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(54) Title of the Invention: **Method to improve the thermal properties of a resistance element embedded in an alumina deposit on a surface of a substrate and application of said method**  
Abstract Title: **A method of densifying an alumina deposit**

(57) A method is disclosed for improving the heat resistance of a resistive element embedded in an alumina deposit covering a surface of a substrate, the alumina deposit comprising a surface portion and a deep portion which is sandwiched between the surface portion and the surface of the substrate and in which the resistive element is located, which method is characterised in that it comprises a densification of the surface portion of the alumina deposit. The densification may be by impregnating the surface portion by a solution comprising alumina particles and aluminium phosphate. The method may be used to form a turbine blade.

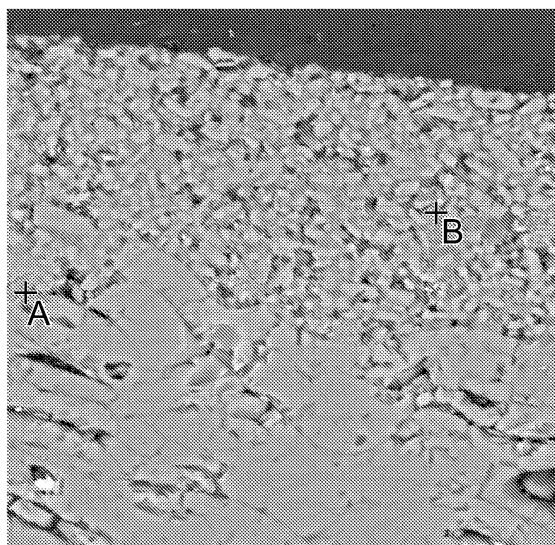


FIG. 1

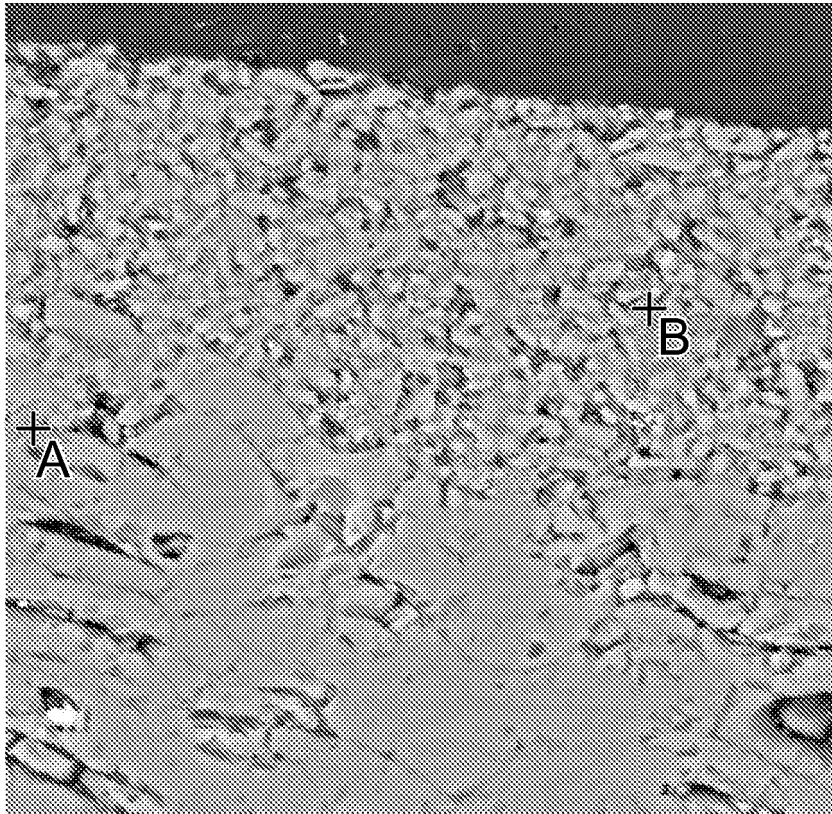


FIG. 1

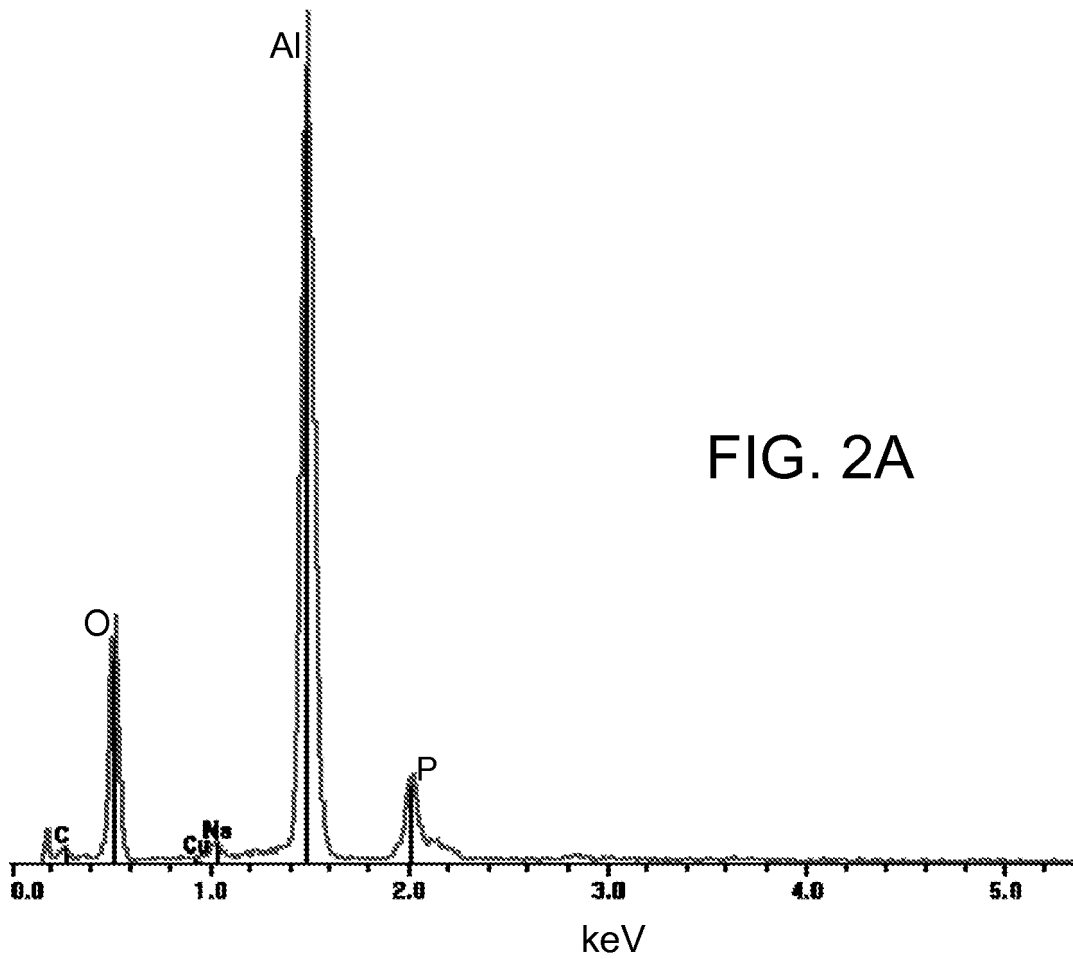
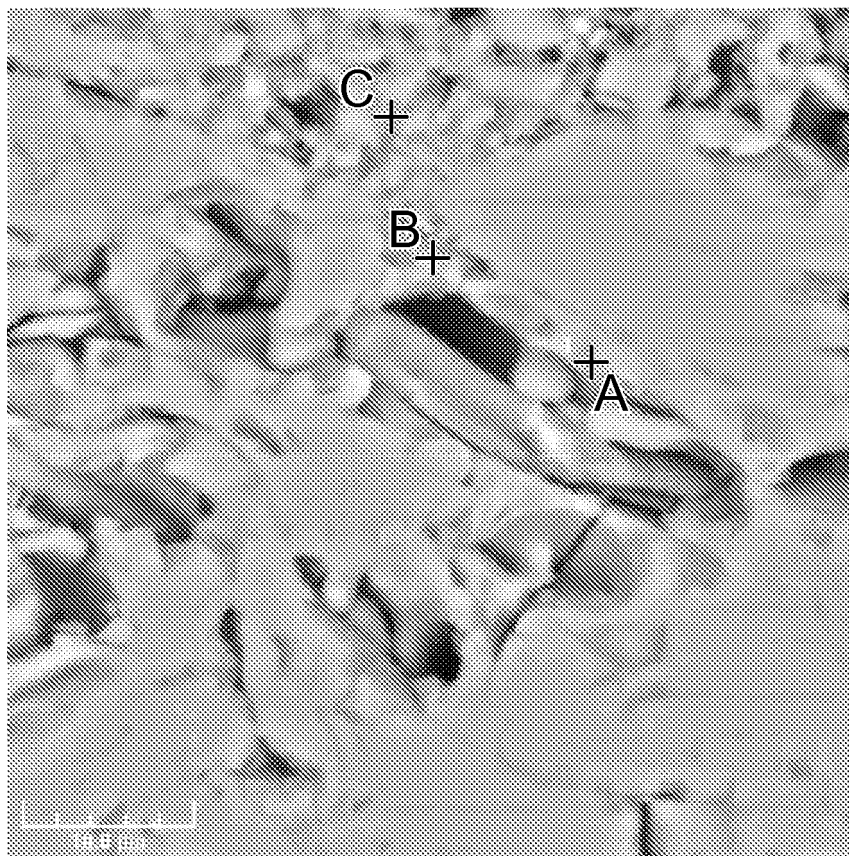
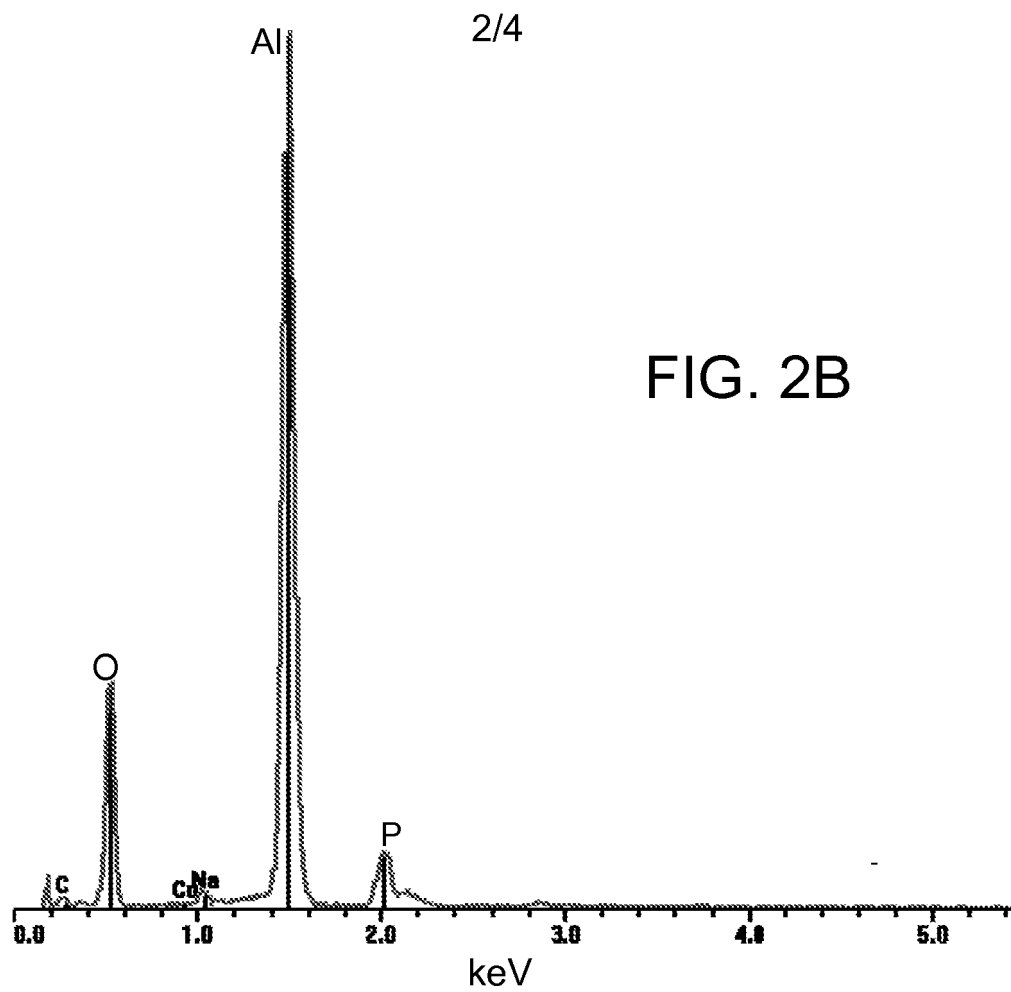
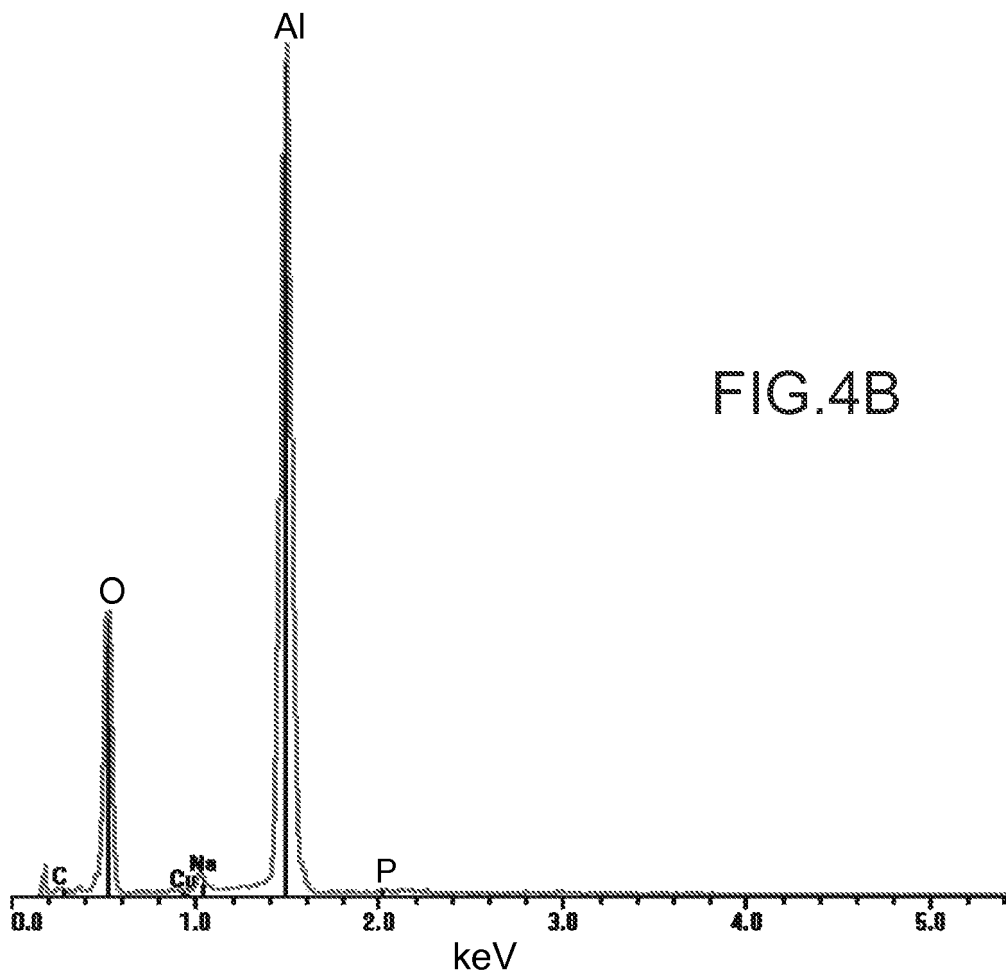
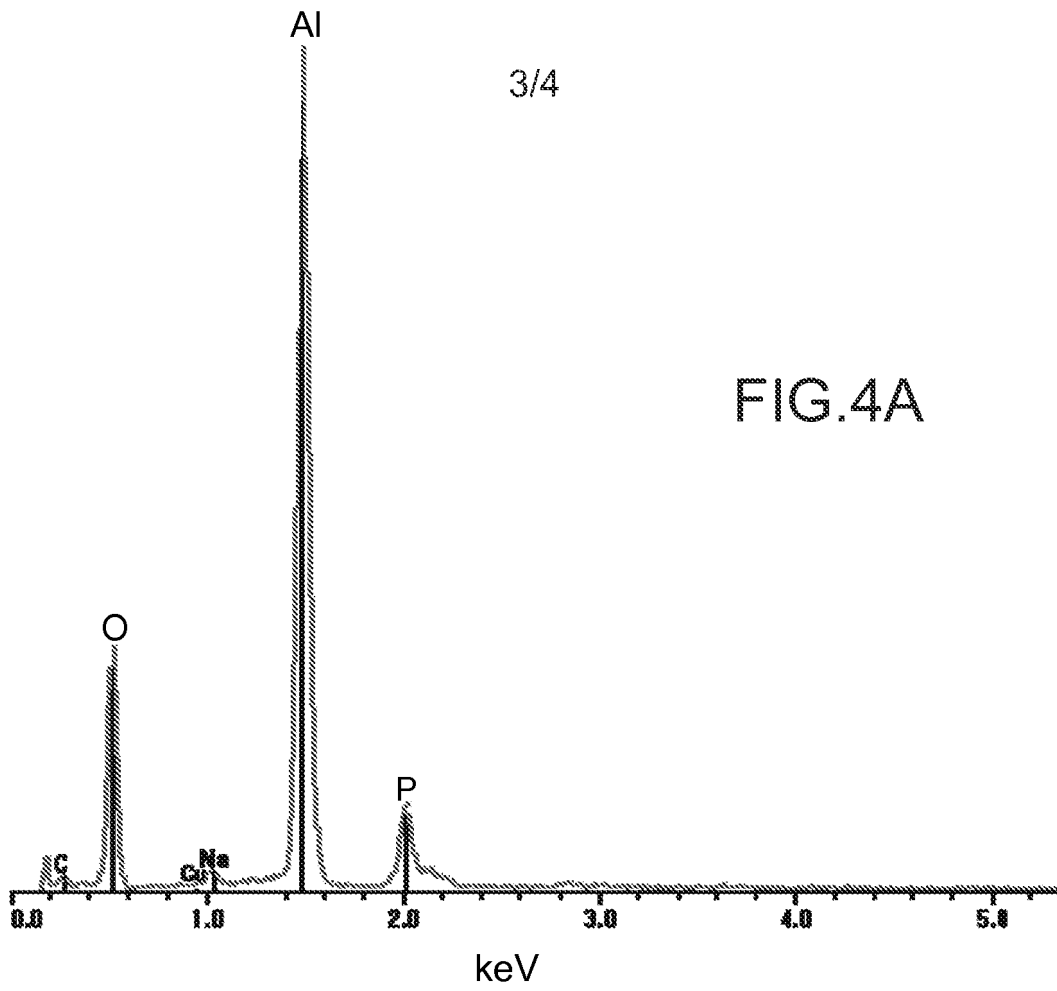


