method of claim 7 wherein said noble metal layer is deposited in the form of a thin strip element having a large strain coefficient of resistivity and a thermocouple adjacent the center of the thin strip element.

12. The method of claim 6 wherein an electric field is imposed across the aluminum oxide layer to prevent further growth thereof, said noble metal layer being the anode and said substrate being the cathode therefor.

13. The method of claim 12 wherein a voltage potential of approximately 2.1 volts is impressed across said aluminum oxide layer.

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14. In a coating for the nickel-base, cobalt-base and iron-base gas turbine engine alloys having a first rare earth and aluminum-containing nickel-, cobalt- or iron-base alloy layer approximately 0.5–5.0 mils thick, said layer containing up to 2%, by weight, rare earth metal and approximately, 5–25%, by weight, aluminum, the improvement which comprises:

a layer of aluminum oxide 0.05–0.1 mil thick bonded

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to said first metal alloy layer, said aluminum oxide layer having an irregular surface; and
a noble metal layer selected from the group consisting of platinum, rhodium, palladium and alloys thereof approximately 0.1–0.2 mil thick mechanically bonded, by virtue of said irregular surface, to said aluminum oxide layer.

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