FIG. 2A





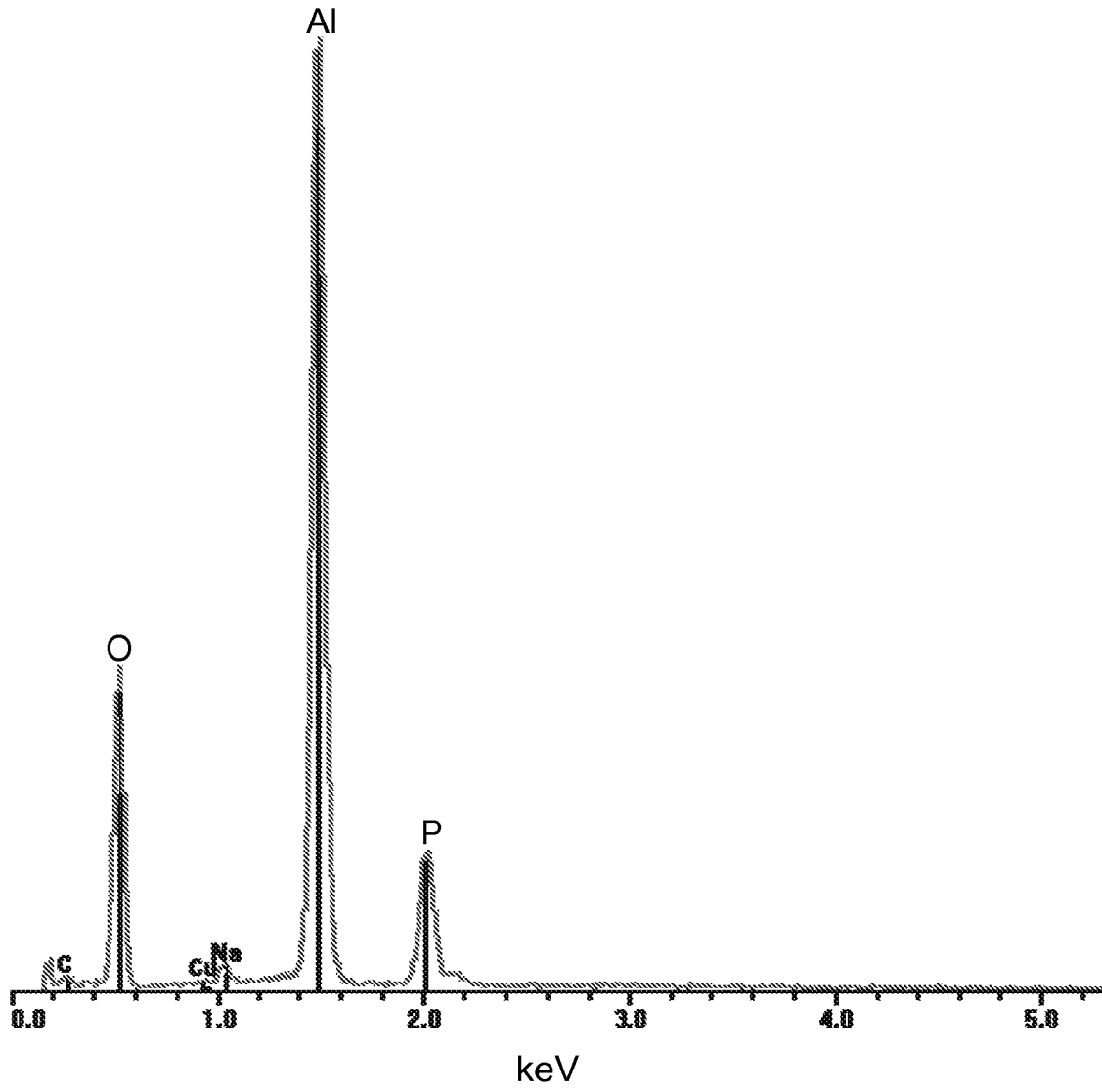


FIG.4C

**METHOD TO IMPROVE THE THERMAL PROPERTIES OF A  
RESISTANCE ELEMENT EMBEDDED IN AN ALUMINA DEPOSIT ON A  
SURFACE OF A SUBSTRATE AND APPLICATION OF SAID METHOD**

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**DESCRIPTION**

**TECHNICAL FIELD**

The invention relates to a method enabling the heat resistance of a resistive element to be improved when this resistive element is embedded in an alumina deposit on a surface of a substrate.

This method notably finds applications in all fields in which an evaluation of the deformation of mechanical parts, which are intended to be exposed to very high temperatures, i.e. typically over 950°C, by means of resistive elements of the strain gauge type, is required and, in particular, in the field of design of engine parts for aeronautics, such as turbine blades.

The invention therefore also relates to a turbine blade comprising a surface covered with an alumina deposit in which at least one resistive element is embedded, the thermal properties of which have been improved by said method.

Lastly, it relates to a turbine comprising such a blade, and also to a turbomachine comprising such a turbine.

**STATE OF THE PRIOR ART**

In the field of design of engine parts for aeronautics, and in particular turbine blades, it is habitual to use strain gauges (also called "deformation

gauges") to measure the deformations which these parts may undergo when they are subjected to an extreme environment and, notably, to very high temperatures. These measurements are essential to analyse the mechanical and thermal resistance properties of these parts, and to obtain from the authorities a certification of the aeronautical device on which they are intended to be used.

Strain gauges are passive sensors, the electrical resistance of which varies according to the deformation of the parts on which they are applied.

They conventionally consist of a grid formed by a thin conductor wire, generally a few micron thick, made of a metal alloy of the nickel-chromium, nickel-copper or platinum-tungsten type, which, depending on the size of the gauge, allows elongation of as much as 5% of the length of wire.

To attach the strain gauges on to the surface of the parts of which it is desired to measure the deformations, and to protect them from the external environment, these gauges are generally embedded in an alumina deposit, which is typically produced by thermal spraying and, notably, by flame spraying.

To facilitate the adhesion of this alumina deposit on the surface of the parts this surface is prepared beforehand, for example, by undertaking in succession kiln cleaning, degreasing and sand-blasting of it.

These operations are followed by the deposition of a metal undercoat, also called a "primer undercoat", which conventionally consists of an alloy

of the MCrAlY type (where M = Co, Fe or Ni), and which enables the adhesion of the alumina on the surface of the parts to be strengthened.

The alumina is then deposited on the primer  
5 undercoat. It generally consists of several  
superimposed layers of alumina, typically three such,  
which are deposited one by one, and each of which has a  
well-defined function. Indeed, while the first layer of  
alumina is intended to insulate the strain gauges  
10 electrically from the surface of the parts, the second  
layer of alumina is intended to lock these gauges in  
place on this surface, whereas the purpose of the third  
layer of alumina is to protect them from the  
environment.

15 It has been observed that, if a strain  
gauge is used to measure the deformations which may be  
undergone by a part at temperatures of over 950°C, the  
electrical and mechanical characteristics of this gauge  
are modified, causing a loss of the electrical signals  
20 which it is supposed to emit and, consequently, reduced  
reliability of the deformation measurements made using  
this gauge.

Reduced reliability of the measurements  
made on parts and, notably, on engine parts of an  
25 aircraft, may prejudice the certification campaign of  
this device and therefore lead to substantial delays in  
its development programme, with the risk of a  
substantial financial loss.

The Inventors therefore set themselves the  
30 goal of resolving this problem.



The Inventors also set themselves the goal that the solution to this problem should apply to any resistive element which is embedded in an alumina deposit, and the electrical and mechanical characteristics of which are likely to be modified in the presence of very high temperatures.

The Inventors also set themselves the goal that this solution should be simple to implement and not require complex and costly equipment.

#### 10 DESCRIPTION OF THE INVENTION

This aim and others are met by the invention which proposes, firstly, a method for improving the heat resistance of a resistive element embedded in an alumina deposit covering a surface of a substrate, where the alumina deposit comprises a surface portion and a deep portion which is sandwiched between the surface portion and the surface of the substrate and in which the resistive element is located, which method is characterised in that it comprises a densification of the surface portion of the alumina deposit.

Indeed, in the course of their work, the Inventors observed that the modifications of the electrical and mechanical characteristics of a resistive element, of the strain gauge type, which are observed when this resistive element is embedded in an alumina deposit on a surface which is subjected to temperatures of over 950°C results from an oxidation of the resistive element, and that this oxidation is due to the existence of interconnected pores and

microcracks (which therefore emerge on the exterior), within the alumina deposit.

By densifying the surface alumina deposit, it is therefore possible to fill the porosity and the microcracks located in the surface portion of this deposit and, hence, to create a barrier able to protect the resistive element located in the underlying portion of the alumina deposit (called in this case the "deep portion") against any oxidation, without however modifying the characteristics of the alumina deposit in this underlying portion.

The porosity and the microcracks present in the underlying portion of the alumina deposit can therefore be preserved, such that the ductility of the alumina (which enables the resistive element to fulfil its function as a measuring instrument when it is a strain gauge) in this portion may be maintained.

In accordance with the invention, the densification of the surface portion of the alumina deposit is preferably achieved by:

- a) impregnating this surface portion by a solution comprising alumina particles and an aluminium phosphate;
- b) drying the surface portion so impregnated ; and
- c) applying a heat treatment to the surface portion so dried.

The solution comprising the alumina particles and the aluminium phosphate, which is used in step a), is advantageously an aqueous solution comprising 20 to 50% by mass of aluminium phosphate,

where the latter is, preferably, aluminium tris(dihydrogenophosphate)  $[\text{Al}(\text{H}_2\text{PO}_4)_3]$ .

For reasons of practicality and also of speed of use of the method according to the invention, such a solution may be obtained by dilution with water and, preferably, distilled water, of the product which is manufactured by Aremco Products and distributed by Polytec under the commercial name Ceramacoat™ 503-VFG-C, in which case this dilution is preferably achieved by adding  $15 \pm 1\%$  by mass of water to this product (i.e. 0.14 to 0.16 gram of water for each gram of product).

As previously mentioned, step a) of the method consists in impregnating the surface portion of the alumina deposit with the solution comprising the alumina particles and the aluminium phosphate.

This impregnation is advantageously accomplished by applying this solution on to the surface of the alumina deposit by means of a paint brush.

However, it is also possible to use a sponge brush or a pneumatic spray device.

Step b) of the method consists, for its part, in drying the surface portion of the alumina deposit which has been impregnated with the solution comprising the alumina particles and the aluminium phosphate.

This drying is advantageously achieved by leaving the substrate and the alumina deposit covering it at ambient temperature, for example for a period of between 1 and 4 hours.

Step c) consists in subjecting the surface portion of the alumina deposit dried in this manner to a heat treatment.

In accordance with the invention, this heat treatment is preferably applied in stages and comprises in succession:

- heating the surface portion of the alumina deposit to a temperature  $T_1$  of between 90 and 100°C, for example 95°C, for a period of 1 to 3 hours, and ideally of 2 hours;

- heating the surface portion of the alumina deposit to a temperature  $T_2$  of between 240 and 280°C, for example of 260°C, for a period of 1 to 3 hours, and ideally of 2 hours; and

- heating the surface portion of the alumina deposit to a temperature  $T_3$  of between 350 and 390°C, for example of 370°C, for a period of 1 to 3 hours, and ideally of 2 hours.

In accordance with the invention, the thickness of the surface portion of the alumina deposit is preferably between 20 and 30%, and ideally 25%, of the total thickness of this deposit.

Furthermore, the resistive element is preferably a strain gauge, whereas the substrate is, for its part, preferably a turbine blade, in which case the alumina deposit advantageously consists of a first, a second and a third layer of alumina, which are superimposed, and which are deposited in this order on the turbine blade, preferably by thermal spraying and, more preferably, by flame spraying, and the surface portion of the alumina deposit is a portion of the

thickness of the third layer of alumina, which portion is typically 100 to 150  $\mu\text{m}$  thick. In other words, the surface portion of the alumina deposit which is impregnated by the solution comprising the alumina particles and the aluminium phosphate is typically 100  
5 to 150  $\mu\text{m}$  thick.

Another object of the invention is a turbine blade which comprises a surface covered with an alumina deposit in which at least one resistive element  
10 is embedded, the alumina deposit comprising a surface portion and a deep portion which is sandwiched between the surface portion and the surface of the blade and in which the resistive element is located, and which is characterised in that the surface portion has been  
15 densified by a method as described above.

Another object of the invention is a turbine comprising such a blade, together with a turbomachine, for example a turbojet, comprising such a turbine.

20 Other characteristics and advantages of the invention will emerge from the additional description which follows, which relates to an example of embodiment of the method according to the invention, and which refers to the appended figures.

25 It is self-evident that this example is given only to illustrate the object of the invention, and in no way constitutes a limitation of it.

## BRIEF DESCRIPTION OF THE FIGURES

Figure 1 is a scanning electron microscopy image (1,000 times enlargement) which partially shows a test piece consisting of a substrate made of a metal alloy covered with a layer of alumina, the surface portion of which has been densified by the method of the invention.

Figures 2A and 2B represent the spectra obtained by energy-dispersive analysis at the points noted A and B in figure 1.

Figure 3 is a scanning electron microscopy image (2,000 times enlargement) which partially shows another test piece consisting of a substrate made of a metal alloy covered with a layer of alumina, the surface portion of which has also been densified by the method of the invention.

Figures 4A, 4B and 4C represent the spectra obtained by energy-dispersive analysis at the points noted A, B and C in figure 3.

## DETAILED DESCRIPTION OF PARTICULAR EMBODIMENTS

Test pieces are produced consisting of a layer of alumina on a substrate made of an Ni53/Fe19/Cr19/Nb/Mo/Ti alloy (Inconel™ 718) by flame spraying of alumina rods (Rokide™). The layer of alumina is 500 µm thick.

A paint brush is then used to apply, on to the surface of the layer of alumina of these test pieces, a layer 50 to 200 µm thick of an aqueous solution, called below an "impregnation solution", which has been previously obtained by dilution of the

product Ceramacoat™ 503-VFG-C (available from the company Polytec) with distilled water, at a proportion of 0.15 g of distilled water for 1 g of product.

After drying at ambient temperature for 4 hours, the test pieces were subjected to a heat treatment comprising a first treatment lasting 2 hours at 95°C, followed by a second treatment lasting 2 hours at 260°C, and finally a third treatment lasting 2 hours at 370°C.

They were then subjected to analyses by scanning electron microscopy (SEM) and to energy-dispersive analyses (EDS).

The results of these analyses are illustrated in figures 1, 2A and 2B for a first test piece, and in figures 3, 4A, 4B and 4C for a second test piece.

Figure 1, which is an image taken using SEM (1,000 times enlargement) on the first test piece, shows a difference of appearance between the surface portion of the layer of alumina (at the top of the image), which is the portion which was impregnated by the impregnation solution and densified, and the underlying portion of this layer which was not.

Figure 2A, which represents the spectrum obtained by EDS at the point noted A in figure 1, shows the presence of phosphorus (through the presence of the phosphorus peak) at this point, and enables it to be concluded that the layer of alumina was impregnated by the impregnation solution at least as far as said point A.

Figure 2B, which represents the spectrum obtained by EDS at the point noted B in figure 1, shows the presence of the different components of the impregnation solution (through the presence of the alumina peak and of the phosphorus peak) at this point.

Figure 3, which is an image taken using SEM (2,000 times enlargement) on the second test piece, also shows a difference of appearance between the surface portion of the layer of alumina (at the top of the image), which is the portion which was impregnated by the impregnation solution and densified, and the underlying portion of this layer which was not.

Figure 4A, which represents the spectrum obtained by EDS at the point noted A in figure 3, shows a phosphorus peak and an alumina peak, marking the presence of phosphorus and, therefore, the impregnation of the layer of alumina by the impregnation solution at this point.

Figure 4B, which represents the spectrum obtained by EDS at the point marked B in figure 3, shows an alumina peak corresponding to the alumina deposit, whereas figure 4C, which represents the spectrum obtained by EDS at the point noted C in figure 3, shows the presence of the different components of the impregnation solution (through the presence of the alumina peak and of the phosphorus peak) at this point.



## CLAIMS

1. Method for improving the heat resistance of a resistive element embedded in an alumina deposit covering a surface of a substrate, the alumina deposit comprising a surface portion and a deep portion which is sandwiched between the surface portion and the surface of the substrate and in which the resistive element is located, characterised in that it comprises a densification of the surface portion of the alumina deposit.

2. Method according to claim 1, characterised in that the densification of the surface portion of the alumina deposit is achieved by:

- a) impregnating this surface portion by a solution comprising alumina particles and an aluminium phosphate;
- b) drying the surface portion so impregnated ; and
- c) applying a heat treatment to the surface portion so dried.

3. Method according to claim 2, characterised in that the solution used in step a) is an aqueous solution which comprises 20 to 45% by mass of aluminium phosphate.

4. Method according to claim 2 or claim 3, characterised in that step b) is achieved at ambient temperature for a period of 1 to 4 hours.

5. Method according to one of claims 2 to 4, characterised in that step c) comprises in succession:

5                   - heating the surface portion of the alumina deposit to a temperature  $T_1$  of between 90 and 100°C for a period of 1 to 3 hours;

                  - heating the surface portion of the alumina deposit to a temperature  $T_2$  of between 240 and  
10 280°C for a period of 1 to 3 hours; and

                  - heating the surface portion of the alumina deposit to a temperature  $T_3$  of between 350 and 390°C for a period of 1 to 3 hours.

15                   6. Method according to any one of the previous claims, characterised in that the thickness of the surface portion of the alumina deposit is between 20 and 30% of the total thickness of this deposit.

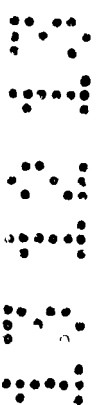
20                   7. Method according to any one of the previous claims, characterised in that the resistance element is a strain gauge, whereas the substrate is a turbine blade.

25                   8. Turbine blade, comprising a surface covered with an alumina deposit in which at least one resistive element is embedded, the alumina deposit comprising a surface portion and a deep portion which is sandwiched between the surface portion and the  
30 surface of the blade and in which the resistive element is located, characterised in that the surface portion

has been densified by a method according to any one of claims 1 to 7.

5 9. Turbine comprising a blade according to claim 8.

10. Turbomachine comprising a turbine according to claim 9.





**Application No:** GB1318171.4

**Examiner:** Nicholas Mole

**Claims searched:** 1-10

**Date of search:** 17 June 2014

**Patents Act 1977: Search Report under Section 17**

**Documents considered to be relevant:**

Category	Relevant to claims	Identity of document and passage or figure of particular relevance
A		EP 0417493 A (ALCOA) see examples and page 9 line 30
A		US 4390583 A (BRAZEL) see whole document
A		WO 2006/129097 A (HUNTERCOMBE) see examples 2 and 3
A		GB 864878 A (KOPPERS) see example V
A		US 5281439 A (LELUAN) see example 1
A		GB 1226949 A (GENERAL REFRACTORIES) see example II

**Categories:**

X	Document indicating lack of novelty or inventive step	A	Document indicating technological background and/or state of the art.
Y	Document indicating lack of inventive step if combined with one or more other documents of same category.	P	Document published on or after the declared priority date but before the filing date of this invention.
&	Member of the same patent family	E	Patent document published on or after, but with priority date earlier than, the filing date of this application.

**Field of Search:**

Search of GB, EP, WO & US patent documents classified in the following areas of the UKC<sup>X</sup> :

Worldwide search of patent documents classified in the following areas of the IPC

C04B; F01D

The following online and other databases have been used in the preparation of this search report

WPI EPODOC INSPEC



**International Classification:**

<b>Subclass</b>	<b>Subgroup</b>	<b>Valid From</b>
C04B	0041/50	01/01/2006
C04B	0035/10	01/01/2006
F01D	0005/28	01/01/2006